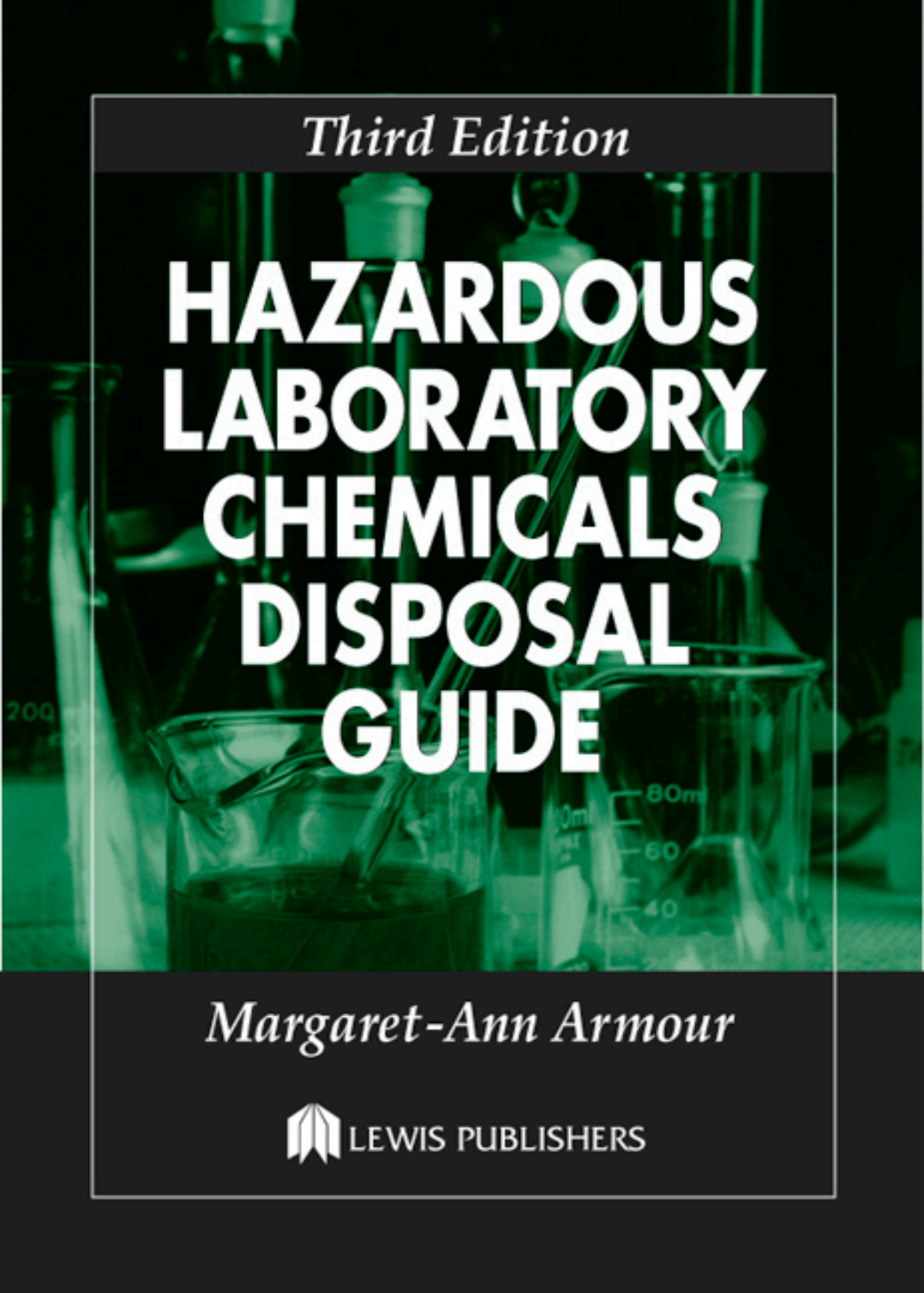

Third Edition

HAZARDOUS LABORATORY CHEMICALS DISPOSAL GUIDE

Margaret-Ann Armour

LEWIS PUBLISHERS

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PREFACE TO THE THIRD EDITION

The information contained in this guide has been compiled from sources believed to be reliable, accurate, and representative of the best opinion on the subject as of 2002. While every reasonable effort has been made to provide dependable information, I cannot assume responsibility for the validity or completeness of all chemical references given or for the consequences of their use.

The first three editions of this book were printed at the University of Alberta. In 1990, the book was published by Lewis Publishers/CRC Press. This is the third edition from the current publisher. The information contained in the first University of Alberta edition was compiled from published material. The second and third University of Alberta editions incorporated new or modified methods for spillage and waste disposal developed in my laboratory. The development and testing of procedures for disposal of small waste or surplus quantities of hazardous chemicals in my laboratory is ongoing. Thus, the previous CRC Press edition included 50 additional entries of chemicals that are animal carcinogens. This edition incorporates 15 chemicals used as pesticides.

I express my gratitude to two colleagues who collaborated in the initiation and early stages of this project. They are Dr. Lois Browne of the Department of Chemistry, University of Alberta, and Gordon Weir of Environmental Health and Safety at the University of Alberta. Without their help and support, this book would not have been possible. Rosemary Bacovsky, a pharmacist especially interested in the disposal of chemotherapeutic drugs, was instrumental in encouraging us to work in this area. The work of developing and testing the disposal methods was undertaken by a team of researchers who also contributed many excellent suggestions and discussions. They include Katherine Ayer, Yvgenia Briker, Hu-Mui Chang, John Crerar, Paul Cumming, William Hunter, Roger Klemm, Jonathan Konrad, Daria Kotovych, Asya Linetsky, Shiraz Merali, Carmen Miller, Carolyn Nelson, Sabina Qureshi, Patricia Rose, Gerard Spytkowski, and Richard Young. I am particularly grateful to Donna Ashick, now at the University of British Columbia, who was involved in the project for over 8 years, both in the practical aspects and in the preparation of the early editions of the guide. Much of the editing work for the second CRC Press edition was done by Sara Salmon; Dr. William Hunter has assisted with the updating of references for this edition. I thank the Chemistry Department at the University of Alberta for infrastructure support and many helpful discussions with its members.

The manuscript was originally entered into a database by Lu Ziola. Diane Dowhaniuk edited the previous editions. The changes to this edition have been entered by Annabelle Wiseman, and her efficiency and willingness to work long hours to meet deadlines are much appreciated. Dr. Kay Simpson, formerly of the Northern Alberta Institute of Technology, directed the checking of several of the waste disposal procedures.

The project has been funded by research grants from the Heritage Grant Program of Alberta Occupational Health and Safety, the Alberta Environmental Research Trust, and the Environmental Partners Program of Environment Canada. Funds have also been received from the Waste Management Branch of Alberta Environmental Protection, and summer students were supported by the Alberta and federal governments through their summer employment programs.

Finally, I have appreciated reactions from readers of the previous editions of this guide. Many of the suggestions have been incorporated into this edition. Comments are solicited on any aspect of the book, and suggestions for chemicals that might be included in future editions are welcome.

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INTRODUCTION

Laboratory workers generate waste chemicals. Increasingly during the past 15 years, it has been recognized that the ideal solution to the disposal of such wastes is to eliminate their generation at source. Much progress has been made and continues to be made in this direction. Chemical users are minimizing waste in several ways. These include scrutinizing procedures to determine whether chemical usage can be modified so that waste is reduced; recovering, recycling and reusing waste where possible; and, especially in academic teaching laboratories, reducing the scale of experiments.

However, the disposal of small quantities of a large variety of chemical wastes that are generated, for example, in school, college, and university laboratories, hospital pharmacies and research, and analytical and quality control laboratories, can be both difficult and expensive. Where the waste cannot be further reduced at the source, there remains the possibility of on-site conversion of at least some of the waste to nontoxic and environmentally acceptable products. This reduces the quantity of material that has to be transported off-site for disposal. On-site conversion has the added advantage that the worker who has been handling or using a chemical may also be the one eliminating or reducing the resulting waste. This worker has access to information about the chemical and would be expected to be knowledgeable in its safe handling. Sources of information about the properties of chemicals such as Material Safety Data Sheets are available in the workplace. However, to be able to convert waste and surplus chemicals to safe products, a detailed practical procedure that can be performed safely and with reproducible and reliable results is required. In school and college laboratories, it is often possible for such procedures to be the last step of an experiment so that students learn the importance of considering the waste generated by a process.

It must be noted that the disposal of any toxic or hazardous material must be in accord with current federal, provincial or state, and local regulations. On-site disposal may require licensing as a treatment center unless the procedure can be considered part of the reaction being performed at the bench.

The chemical treatment of wastes can result in the material being converted to products that are nontoxic and environmentally acceptable. The methods fall into several categories that include acid/base neutralization, oxidation or reduction, and precipitation of toxic ions as insoluble solids. Many of the disposal procedures suggested in this book fall into these categories.

Physical, chemical, and physiological properties, hazardous reactions, and spill and waste disposal procedures are listed for each chemical included in this handbook. The data about properties and hazardous reactions are intended to provide the user with sufficient information to handle the chemicals safely with appropriate precautions and personal protection.

Two important factors are included in the spill and waste disposal procedures. First, procedures are listed for individual chemicals. In this way, it is possible to detail precise conditions and exact quantities of reagents for each chemical to be destroyed. Second, the majority of the procedures were tested in the laboratory for safety to the operator, reliability, and to check that they met the desired criteria for conversion of the hazardous material to nonhazardous products. Thus, to be chosen as an acceptable disposal procedure, more than 99% of the starting material had to be destroyed under the conditions and length of time given. As far as possible, the products have been identified and, in these cases, equations for the reaction taking place are included. Where it was not possible to identify all of the fragments produced, and especially when the chemical to be

destroyed was a suspect carcinogen, the reaction mixture was submitted for an Ames test, which detects whether the material formed has excess mutagenicity over the background.

The disposal procedures presented are written for relatively small quantities. Laboratory testing has shown them to be safe on this scale. The methods should not be used for larger quantities unless a qualified chemist has shown that it is safe to increase the scale.

The book is intended to provide helpful information for bench workers to dispose of small amounts of waste and surplus chemicals at their bench, and for those charged with the responsibility for the management and disposal of hazardous waste. For both groups of readers, this book should facilitate complying with both legal and moral obligations in the routine treatment of waste, as well as allowing them to develop a plan of action for emergencies, such as spills of any of the chemicals listed.

INDEX OF COMPOUNDS

In the compound list, numbers in parentheses following the names of chemicals refer to hazard ratings as described in NFPA 704–11.

First Number — HEALTH HAZARD

4 Materials that on very short exposure could cause death or major residual injury.
3 Materials that on short exposure could cause serious temporary or residual injury.
2 Materials that on intense or continued but not chronic exposure could cause temporary incapacitation or possible residual injury.
1 Materials that on exposure would cause irritation but only minor residual injury.
0 Materials that on exposure under fire conditions would offer no hazard beyond that of ordinary combustible material.

Second Number — FLAMMABILITY

4 Materials that will rapidly or completely vaporize at atmospheric pressure and normal ambient temperature, or that are readily dispersed in air and that will burn readily.
3 Liquids and solids that can be ignited under almost all ambient temperature conditions.
2 Materials that must be moderately heated or exposed to relatively high ambient temperatures before ignition can occur.
1 Materials that must be preheated before ignition can occur.
0 Materials that will not burn.

Third Number — REACTIVITY/STABILITY

4 Materials that in themselves are readily capable of detonation or of explosive decomposition or reaction at normal temperatures and pressures.
3 Materials that in themselves are capable of detonation or explosive decomposition or reaction but require a strong initiating source or which must be heated under confinement before initiation or which react explosively with water.
2 Materials that readily undergo violent chemical change at elevated temperatures and pressures or which react violently with water or which may form explosive mixtures with water.
1 Materials that in themselves are normally stable, but which can become unstable at elevated temperatures and pressures.
0 Materials that in themselves are normally stable, even under fire exposure conditions, and which are not reactive with water.

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DEFINITIONS OF ABBREVIATIONS

TLV-TWA: Threshold Limit Value — Time-Weighted Average. These are the time-weighted average concentrations for a normal 8-hour workday and a 40-hour work week to which nearly all workers may be repeatedly exposed, day after day, without adverse effects.

TLV-STEL: Threshold Limit Value — Short-Term Exposure Limit. These are the concentrations to which workers can be exposed continuously for a short period of time without suffering (1) irritation, (2) chronic or irreversible tissue damage, or (3) narcosis of sufficient degree to increase the likelihood of accidental injury, or materially reduce work efficiency, and, provided that the daily TLV-TWA is not exceeded. A STEL is defined as a 15-minute TWA exposure that should not be exceeded at any time during a workday even if the 8-hour TWA is within the TLV-TWA. Exposure above the TLV-TWA up to the STEL should not be longer than 15 minutes and should not occur more than four times a day. There should be at least 60 minutes between successive exposures in this range.

TLV-STEL-C: Threshold Limit Value — Short-Term Exposure Limit — Ceiling. The concentration that should not be exceeded during any part of the working exposure.

LD_{50}: Lethal Dose to 50% of specified population.

REFERENCE ABBREVIATIONS

In the references section for each entry, the following abbreviations are used.

Abbreviation	Literature Reference
ACGIH	American Conference of Government Industrial Hygienists, 2002 Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices, ACGIH, Cincinnati, OH, 2002.
Agro. Desk Ref.	Montgomery, J.A., *The Agrochemicals Desk Reference*, Lewis Publishers, Boca Raton, FL, 1993.
Agro. Handbook	*The Agrochemicals Handbook*, 3rd ed., Royal Society of Chemistry Information Services, Cambridge, 1992.
Ald	Lenga, R.E., ed., *The Sigma-Aldrich Library of Chemical Safety Data*, 2nd ed., Aldrich Chemical Company, Milwaukee, WI, 1988.
Aldrich	2000–01 *Aldrich Handbook of Fine Chemicals and Laboratory Equipment*, Aldrich Chemical Company, Milwaukee, WI, 2001.
B	Bretherick, L., *Handbook of Reactive Chemical Hazards*, 4th ed., Butterworths-Heinnemann, London, 1990.
Beilstein	*Beilstein's Handbuch der Organischen Chemie*, Springer-Verlag, Berlin, 1928–present.
Cas 43	Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamines*, No. 43, IARC Scientific Publications, Lyon, 1982.
Cas 49	Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Polycyclic Aromatic Hydrocarbons,* No. 49, IARC Scientific Publications, Lyon, 1983.
Cas 54	Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Hydrazines*, No. 54, IARC Scientific Publications, Lyon, 1983.
Cas 64	Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Aromatic Amines and 4-Nitrobiphenyl*, No. 64, IARC Scientific Publications, Lyon, 1985.
CRC	Lide, David R., ed., *Handbook of Chemistry and Physics*, 75th ed., CRC Press, Boca Raton, FL, 1994–1995.
DOC	Buckingham, J., ed., *Dictionary of Organic Compounds*, 5th ed., Chapman & Hall, Cambridge, 1982.
F & F	Fieser, L.F. and Fieser, M.A., *Reagents for Organic Synthesis*, Wiley, New York.
Haz. Mat. Spills Tec.	Fingas, M., ed., *The Handbook of Hazardous Materials Spills Technology*, McGraw-Hill, New York, 2001.
IARC	International Agency for Research on Cancer Monographs on the Evaluation of the Carcinogenic Risk of Chemicals to Humans.
ITI	The International Technical Information Institute, *Toxic and Hazardous Industrial Chemicals Safety Manual for Handling and Disposal with Toxicity and Hazard Data*, ITI, Tokyo, 1981.
LSS	Lab Safety Supply, September 1994, General Safety Catalogue, Lab Safety Supply, P.O. Box 1368, Janesville, WI, 2002.

Lux	Luxon, S.G., ed., *Hazards in the Chemical Laboratory*, 5th ed., Royal Society of Chemistry, London, 1992.
Mellor	Mellor, J.W., *A Comprehensive Treatise on Inorganic and Theoretical Chemistry*, Longmans, London.
Merck	Budavari, S. et al., *The Merck Index*, 13th ed., Merck and Co., Whitehouse Station, NJ, 2001.
NFPA	National Fire Protection Association, *Fire Protection Guide on Hazardous Materials*, 12th ed., NFPA, Boston, MA, 1997.
NIEHS	Ninth Report on Carcinogens –– U.S. Department of Health and Human Services, Public Health Service. National Toxicology Program, Rockville, MD, 2001. Available at: http://ehp.nichs.nih.gov.roc/toc9.html.
PP	*Prudent Practices for Disposal of Chemicals from Laboratories*, National Research Council, National Academy Press, Washington, D.C., 1983.
PPL	*Prudent Practices in the Laboratory, Handling and Disposal of Chemicals*, National Academy Press, Washington, D.C., 1995.
Sax	Lewis, R.J., *Sax's Dangerous Properties of Industrial Materials*, Vols. I, II, and III, 8th ed., Van Nostrand Reinhold, New York, 1992
Sax5	Sax, N.I., *Dangerous Properties of Industrial Materials*, 5th ed., Van Nostrand Reinhold, New York, 1979.
Vogel	Vogel, A.I., *Vogel's Textbook of Practical Organic Chemistry*, 5th ed., revised by Furniss, B.S. et al., Addison-Wesley, 1989.

INCOMPATIBILITY TABLE

Compound/Class	Avoid Storage Near or Contact With
Acetic acid	Chromic acid, nitric acid, hydroxyl compounds, ethylene glycol, perchloric acid, peroxides, permanganates
Acetone	Conc. nitric acid and sulfuric acid mixtures
Acetylene	Fluorine, chlorine, bromine, copper, silver, mercury
Alkaline metals (e.g., Na, K, Mg, Ca, Al)	Carbon dioxide, carbon tetrachloride or other chlorinated hydrocarbons, halogens, water
Ammonia (anhydrous)	Mercury, chlorine, bromine, iodine, hydrofluoric acid, calcium hypochlorite
Ammonium nitrate	Acids, metal powders, flammable liquids, chlorates, nitrites, sulfur, finely divided organic or combustible materials
Aniline	Nitric acid, hydrogen peroxide
Arsenicals	Reducing agents (or will generate arsine)
Azides	Acids (or will generate hydrogen azide)
Bromine	Ammonia, acetylene, butadiene, methane, propane, butane (or other petroleum gases), hydrogen, sodium carbide, turpentine, benzene, finely divided metals
Calcium oxide	Water
Carbon, activated	Calcium hypochlorite, oxidizing agents
Chlorates	Ammonium salts, acids, metal powders, sulfur, finely divided organic or combustible materials
Chromic acid, chromium trioxide	Acetic acid, naphthalene, camphor, glycerol, turpentine, alcohol or other flammable liquids
Chlorine	Ammonia, acetylene, butadiene, methane, propane, butane (or other petroleum gases), hydrogen, sodium carbide, turpentine, benzene, finely divided metals
Chlorine dioxide	Ammonia, methane, phosphine, hydrogen sulfide
Copper	Acetylene, hydrogen peroxide
Cumene hydroperoxide	Organic or inorganic acids
Cyanides	Acids (or will generate hydrogen cyanide)
Flammable liquids	Ammonium nitrate, chromic acid, hydrogen peroxide, nitric acid, sodium peroxide, halogens
Fluorine	Isolate from everything
Hydrazine	Hydrogen peroxide, nitric acid, other oxidants
Hydrocarbons (propane, butane, benzene, gasoline, turpentine, etc.)	Fluorine, chlorine, bromine, chromic acid, sodium peroxide
Hydrocyanic acid	Nitric acid, alkalies
Hydrofluoric acid (anhydrous)	Ammonia (aqueous or anhydrous)
Hydrogen peroxide	Copper, chromium, iron, most other metals or their salts, alcohols, acetone, or other flammable liquids, aniline, nitromethane, or other organic or combustible materials
Hydrogen sulfide	Fuming nitric acid, oxidizing gases
Hypochlorites	Acids (or will generate chlorine or hypochlorous acid)

Iodine	Acetylene, ammonia (aqueous or anhydrous), hydrogen
Mercury	Acetylene, ammonia, fulminic acid (produced in nitric acid–ethanol mixtures)
Nitrates	Sulfuric acid (or will generate nitrogen dioxide)
Nitric acid (conc.)	Acetic acid, aniline, chromic acid, acetone, alcohol, or other flammable liquids, hydrocyanic acid, hydrogen sulfide, or other flammable gases, nitratable substances; copper, brass, or any heavy metals (or will generate nitrogen dioxide/nitrous fumes)
Nitrites	Acids (or will generate nitrous fumes)
Nitroparaffins	Inorganic bases, amines
Oxalic acid	Silver, mercury
Oxygen	Oils, grease, hydrogen, other flammable gases, liquids, or solids
Perchloric acid	Acetic acid, bismuth and its alloys, alcohol, paper, wood, grease oils
Peroxides (organic)	Organic or inorganic acids. Also: avoid friction and store cold
Phosphorus (white)	Air, oxygen, caustic alkalies as reducing agents (or will generate phosphine)
Potassium	Carbon tetrachloride, carbon dioxide, water
Potassium chlorate	Acids, especially sulfuric acid
Potassium permanganate	Glycerol, ethylene glycol, benzaldehyde, sulfuric acid
Selenides	Reducing agents (or will generate hydrogen selenide)
Silver	Acetylene, oxalic acid, tartaric acid, fulminic acid (produced in nitric acid–ethanol mixtures), ammonium compounds
Sodium	Carbon tetrachloride, carbon dioxide, water
Sodium nitrite	Ammonium nitrate and other ammonium salts
Sodium peroxide	Any oxidizable substance such as methanol, ethanol, glycerol, ethylene glycol, glacial acetic acid, acetic anhydride, benzaldehyde, furfural, methyl acetate, ethyl acetate, carbon disulfide
Sulfides	Acids (or will generate hydrogen sulfide)
Sulfuric acid	Light metals (lithium, sodium, potassium), chlorates, perchlorates, permanganates
Tellurides	Reducing agents (or will generate hydrogen telluride)

ACETIC ACID, GLACIAL CH_3COOH

CORROSIVE

Physical Properties

Clear, colorless liquid with pungent odor; contracts slightly on freezing; mp, 17°C; bp, 118°C.[1,2]

Fire Hazard

Flammable; flash point, 39°C; explosive limits, 5.4–16%; auto-ignition temperature, 426°C.[2] Extinguish fire with water spray, dry powder, carbon dioxide, or foam.[2]

Chemical Properties

Excellent solvent for many organic compounds; dissolves phosphorus, sulfur, and halogen acids; miscible with water, alcohol, glycerol, ether, and carbon tetrachloride; practically insoluble in carbon disulfide.[1]

Hazardous Reactions

Acetaldehyde. Exothermic polymerization initiated by acetic acid.[3]

Acetic Anhydride and Water. Violent reaction occurred when aqueous acetic acid added to tank of the anhydride.[4]

Ammonium Nitrate. When concentrated in acetic acid, the resulting mixture ignites on warming.[5]

5-Azidotetrazole and Acetone. Mixture explodes if traces of acetic acid are present.[6]

Carbonates. Highly reactive mixture results when added to acetic acid.[1]

Chlorine Trifluoride. Reaction with acetic acid is very violent and sometimes explosive.[7]

Diallyl Methyl Carbinol and Ozone. Explosion has resulted when acetic acid added to this reaction mixture.[8]

Hydrogen Peroxide and Organic Materials. Reactions in acetic acid have resulted in formation of explosive mixtures.[9]

Hydroxide. Addition of acetic acid to a vessel contaminated with potassium hydroxide caused eruption of the acid.[10]

Nitric Acid and Sodium Hexahydroxyplatinate(IV). Reaction in nitric acid–acetic acid solvent mixture produced an explosive solid.[11]

Oxides. Many are incompatible with acetic acid.[1]

Perchloric Acid, Acetic Anhydride, and Organic Materials. Anhydrous perchloric acid is formed in the presence of acetic anhydride; the mixture is heat and shock sensitive and may explode violently in the presence of organic material.[12]

Phosphates. Many are incompatible with acetic acid.

Phosphorus Isocyanate. Reacts violently with acetic acid.[13]

Potassium *tert*-Butoxide. Can react violently with acetic acid.[14]

p-Xylene. Oxidation with acetic acid can result in an explosive mixture if experimental conditions are not carefully controlled.[15]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and the eyes. The liquid burns skin; swallowing results in internal irritation and damage. Chronic exposure may cause erosion of dental enamel, bronchitis,

and eye irritation.[1] TLV-TWA 10 ppm (25 mg/m^3); TLV-STEL 15 ppm (37 mg/m^3).[16] LD_{50} (rat, oral) 3.53 g/kg.[1]

Spillage Disposal

Wear neoprene gloves,[17] laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a beaker or pail. In the fume hood, slowly add the acid mixture to a pail of cold water. When reaction ceases, neutralize with more sodium carbonate if necessary. When solids have settled, decant liquid into drain with 50 times its volume of water. Discard solid residue with normal refuse. Ventilate site of spillage well to evaporate remaining liquid and dispel vapor.[18,19]

Waste Disposal

Package Lots. Label for recycling or follow disposal of small quantities.

Small Quantities. Wear neoprene gloves, laboratory coat and eye protection. Work in the fume hood. Add slowly to large volume of cold water in a plastic pail. Neutralize with 5% sodium hydroxide solution or sodium carbonate and pour into the drain.[18]

Reactions for Spillage and Waste Disposal

$$CH_3COOH + NaOH \xrightarrow{H_2O} \underset{\text{sodium acetate}}{CH_3COONa} + H_2O$$

$$2CH_3COOH + Na_2CO_3 \xrightarrow{H_2O} \underset{\text{sodium acetate}}{CH_3COONa} + H_2O + CO_2$$

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ACETIC ANHYDRIDE $(CH_3CO)_2O$

FLAMMABLE, CAUSES BURNS

Physical Properties

Colorless liquid with strong acrid odor; bp, 139°C.[1]

Fire Hazard

Flammable. Flash point, 52°C; explosive limits, 3–10%; ignition temperature, 332°C. Extinguish fire with water spray, dry chemical foam, or carbon dioxide.[2]

Chemical Properties

Reacts slowly with water to form acetic acid.[1]

Hazardous Reactions

Boric Acid. Heating a mixture to 60°C may result in explosion.[3,4]

Chromic Acid. Strongly exothermic reaction occurs when the anhydride is added to an aqueous solution of chromium trioxide.[5,6]

Glycerol and Phosphoryl Chloride. Violent acylation of glycerol occurs using phosphoryl chloride as catalyst in the absence of solvent.[7]

Hydrogen Peroxide. The mixture must be kept acid and an excess of the anhydride avoided to prevent the formation of highly explosive diacetyl peroxide.[8]

Hypochlorous Acid. Mixture may explode violently.[9]

Metal Nitrates. Nitrating agents composed of the anhydride and copper and sodium nitrates cause violent reactions.[10] Improved procedures have been published.[11,12]

Nitric Acid. Mixtures are detonatable.[13]

Perchloric Acid and Water. The anhydride is used to prepare anhydrous perchloric acid. It must be added slowly to the acid at about 10°C.[14,15]

Potassium Permanganate. Contact of the anhydride with potassium permanganate may cause explosion.[16]

Water. Addition of water can cause vigorous acid-catalyzed hydrolysis and explosive boiling.[17,18]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and the eyes. The liquid burns the eyes severely and irritates and burns skin, causing blistering. Swallowing the liquid causes severe pain and vomiting. Avoid breathing vapor. Prevent contact with eyes and skin.[19] TLV-TWA 5 ppm (21 mg/m^3).[20] LD_{50} (rat, oral) 1.78 g/kg.

Spillage Disposal

Shut off all possible sources of ignition. Wear nitrile rubber gloves, laboratory coat, and eye protection. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the acetic anhydride has been absorbed, scoop into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate.

Decant the solution to the drain with water. Treat the solid residue as normal refuse. The spillage site should be washed thoroughly with soap and water.[19–22]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[23]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To decompose 5 mL (5.4 g) of acetic anhydride, place 60 mL of a 2.5 M sodium hydroxide solution (prepared by dissolving 6.0 g of NaOH in 60 mL of water) in a 250-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Add the acetic anhydride to the dropping funnel and run it dropwise into stirred solution at such a rate that the temperature does not rise above 35°C. Allow to stir at room temperature overnight. Neutralize solution to pH 7 with 2 M hydrochloric acid (slowly add 16 mL of concentrated acid to 80 mL of cold water) and pour into the drain.[23]

Reactions for Spillage and Waste Disposal

$$(CH_3CO)_2O + Na_2CO_3 \xrightarrow{H_2O} \underset{\text{sodium acetate}}{2CH_3COONa} + CO_2$$

$$(CH_3CO)_2O + 2NaOH \xrightarrow{H_2O} \underset{\text{sodium acetate}}{CH_3COONa} + H_2O$$

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22. *Haz. Mat. Spills Tec.*, 29.5.
23. PPL 161.

ACETONE C_3H_6O

HIGHLY FLAMMABLE

Synonyms

2-Propanone, dimethyl ketone, β-keto-propane, pyroacetic ether.[1]

Physical Properties

Volatile, highly flammable liquid; characteristic odor; pungent, sweetish taste; bp, 56.5°C, mp, –94°C.[1]

Fire Hazard

Highly flammable. Flash point, closed cup, 0°F (–18°C).[1] Explosive limits, 2.6–12.8%; auto-ignition temperature, 538°C. Extinguish fire with dry powder, carbon dioxide, or alcohol-resistant foam.[2]

Chemical Properties

Miscible with water, alcohol, dimethylformamide, chloroform, ether, and most oils. Keep away from fire. Keep away from plastic eye-glass frames, jewelry, pens and pencils, rayon stockings, and other rayon garments.[1]

Hazardous Reactions

Vigorously oxidized by air in the presence of several inorganic oxidants.[2]

Bromoform, Potassium Hydroxide. Interaction in presence of powdered potassium hydroxide (or other bases) is violently exothermic,[3] even in the presence of diluting solvent.[4]

Carbon, Air. Recovering acetone from the air, in plants, with active carbon resulted in fires due to the air flow being too low to cool effectively.[5]

Chloroform, Alkali. Vigorous exothermic reaction with chloroform in the presence of solid potassium hydroxide or calcium hydroxide.[6]

2-Methyl-1,3-butadiene (Isoprene). Isoprene-acetone mixtures may form peroxides.[7]

Nitric Acid, Sulfuric Acid. Acetone is oxidized by the mixed acids with explosive violence.[8,9]

Potassium *tert*-Butoxide. Contact with 1.5-g portion of the solid butoxide with the vapor of acetone caused ignition after 4 minutes.[1]

Sulfur Dichloride. Care is necessary when cleaning traces of sulfur dichloride with acetone; the reaction is vigorous if more than a trace is present.[11]

Thiotrithiazyl Perchlorate. The precipitated perchlorate salt explodes on washing with acetone.[12]

Physiological Properties and Health Hazards

Prolonged or repeated topical use may cause dryness and inflammation. Inhalation may produce headache, fatigue, excitement, bronchial irritation, and, in large amounts, narcosis. TLV-STEL 750 ppm. Serious poisoning is rare.[1,2] LD_{50} (rat, oral) 10.7 mL/kg.[1] TLV-TWA 500 ppm.[13]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all of the liquid has been absorbed, scoop the mixture into a plastic pail or other wide-mouth container and place in the fume hood. If local regulations permit, allow the acetone vapor to

evaporate for several days until no odor remains. The spill mix can then be discarded with normal refuse.[14,15] Otherwise, package and label for disposal by burning.[15,16]

Waste Disposal

Distill for reuse or package and label for disposal by burning.[15]

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2-ACETYLAMINOFLUORENE $C_{15}H_{13}NO$

ANIMAL CARCINOGEN

Synonyms

N-2-Fluoroenylacetamide, N-9H-fluoren-2-ylacetamide.

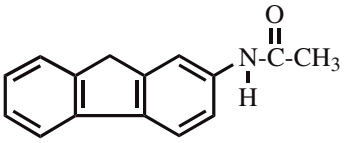
Physical Properties

Powder; mp, 194°C (dec).[1,2]

Chemical Properties

Powder; mp, 194°C (dec).[1,2] Insoluble in water; soluble in alcohols, glycols, and fat solvents.[2]

Structure



Physiological Properties and Health Hazards

Animal carcinogen. Oral or parenteral administration to rodents causes tumors in the liver, mammary gland, urinary bladder, intestinal epithelium, and sebaceous glands of the ear duct. Mutagenic and slightly toxic. Reasonably anticipated to be a human carcinogen.[3]

Spillage Disposal

Wear a dust mask, goggles, rubber gloves, and protective clothing. Carefully scoop the solid into a beaker and transport to the fume hood. Destroy the material using waste disposal procedure. Wash the site of the spill with soap and water, disposing of the cleaning materials in a sealed bag or container labeled for disposal by burning.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. The compound should be mixed with vermiculite and dry caustics, wrapped in paper, and burned in a chemical incinerator equipped with afterburner and scrubber.

Small Quantities. Wear goggles and protective gloves and clothing. Work in the fume hood. To 0.25 g of 2-acetylaminofluorene in a 50-mL round-bottom flask add 10 mL of concentrated hydrochloric acid. Fit the flask with a condenser and heat under reflux for at least 10 hours when all trace of yellow should have disappeared. Cool the contents of the flask to 0°C in an ice-salt bath and, over a period of 5 minutes, add dropwise a solution of 0.13 g of sodium nitrite in 0.3 mL of water. Stir the mixture for 30 minutes, then slowly add 2.7 mL of ice-cold 50% hypophosphorus acid. After stirring at room temperature for 16 hours, filter the mixture, wash the filtrate into the drain with water, and discard the solid (fluorene) with normal refuse or package and label for disposal by incineration.[4–6]

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ACETYL BROMIDE CH_3COBr

CAUSES BURNS

Physical Properties

Colorless to yellow fuming liquid; bp, 76°C.[1]

Chemical Properties

Violently decomposed by alcohol and by water with formation of hydrobromic acid and acetic acid. Miscible with ether, chloroform, and benzene.[1,2]

Hazardous Reactions

Alcohols and Water. Acetyl bromide reacts violently with alcohols or water.[1]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and eyes. The liquid burns skin and eyes. Swallowing causes severe internal irritation and damage. Avoid breathing vapor. Prevent contact with skin and eyes.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the acetyl bromide has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain, flushing with at least 50 times its volume of water. Treat the solid residue as normal refuse.[3–5] Wash the area of the spill thoroughly with soap and water.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve in a flammable solvent and burn in a furnace with a scrubber.[3]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To decompose 10 mL (16.6 g) of acetyl bromide, place 160 mL of a 2.5 M sodium hydroxide solution (prepared by dissolving 16 g of NaOH in 160 mL of water) in a 250-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Add the acetyl bromide to the dropping funnel and run it dropwise into the stirred solution at such a rate that the temperature does not rise above 35°C. Continue to stir at room temperature overnight. Neutralize the solution to pH 7 with 2 M hydrochloric acid (prepared by cautiously adding 40 mL of concentrated acid to 200 mL of cold water) and wash into the drain with at least 50 times its volume of water.[6]

To decompose 1–2 mL of acetyl bromide, in the fume hood pour the liquid into an evaporating dish. Cover the acetyl bromide with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test pH of the solution and neutralize if necessary with 2 M hydrochloric acid. Pour the solution into the drain with at least 50 times its volume of water. Discard any solid with normal refuse.

Reactions for Spillage and Waste Disposal

$$CH_3COBr + Na_2CO_3 \xrightarrow{H_2O} CH_3COONa + NaBr + CO_2$$

sodium acetate

$$CH_3COBr + 2NaOH \xrightarrow{H_2O} CH_3COONa + NaBr + H_2O$$

sodium acetate

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2. Lux 195.
3 ITI 009.
4 Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
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6. PPL 161.

ACETYL CHLORIDE CH_3COCl

HIGHLY FLAMMABLE, CAUSES BURNS

Physical Properties

Colorless, fuming, volatile liquid with a pungent odor; bp, 52°C.[1]

Fire Hazard

Flash point, 4°C; ignition temperature, 390°C. Extinguish fire with dry chemical or carbon dioxide.[2]

Chemical Properties

Violently decomposed by alcohol and rapidly decomposed by water with formation of hydrochloric acid and acetic acid. Miscible with benzene, chloroform, ether, glacial acetic acid, and petroleum ether.[1–3]

Hazardous Reactions

Dimethyl Sulfoxide. Dimethyl sulfoxide decomposes violently on contact with acetyl chloride.[4]

Ethyl Alcohol. Acetyl chloride reacts violently with ethyl alcohol.[5]

Phosphorus Trichloride with Acetic Acid. Several laboratory explosions have occurred using this reaction to form acetyl chloride. Poor heat control probably caused formation of phosphine.[6]

Water. Acetyl chloride reacts violently with water.[2]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and eyes. The liquid burns skin and eyes. Swallowing results in severe internal irritation and damage. Avoid breathing vapor. Prevent contact with eyes and skin.[3]

Spillage Disposal

Shut off all possible sources of ignition. Wear nitrile rubber gloves, laboratory coat, and eye protection. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the acetyl chloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain, flushing with at least 50 times its volume of water. Treat the solid residue as normal refuse. Wash spill area thoroughly with soap and water.[3,7,9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve in a flammable solvent and burn in a furnace with a scrubber.[8]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To decompose 10 mL (11 g) of acetyl chloride, place 160 mL of a 2.5 M sodium hydroxide solution (prepared by dissolving 16 g of NaOH in 160 mL of water) in a 250 mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Add the acetyl chloride to the dropping funnel and run it dropwise into the stirred solution at such a rate

that the temperature does not rise above 35°C. Continue to stir at room temperature overnight. Neutralize the solution to pH 7 with 2 M hydrochloric acid (prepared by cautiously adding 40 mL of concentrated acid to 200 mL of cold water) and wash into the drain with at least 50 times its volume of water.[10]

To decompose 1–2 mL of acetyl chloride, in the fume hood pour the liquid into a large evaporating dish. Cover the acetyl chloride with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test pH of the solution and neutralize if necessary. Pour the solution into the drain, flushing with at least 50 times its volume of water.[7]

Reactions for Spillage and Waste Disposal

$$CH_3COCl + Na_2CO_3 \xrightarrow{H_2O} CH_3COONa + NaCl + CO_2$$

sodium acetate

$$CH_3COCl + 2NaOH \xrightarrow{H_2O} CH_3COONa + NaCl + H_3O$$

sodium acetate

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8. ITI 010.
9. *Haz. Mat. Spills Tec.*, 29.5.
10. PPL 161.

ACETYLENE C_2H_2

TOXIC WHEN INHALED, EXPLOSIVE

Physical Properties

Gas; not unpleasant odor unless impure (due to phosphine); sublimes[1]; bp, –83°C.[2]

Fire Hazard

Highly flammable and explosive. Explosive limits, 3–82%; ignition temperature, 335°C.[2] Extinguish fire with water spray, dry chemical foam, or carbon dioxide.[3]

Chemical Properties

Burns brilliantly in air with sooty flame; soluble in water, ether, benzene, glacial acetic acid, acetone, and alcohol.[1]

Hazardous Reactions

Endothermic gas may explosively decompose alone or mixed with air.[2]

Air. Mixture with air containing between 3–82% gas is explosive.[2]

Bleaching Powder. Contact with acetylene may lead to formation of explosive chloroacetylenes.[4]

Cobalt. Finely divided metal decomposes and polymerizes acetylene on contact, becoming incandescent.[5]

Copper. Metal forms explosive compounds with acetylene.[1]

Copper Acetylides. If warmed in air or oxygen, or on impact, the copper acetylides may explode in subsequent contact with acetylene.[6]

Halogens. Interaction with acetylene can be violent or explosive.[7]

Nitric Acid and Mercury. Contact of acetylene with concentrated nitric acid in presence of mercury forms explosive trinitromethane or tetranitromethane if sulfuric acid is subsequently added.[8]

Nitric Oxide Products. Mixture with acetylene will ignite at 30–50°C.[9]

Oxygen. Mixtures with acetylene are very explosive even at very low oxygen concentrations (below 2.5%).[10]

Potassium. The molten metal ignites in acetylene and then explodes.[11]

Silver. Metal forms explosive compounds with acetylene.[1]

Trifluoromethyl Hypofluorite. Interaction with acetylene, in absence of nitrogen as diluent, is explosive on mixing.[12]

Physiological Properties and Health Hazards

Inhalation may cause dizziness, headache, and nausea; mixed with oxygen, it can have narcotic properties, but it is primarily an asphyxiant. Avoid breathing gas.[2]

Spillage Disposal

See waste disposal.

Waste Disposal

Gas Leakage. By forced ventilation, maintain concentration of gas below the range of explosive mixture. Move the tank or cylinder to an open area. Leave to bleed off in the atmosphere.[12]

Disposal. Fit a pipe line into a furnace or a pit and burn with care.[12]

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13. ITI 011.

ACROLEIN CH_2=CH-CHO

STORE UNDER REFRIGERATION, EXTREMELY FLAMMABLE, GIVES OFF POISONOUS VAPOR

Synonyms

Acraldehyde, acryl aldehyde, propenal.

Physical Properties

Colorless volatile liquid with pungent, choking odor; bp, 52.5°C.[1]

Fire Hazard

Flash point, –26°C; explosive limits, 3–31%; ignition temperature, 235°C. Extinguish fire with dry chemical, foam, or carbon dioxide.[2]

Chemical Properties

Somewhat soluble in water; soluble in alcohol and ether.[1]

Hazardous Reactions

Acids or Bases. Liable to polymerize violently, especially in contact with strong acidic or basic catalysts. Hydroquinone is added as stabilizer but may lose its effectiveness after a short storage time.[3]

Physiological Properties and Health Hazards

The vapor irritates all parts of the respiratory system and may cause unconsciousness. Short exposure may cause pain to the nose and eyes in addition to intense irritation. Poisoning can occur by absorption of acrolein through the skin. Swallowing would cause severe poisoning.[4] Avoid breathing vapor. Prevent contact with eyes and skin. TLV-STEL-C 0.1 ppm (0.23 mg/m^3).[5] LD_{50} (rat, oral) 0.046 g/kg.[1]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[6] When the acrolein has been absorbed, scoop the mixture into a plastic container and either package for disposal by burning[4] or estimate the volume of acrolein spilled, and for each 7 mL, add a solution of 63 g of potassium permanganate in 700 mL of water. Allow it to stand overnight, then neutralize the solution with 3 M sulfuric acid (prepared by cautiously adding 16 mL of concentrated acid to 86 mL of cold water). Slowly add solid sodium bisulfite to the mixture, while stirring, until the solution is colorless. Decant the solution into the drain with water and discard the solid as normal refuse.[7,8] Ventilate area well to evaporate remaining acrolein and dispel vapor.[4–6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner.[7]

Small Quantities. Work in the fume hood. Wear eye protection, nitrile rubber gloves and laboratory coat. Place 6.0 g (7 mL, 0.107 mol) of acrolein in a 1-L, three-necked, round-bottom flask equipped with a thermometer, stirrer, and dropping funnel. Over a period of 10 minutes, add 50 mL of a solution of 63 g (0.4 mol) of potassium permanganate in 700 mL of water. If the purple color is not discharged, warm the mixture on steam bath until it becomes brown. Add the remainder of the permanganate solution at such a rate that the temperature does not exceed 45°C. When addition is complete, heat the mixture on the steam bath to 70–80°C, while stirring, for 1 hour. Cool the mixture to room temperature, and acidify to pH 1 with 3 M sulfuric acid (16 mL of concentrated acid cautiously added to 84 mL of cold water). Add solid sodium bisulfite, while stirring, until a colorless solution is produced. Wash the solution into the drain with water.[7]

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2. NFPA 49.
3. B 358.
4. Lux 197.
5. ACGIH 13.
6. Armour, M.A., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
7. PPL 162.
8. *Haz. Mat. Spills Tec.*, 29.5.

ACRYLIC ACID CH_2=CHCOOH

FLAMMABLE, CAUSES BURNS

Physical Properties

Colorless solid or liquid with acrid odor; mp, 14°C; bp, 141°C.[3]

Fire Hazard

Flash point, 50°C; ignition temperature, 438°C. Flammable limits, 2–8%. Extinguish fire with water spray, dry powder, carbon dioxide or "alcohol-resistant" foam.[2]

Chemical Properties

Miscible with water, alcohol, and ether. Polymerizes.[1]

Hazardous Reactions

Uninhibited acid polymerizes exothermally at ambient temperature, and a violent reaction or explosion may occur.[3,4]

Physiological Properties and Health Hazards

Liquid or strong solutions severely irritate the skin, eyes, and respiratory system. Swallowing causes extreme irritation. Avoid breathing vapor. Prevent contact with skin and eyes.[4] TLV-TWA 2 ppm (5.9 mg/m^3).[5] LD_{50} (rat, oral) 2.59 g/kg.[1]

Spillage Disposal

Shut off possible sources of ignition. Wear nitrile rubber gloves, laboratory coat, and eye protection. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. In the fume hood, transfer to an appropriately labeled container for disposal by burning.[6–10] The area of the spill should be washed thoroughly with soap and water.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner.[7,8]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve the acrylic acid in water to give a 10% solution. For each 5 mL of this solution, add 250 mL of 2% aqueous sodium hydroxide and, while stirring, 10 g of potassium permanganate. Stir at room temperature for 48 hours. Add solid sodium bisulfite until the solution is colorless, neutralize with 5% hydrochloric acid, and pour the liquid into the drain. Discard any small amount of brown solid (manganese dioxide) with regular refuse.[7]

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3. B 359.
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7. Armour, M.A. and Linetsky, A., *A Laboratory Disposal Method for Acrylic Acid*, University of Alberta, Edmonton, 1999.
8. ITI 016.
9. PPL 161.
10. *Haz. Mat. Spills Tec.*, 29.5.

ACRYLONITRILE CH_2=CHCN

SUSPECTED HUMAN CARCINOGEN, HIGHLY FLAMMABLE

Synonyms

Vinyl cyanide.

Physical Properties

Colorless volatile liquid with ethereal odor; bp, 77.3°C.[1]

Fire Hazard

Vapor forms explosive mixtures with air. Flammable limits, 3–17%. Flash point, 0°C. Ignition temperature, 481°C. Dilute solutions are also hazardous. Extinguish fires with water spray, dry chemical, foam, or carbon dioxide.[2]

Chemical Properties

One part dissolves in about 15 parts water at 25°C.[3] Miscible with most organic solvents.[1]

Hazardous Reactions

Tends to polymerize violently, so it must never be stored uninhibited, in light or close to bases.

Acids. Vigorous reaction with sulfuric or nitric acids. Small quantities of acid may neutralize the inhibitor (aqueous ammonia).[4,5]

Bases. Violent polymerization in contact with bases.[4]

Bromine. Violent polymerization in the presence of bromine, possibly due to catalysis by hydrogen bromide.[4]

Initiators. Free radical polymerization initiated by azoisobutyronitrile, benzoyl peroxide, or di-*tert*-butyl peroxide may be explosive.[4]

Silver Nitrate. May polymerize violently in the presence of solid silver nitrate.[6]

Physiological Properties and Health Hazards

Vapor may cause dizziness, nausea, and unconsciousness. Contact of liquid with skin may cause dermatitis or severe burns. Swallowing or absorption through skin leads to poisoning. Prolonged breathing of vapor may result in jaundice. Human carcinogen. Prevent inhalation of vapor. Prevent contact with skin and eyes.[3] TLV-TWA 2 ppm (4.3 mg/m^3) (skin).[7] LD_{50} (oral, rat) 0.093 g/kg.[1]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the acrylonitrile has been absorbed, scoop the mixture into a plastic container and package for disposal by burning. Ventilate area well to evaporate remaining acrylonitrile and dispel vapor.[3,8–10]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[9]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 25 mL of concentrated hydrochloric acid in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. Heat the solution to gentle reflux and add the acrylonitrile (5.3 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring (to prevent bumping) for 5–10 hours. Cool, dilute the reaction mixture with water, and wash down the drain with at least 50 times its volume of water.[9]

Reactions for Spillage and Waste Disposal

$$CH_2=CHCN + H^+ + 2H_2O \rightarrow \underset{\text{acrylic acid}}{CH_2=CHCOOH} + NH_4^+$$

REFERENCES

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2. NFPA 49.
3. Lux 199.
4. B 348.
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6. B 350.
7. ACGIH 13.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
9. PP 61, 68; PPL 252–253.
10. *Haz. Mat. Spills Tec.*, 29.5.

AFLATOXINS B_1, B_2, G_1, G_2, M_1, M_2

HIGHLY TOXIC, HUMAN CARCINOGENS

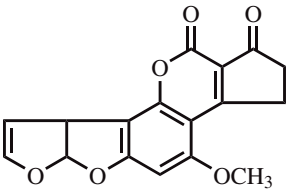
Physical Properties

All compounds are crystalline, colorless to pale yellow solids; melting point range, 237–299°C; fluorescent to UV light.[1–3]

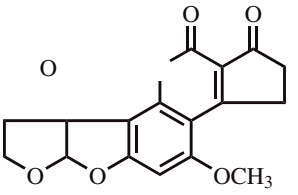
Chemical Properties

Slightly soluble in water. Insoluble in nonpolar solvents. Relatively unstable to light and air, particularly in highly polar solvents.[1,3]

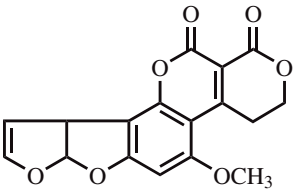
Structure



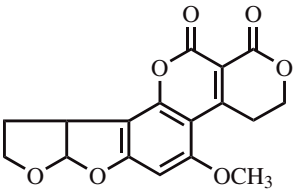
B_1



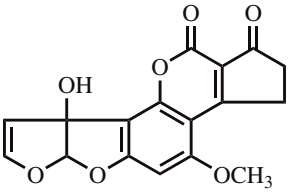
B_2



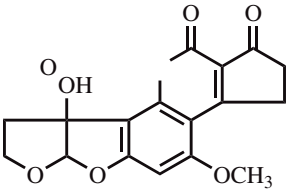
G_1



G_2



M_1



M_2

Mode of Action

The extreme toxicity of aflatoxins is thought to be due to the inhibition of nucleic acid synthesis.[2]

Physiological Properties and Health Hazards

B_1 and G_1 are the most highly toxic and are animal carcinogens. B_1 is teratogenic in rodents. In countries where food contains aflatoxins, there is an increased incidence of liver cancer. Listed as known human carcinogens.[3]

Spillage Disposal

Wear protective gloves, laboratory coat, and goggles. If a solution is spilled, absorb on tissue. If solid is spilled, as far as possible, scoop in a container and then rinse the area of the spill with methanol and absorb the methanol onto tissues. Place the tissues in the container with the solid and add sufficient household bleach (5% sodium hypochlorite solution) to cover. Allow to stand at room temperature for at least 2 hours. Add an equal volume of 5% aqueous acetone. Allow to react for 30 minutes and then wash into the drain with water.[4]

Waste Disposal

Package Lots. Place in a separate, labeled container for disposal by burning. Dissolve or mix the material into a combustible solvent and burn in furnace equipped with afterburner and scrubber.

Small Quantities. Wear protective gloves, laboratory coat, and goggles. Work in the fume hood. If the aflatoxin is dissolved in an organic solvent other than methanol, remove the solvent under reduced pressure. Dissolve the residue in methanol and, for each microgram of aflatoxin, add 3 mL of household bleach (5% sodium hypochlorite solution). Shake the mixture and allow to stand at room temperature for at least 2 hours. Dilute the solution with five times its volume of water and then add acetone equal to one-twentieth of the total volume. Allow to react for 30 minutes. Wash the solution into the drain with water.[4]

REFERENCES

1. IARC 1, 145.
2. Merck 182.
3. NIEHS III–1.
4. Castegnaro, M. et al., *Laboratory Decontamination and Destruction: B1, B2, G1, G2 in Laboratory Wastes*, No. 37, IARC Scientific Publications, Lyon, 1980, p. 35.

ALDICARB $C_7H_{14}N_2O_2S$

SYSTEMIC INSECTICIDE, ACARICIDE, NEMATOCIDE

Synonyms

2-Methyl-2-(methylthio)-propionaldehyde O-(methylcarbamoyl)oxime, Temik, UC21149, OMS 771.

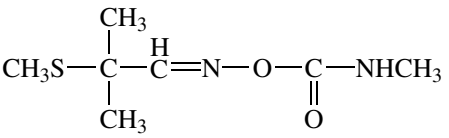
Physical Properties

White crystalline solid with a slightly sulfurous odor; mp, 99–101°C.[1,2]

Chemical Properties

Solubility at 25°C (w/w) in water 0.6%; acetone 35%, benzene 15%, xylene 5%; methylene chloride 30%.[3] Noncorrosive to common metals and plastics.

Structure



Aldicarb

Physiological Properties and Health Hazards

LD_{50} 0.93 mg/kg (oral, rat), above 5 mg/kg (dermal, rabbit).[2] LC_{50} (rainbow trout, 96 hours) 0.88 mg/L.[3]

Waste Disposal

Wear eye protection and gloves. To each 1 g of aldicarb, add 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid added to 83 mL of cold water) and 4.7 g of potassium permanganate. The mixture is stirred at room temperature for 30 minutes. Add a saturated solution of sodium bisulfite until the solution is colorless. The solution is neutralized by the careful addition of soda ash (foaming may occur) or 5% aqueous sodium hydroxide. Wash the clear solution into the drain with water.[4]

REFERENCES

1. Merck 330.
2. Agro. Handbook, A0005.
3. Agro. Desk Ref., 10.
4. Armour, M.A. and Briker, Y., *A Laboratory Disposal Method for Aldicarb*, University of Alberta, Edmonton, 2000.

ALUMINUM BROMIDE ANHYDROUS $AlBr_3$

CAUSES BURNS, REACTS VIOLENTLY WITH WATER

Physical Properties

Colorless to yellowish-red solid; mp, 97°C.[1]

Fire Hazard

Extinguish fire with carbon dioxide, dry chemical, or foam. Do not use water.[2]

Chemical Properties

Violently decomposed by water with evolution of hydrogen bromide.[2] Soluble in benzene, toluene xylene, nitrobenzene, and hydrocarbon solvents.[1]

Hazardous Reactions

Water. Hydrolysis is very violent.[3]

Physiological Properties and Health Hazards

Dust irritates mucous membranes and causes painful eye burns. Interaction with moisture on the skin results in heat and acid burns. Swallowing causes severe burns. Avoid breathing dust. Prevent contact with skin and eyes.[2] TLV-TWA 2 mg(Al)/m^3.[4]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain with at least 50 times its volume of water. Treat the solid residue as normal refuse.[5–7] The area of the spill should be washed thoroughly with soap and water.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, place the aluminum bromide in a large evaporating dish. Cover the aluminum bromide with excess solid sodium carbonate or calcium carbonate. Very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test pH of the solution and neutralize if necessary. Pour the solution into the drain, flushing with at least 50 times its volume of water. Treat the solid residue as normal refuse.[5,6]

Reactions for Spillage and Waste Disposal

$$2AlBr_3 + 3H_2O + 3Na_2CO_3 \rightarrow \underset{\substack{\text{aluminum}\\ \text{hydroxide}\\ \text{(insoluble)}}}{2Al(OH)_3} + 6NaBr + 3CO_2$$

REFERENCES

1. Merck 330.
2. Lux 204.
3. Nicholson, D.G. et al., *Inorg. Synth.*, 3, 35, 1950.
4. ACGIH 14.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
6. PPL 165.
7. Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 24, 1999.

ALUMINUM CHLORIDE ANHYDROUS $AlCl_3$

CAUSES BURNS, REACTS VIOLENTLY WITH WATER

Physical Properties

Yellow or off-white pieces, granules, or powder; volatilizes without melting, fumes in air.[1]

Fire Hazard

Extinguish using dry chemical, carbon dioxide, or foam. Do not use water.[2]

Chemical Properties

Violently decomposed by water, with the formation of hydrogen chloride.[2] Soluble in alcohol, ether, benzene, carbon tetrachloride, and chloroform.[1]

Hazardous Reactions

Spontaneous decomposition may occur on long storage and can result in explosion when opening the container.[3]

Alkenes. Highly exothermic polymerization of alkenes, such as isobutene over aluminum chloride, may become uncontrollable.[3]

Benzoyl Chloride and Naphthalene. A violent reaction occurs if aluminum chloride is added below the melting point of the mixture.[4]

Ethylene Oxide. Polymerizes violently in the presence of aluminum chloride.[5]

Lewis Acids. In the absence of a diluent (carbon disulfide), aluminum trichloride explodes violently with sulfuric acid and Lewis acids.[6]

Nitrobenzene. Mixtures react violently at 120°C, possibly due to the formation and polymerization of *o*- and *p*-chloronitrosobenzenes.[7]

Nitrobenzene and Phenol. Mixtures react violently at 120°C.[8]

Nitromethane and Ethylene. Mixtures of ethylene, aluminum chloride, and nitromethane may explode at 75°C.[9]

Nitromethane, Phenol, and Carbon Monoxide. Heating a mixture of nitromethane, aluminum chloride, phenol, and carbon monoxide at 110°C in an autoclave led to an explosion.[10]

Sodium Borohydride. Contact of borohydride solution with solid aluminum chloride can lead to ignition of hydrogen.[11]

Water. Violent reaction with water.[12]

Physiological Properties and Health Hazards

Dust irritates mucous membranes and causes painful eye burns. Interaction with moisture on the skin results in heat and acid burns. Swallowing causes severe burns. Avoid breathing dust. Prevent contact with eyes and skin.[2] TLV-TWA 2 mg(Al)/m^3.[13]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain with at least 50 times its volume of water. Treat the solid residue as normal refuse.[14–16] The area of the spill should be washed thoroughly with soap and water.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, place the aluminum chloride in a large evaporating dish. Cover with excess solid sodium carbonate or calcium carbonate. Very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test pH of the solution and neutralize if necessary. Pour the solution to the drain with at least 50 times its volume of water. Treat the solid residue as normal refuse.[14,15]

Reactions for Spillage and Waste Disposal

$$2AlCl_3 + 3H_2O + 3Na_2CO_3 \rightarrow \underset{\substack{\text{aluminum}\\\text{hydroxide}\\\text{(insoluble)}}}{2Al(OH)_3} + 6NaCl + 3CO_2$$

REFERENCES

1. Merck 335.
2. Lux 205.
3. B 36.
4. Clar, E. et al., *Tetrahedron*, 30, 3296, 1974.
5. B 267.
6. Boyer, J.H. et al., *Chem. Rev.*, 54, 29, 1954.
7. B 37.
8. Anonymous, *Chem. Eng. News*, 31, 4915, 1953.
9. Cowen, F.M. et al., *Chem. Eng. News*, 26, 2257, 1948.
10. Webb, H.F., *Chem. Eng. News*, 55, 4, 1977.
11. de Jongh, H.A.P., *Chem. Eng. News*, 55, 31, 1977; Brown, H.C., *Chem. Eng. News*, 55, 5, 1977.
12. Anonymous, *Ind. Eng. Chem.* (News Ed.), 12, 194, 1934.
13. ACGIH 14.
14. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
15. PPL 165.
16. Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 24, 1999.

ALUMINUM HYDRIDE AlH_3

IGNITES IN AIR

Physical Properties

Colorless, nonvolatile solid.[1]

Chemical Properties

Reducing agent.[1]

Hazardous Reactions

May decompose explosively at room temperature; ignites in air.[2,3]

Carbon Dioxide, Methyl Ethers. Decomposes violently in dimethyl or di-(2-methoxyethyl) ethers if carbon dioxide is present in the solution. If aluminum chloride is also present, the decomposition is explosive.[4]

Tetrazoles. Complexes of aluminum hydride and a variety of tetrazoles are explosive.[5]

Physiological Properties and Health Hazards

TLV-TWA 5 mg(Al)/m^3.[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection, and work from behind body shield where possible. Keep available pulverized dolomite or dry graphite for fire-fighting. Eliminate all sources of ignition. Follow waste disposal procedure.

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Cover the hydride with a 1:1:1 mixture by weight of sodium or calcium carbonate, clay cat litter (bentonite), and sand. Mix carefully. Place material in a large container behind a safety shield in the hood. Slowly add dry butyl alcohol (31 mL per gram of aluminum hydride). After reaction ceases, slowly and cautiously add water (three times the volume of alcohol added). Neutralize with 6 M hydrochloric acid (prepared by adding concentrated acid to an equal volume of cold water), and let stand until solids settle. Decant the liquid into drain and discard the solid residue as normal refuse.[7,8]

Reactions for Spillage and Waste Disposal

$$AlH_3 + 3CH_3CH_2CH_2CH_2OH \rightarrow \underset{\text{aluminum butoxide}}{Al(OCH_2CH_2CH_2CH_3)_3} + 3H_2$$

$$3Al(OCH_2CH_2CH_2CH_3)_3 + 3H_2O + H^+ \rightarrow \underset{\substack{\text{aluminum}\\ \text{hydroxide}\\ \text{(insoluble)}}}{Al(OH)_3} + 3CH_3CH_2CH_2CH_2OH + H^+$$

REFERENCES

1. Merck 341.

2. Mirviss, S.B. et al., *Ind. Eng. Chem.*, 53, 54A, 1961.
3. B 40.
4. Barbaras, G. et al., *J. Am. Chem. Soc.*, 70, 877, 1948.
5. B 41.
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7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
8. PPL 163.

2-AMINOAZOTOLUENE $C_{14}H_{15}N_3$

ANIMAL CARCINOGEN

Synonyms

4'-Amino-2,3'-dimethylazobenzene, Solvent Yellow 3, Fast Garnet GBC base, Cl 11160.

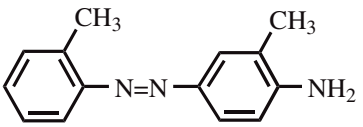
Physical Properties

Golden crystals; mp, 101–102°C.[1]

Chemical Properties

Practically insoluble in water; soluble in alcohol, ether, and chloroform.[1]

Structure



Hazardous Reactions

When heated to decomposition, emits toxic fumes.[2]

Physiological Properties and Health Hazards

In humans, produces eczema-like allergic reactions.[3] Carcinogen in mice, rats, hamsters, and dogs. Ingestion in these animals produces liver, bladder, and lung tumors. Reasonably anticipated to be a human carcinogen.[4]

Waste Disposal

Place in a separate labeled container for disposal by burning. Dissolve or mix the material into a combustible solvent and burn in furnace equipped with afterburner and scrubber.[5]

REFERENCES

1. Merck 419.
2. Sax 144.
3. IARC 8, 61.
4. NIEHS III–72.
5. Ald 1677B.

4-AMINOBIPHENYL $C_{12}H_{11}N$

HUMAN CARCINOGEN

Synonyms

p-Biphenylamine.

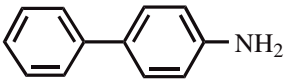
Physical Properties

Colorless leaflets from alcohol or water; mp, 53°C; bp, 302°C.[1,2]

Chemical Properties

Slightly soluble in cold water; readily soluble in hot water, alcohol, and chloroform; volatile with steam.[1]

Structure



Hazardous Reactions

Low to moderate fire hazard when exposed to heat, flames, or powerful oxidizers. When strongly heated, it emits toxic fumes.[2]

Physiological Properties and Health Hazards

Acute exposure produces headaches, lethargy, cyanosis, urogenital problems, and edema.[3] Occupational exposure to the dust can cause bladder tumors.[4] Animal carcinogen; bladder cancer strongly associated with occupational exposure.[5–7] Listed as known human carcinogen.[7]

Spillage Disposal

Wear dust mask, goggles, rubber gloves, and protective clothing. Carefully scoop the solid into beaker and transport to the fume hood. Dispose of material using waste disposal procedure. Wash the site of the spill with soap and water, disposing of the cleaning materials in a sealed bag or container for disposal by burning.[8]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning.[6]

Small Quantities. Wear goggles, rubber gloves, and protective clothing. Work in the fume hood. To 1.0 g of 4-aminobiphenyl in a 125-mL Erlenmeyer flask, add a mixture of 0.8 mL of water and 2.5 mL of concentrated hydrochloric acid. Stir the mixture for 10–15 minutes until a homogeneous slurry is formed. Cool the slurry to 0°C in an ice-salt bath and add a solution of 1.0 g of sodium nitrite in 2.5 mL of water dropwise at such a rate that the temperature of the mixture does not rise above 5°C. After stirring for 1 hour, slowly add 13 mL of ice-cold 50% hypophosphorus acid. Some foaming will occur. After addition of the acid is complete, stir for 18 hours at room temperature. Collect the solid by filtration, wash the filtrate into the drain with water, and discard the solid (biphenyl) with normal refuse or dispose of by burning.[8,9]

REFERENCES

1. Merck 1235.
2. Sax 168.
3. Sittig, M., *Handbook of Toxic and Hazardous Chemicals and Carcinogens*, Noyes Publications, Park Ridge, NJ, 1991, p. 85.
4. Lux 210.
5. IARC 1, 74.
6. Ald 143D.
7. NIEHS III–3.
8. Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 283.
9. Vogel 920–927.

2-AMINOFLUORENE $C_{13}H_{11}N$

SUSPECTED CANCER AGENT

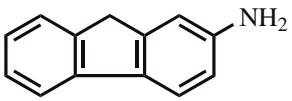
Physical Properties

Needles from 50% ethanol; mp, 129°C.[1]

Chemical Properties

Nearly insoluble in neutral aqueous solutions; readily soluble in dilute acid, alcohol, and ether. May oxidize slowly in solution or as a solid; most stable in acidic solution and when stored at 0°C or below.[2]

Structure



Physiological Properties and Health Hazards

Suspected cancer agent.[3] Oral or parenteral administration to rodents results in tumors of the liver, mammary gland, urinary bladder, intestine, and sebaceous glands of the ear duct. Mutagenic and slightly toxic.[2]

Spillage Disposal

Wear a dust mask, goggles, rubber gloves, and protective clothing. Carefully scoop the solid into a beaker and transport to the fume hood. Destroy the material using the waste disposal procedure. Wash the site of the spill with soap and water, and place the cleaning materials in a sealed bag or container for disposal by burning.[3,4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve the material in a combustible solvent and burn in furnace equipped with afterburner and scrubber.[3]

Small Quantities. Wear goggles, rubber gloves, and protective clothing. Work in the fume hood. To 1.0 g of 2-aminofluorene in a 125-mL Erlenmeyer flask, add a mixture of 0.8 mL of water and 2.5 mL of concentrated hydrochloric acid. Stir the mixture for 10–15 minutes until a homogeneous slurry is formed. Cool the slurry to 0°C in an ice-salt bath and add a solution of 1.0 g of sodium nitrite in 2.5 mL of water dropwise at such a rate that the temperature of the mixture does not exceed 5°C. After stirring for 1 hour, slowly add 13 mL of ice-cold 50% hypophosphorus acid, and then stir for 18 hours at room temperature. Collect the solid (fluorene) by filtration and discard as normal refuse or dispose of by burning. Wash the filtrate into the drain with water.[4,5]

REFERENCES

1. DOC 1, 133.
2. Beilstein 12, 1331.

3. Ald 175B.
4. Armour, M.A., Disposal methods for some known or suspect chemical carcinogens, in *Recent Advances of Chemistry and Molecular Biology in Cancer Research*, Dai, Q., Armour, M.A., and Zheng, Q., Eds., Springer-Verlag, Heidelberg, 1993, p. 315; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 283.
5. Vogel 920–927.

AMMONIA (solution) NH_4OH

CAUSES BURNS

Physical Properties

Ammonia solution is commonly supplied to laboratories as a 35% solution in water (0.88 specific gravity). In warm weather this strong solution develops pressure in its bottle and the cap must be released with care.

Colorless liquid; intense, pungent, suffocating odor.[1]

Fire Hazard

Explosive limits, 16–25%; ignition temperature, 651°C.

Hazardous Reactions

Nitromethane. Addition to nitromethane renders it susceptible to initiation by a detonator.[1,2]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system. The solution causes severe eye and skin burns. Swallowing the solution results in severe internal damage. Avoid breathing vapor. Prevent contact with eyes and skin.[3] TLV-TWA 25 ppm (17 mg/m^3); TLV-STEL 35 ppm (24 mg/m^3).[4]

Spillage Disposal

Wear a self-contained breathing apparatus, laboratory coat, eye protection, and nitrile rubber gloves (and rubber boots or overshoes if spillage is large). Cover the spilled liquid with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container and, in the fume hood, add to a pail of cold water. Neutralize the mixture with 5% hydrochloric acid, let stand overnight, and then pour the liquid into the drain. Dispose of any solid with normal refuse. Wash the area of the spill with plenty of water.[5–7]

Waste Disposal

Small Quantities. Wear nitrile rubber gloves, eye protection, all-purpose or special canister respirator for ammonia, and laboratory coat. Pour into a large container of water and neutralize with 5% hydrochloric acid. Pour into the drain.[6]

Reactions for Spillage and Waste Disposal

$$NH_4OH + HCl \rightarrow \underset{\text{ammonium chloride}}{NH_4Cl} + H_2O$$

REFERENCES

1. Merck 494.
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AMMONIUM DICHROMATE $(NH_4)_2Cr_2O_7$

HUMAN CARCINOGEN AS DUST, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE, FLAMMABLE

Physical Properties

Bright orange-red crystals; dec, 180°C.[1]

Fire Hazard

Self-sustaining decomposition occurs at 225°C with swelling and release of heat and nitrogen gas, leaving Cr_2O_3. Extinguish with water.[2]

Chemical Properties

Very soluble in water; oxidizing agent.[1]

Hazardous Reactions

Decomposes and ignites on heating to 190°C. Green Cr_2O_3 is emitted and explosion occurs if confined.[3]

Physiological Properties and Health Hazards

The dust irritates the respiratory tract and eyes. Swallowing causes internal irritation and damage. Prolonged exposure to the dust may cause ulceration, liver and kidney damage, and ultimately cancer. Avoid inhaling dust. Avoid contact with eyes and skin.[4] TLV-TWA (as Cr) 0.5 mg/m^3.[5]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a container of water. Add 3 M sulfuric acid (prepared by cautiously adding 10 mL of concentrated acid to 50 mL of cold water) to neutralize the solution, and then add a further 20 mL for each 1 g of dichromate (to give a pH of 1). While stirring, add sodium thiosulfate crystals until the solution becomes blue colored and cloudy (approximately 10 g/g dichromate). Slowly and cautiously neutralize the solution with sodium carbonate (approximately 3.2 g/mL of 3 M sulfuric acid). The mixture can be filtered immediately through Celite or allowed to stand for 1 week and the liquid decanted. In both cases the solution may be washed into the drain with 50 times its volume of water. The solid residue should be washed thoroughly with hot water, air dried, packaged, labeled, and sent to a secure landfill site.[6,7] The spillage area should be washed with soap and water and clothing soaked in the solution should also be washed thoroughly.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. The procedure is given for 1 g of dichromate. Dissolve in 10 mL of water. Acidify with 5.5 mL of 3 M sulfuric acid (prepared by cautiously adding 10 mL of concentrated acid to 50 mL of cold water) to give a pH of about 1. Slowly add, while stirring, 10 g of sodium thiosulfate. The solution becomes blue colored and cloudy. Slowly and cautiously add about 20 g of

solid sodium carbonate to neutralize the solution. Filter through Celite or let stand for 1 week while solids settle and decant the liquid. In both cases, the solution may be poured into the drain. The solid residue is washed with hot water, dried, packaged, labeled, and sent to a secure landfill site.[6]

Reactions for Spillage and Waste Disposal

$$Cr_2O_7^{2-} + 3S_2O_3^{2-} + 2H_3O^+ \rightarrow \underset{\text{chromium hydroxide (insoluble)}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

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AMMONIUM NITRATE NH_4NO_3

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIALS

Physical Properties

Colorless crystals; mp, 169°C; bp decomposes at above 210°C, mostly into H_2O and N_2O.[1,2]

Fire Hazard

Mixtures of ammonium nitrate and combustible material are readily ignited; mixtures with finely divided combustible materials can react explosively. Extinguish fire with water spray.[2]

Chemical Properties

Soluble in water.[2] One gram dissolves in 0.5 mL of water, 0.1 mL of boiling water, about 20 mL of ethanol, and about 8 mL of methanol.[1]

Hazardous Reactions

In the absence of impurities it is difficult, but not impossible, to cause ammonium nitrate to detonate.[3]

Acetic Acid. Concentrated mixtures ignite on heating.[3]

Acetic Anhydride, Hexamethylenetetramine Acetate, and Nitric Acid. Explosive mixture due to formation of acetyl nitrate.[4]

Alkali Metals. Sodium progressively reduces the nitrate, forming a yellow explosive solid.[10]

Aluminium, Calcium Nitrate, and Formamide. A mixture containing 51% calcium nitrate and 12% ammonium nitrate with 27% formamide and 10% water is detonatable at −20°C.[5]

Ammonia. Dependent on conditions, the presence of free ammonia in ammonium nitrate may either stabilize or destabilize the salt.[6]

Ammonium Chloride (Barium Nitrate), Water, and Zinc. Addition of water to a mixture of zinc powder and salts causes spontaneous ignition.[7]

Ammonium Phosphate and Potassium Sulfate. During the manufacture of 26/16/10 N/P/K fertilizer, the possibility of a thermal explosion exists.[8]

Ammonium Sulfate and Potassium. Ammonium nitrate containing the sulfate readily explodes on contact with potassium or its alloy with sodium.[9]

Charcoal and Metal Oxides. The explosive in the form of pellets normally ignites at 160–165°C, but presence of rust, or copper oxide or zinc oxide lowers the temperature to 80–120°C.[11]

Chloride Salts. The presence of 0.1% of ammonium chloride, calcium chloride, or iron(III) chloride lowers the initiation temperature of decomposition. With calcium and iron chlorides, it may be lowered enough to give violent or explosive decomposition.[12]

Copper Iron(II) Sulfide. This mixture resulted in a premature explosion, using ammonium nitrate-based blasting cartridges.[13]

Fertilizer Materials. Mixtures of ammonium nitrate, superphosphate, and organic materials stored in bulk may ignite if the internal temperature exceeds 90°C.[14]

Metals. Many powdered metals react violently or explosively below 200°C, including aluminium, antimony, bismuth, cadmium, cobalt, copper, iron, lead, magnesium, manganese, nickel, potassium, sodium, tin, titanium, and zinc, as well as brass and stainless steel.[15,16]

Metal Salts. Metal salts have a catalytic effect on the thermal decomposition and burning under nitrogen of the nitrate.[17,18]

Nonmetals. With charcoal at 170°C or with glowing charcoal, the nitrate explodes. Phosphorus ignites on the fused salt.[19]

Organic Fuels. Mixtures present an explosion hazard; 2–4% oil mixtures are used as commercial explosives.[20–22]

Potassium Nitrite. With fused nitrite causes incandescence.[23]

Potassium Permanganate. A mixture with 0.5% potassium permanganate exploded after 7 hours.[24]

Sawdust. Explosion occurred in a store with sawdust-covered floor.[25]

Sugar. Mixtures with sugar syrup are explosives.[26]

Sulfide Ores. In sulfide ore mines, spontaneous explosions of blasting explosives are due to the presence of ammonium nitrate, pyrites, and their weathering products, as well as pH and temperature.[27]

Sulfur. Nitrate-sulfur mixtures present a fire and explosion risk.[28,29]

Urea. Concentrated solutions of ammonium nitrate and urea exploded during large-scale mixing operations.[30]

Water. Hot aqueous solutions of the nitrate in concentrations above 50% may decompose explosively.[31]

Physiological Properties and Health Hazards

Irritant to skin, eyes, upper respiratory tract, and mucous membranes. Prolonged exposure may produce gastro-intestinal upsets and blood disorders.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and goggles. Scoop the solid into a pail of water. Slowly add dry sodium carbonate to pH 7. The solution can be poured into the drain. The site of spillage should be washed thoroughly to remove all of the ammonium nitrate which may render organic matter (e.g., wood) dangerously explosive.[32,33]

Waste Disposal

Package and label for recycling or disposal.[34] Alternatively, dissolve in a large container of water, add sodium carbonate slowly while stirring until the solution is neutral (pH 7), and then pour into the drain with water.[32,33]

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AMMONIUM SULFIDE SOLUTION $(NH_4)_2S$

LIBERATES HIGHLY TOXIC HYDROGEN SULFIDE ON CONTACT WITH ACID

Physical Properties

Commercial product is 16–20% ammonium sulfide in water. Colorless liquid becomes yellow on standing due to formation of polysulfide.[1]

Chemical Properties

Strongly alkaline solution.[1]

Hazardous Reactions

Contact with acid liberates highly toxic hydrogen sulfide.[1,2]

Physiological Properties and Health Hazards

Ammonium sulfide solution is toxic if swallowed or absorbed through the skin. The liquid is corrosive to the skin, eyes, and mucous membranes.[2] Avoid inhalation of the vapor.

Spillage Disposal

Aqueous solutions of ammonium salts. Wear nitrile rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all of the liquid has been absorbed, scoop the mixture into a plastic pail and place in the fume hood. Add sufficient 1% aqueous sodium hydroxide solution to dissolve the sodium carbonate in the spill mix and to ensure that the liquid is basic. Estimate the volume of ammonium sulfide solution that was spilled and add 200 mL of household chlorine bleach containing 5.25% sodium hypochlorite for each 10 mL of commercial ammonium sulfide solution (commercial ammonium sulfide solution is about 20%; this volume of bleach allows a 25% excess). Allow the mixture to stand in the fume hood overnight and decant the liquid into the drain. Dispose of the solid (sand and calcium bentonite) in normal refuse.[3,4]

Waste Disposal

Package Lots. Place in labeled container for disposal in accordance with local regulations.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in fume hood. For each 10 mL of commercial ammonium sulfide solution, add 100 mL of 1% aqueous sodium hydroxide solution and 200 mL of household bleach. Allow the solution to stand overnight, and then pour into the drain.[3]

Reactions for Spillage and Waste Disposal

$$\underset{\text{ammonium sulfide}}{(NH_4)_2S} + 4NaOCl \rightarrow \underset{\text{ammonium sulfate}}{(NH_4)_2SO_4} + NaCl$$

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AMSACRINE $C_{21}H_{19}N_3O_3S$

ANTINEOPLASTIC

Synonyms

N-[4-(9-Acridinylamino)-3-methoxy-phenyl]methanesulfonamide, 4'-(9-acridinylamino)methanesulfon-*m*-anisidine, Amsidine, NSC 249992, *M*-AMSA.[1]

Physical Properties

The color ranges from bright yellow to deep red, depending on various derivatives and amount of hydration.[2]

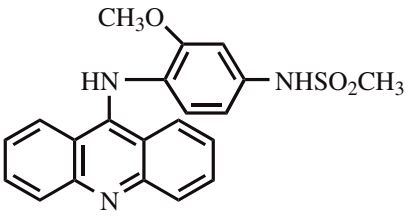
Mode of Action

Cytostatic agent with antiviral and immunosuppressive properties.[1]

Chemical Properties

Insoluble in water.[3]

Structure



Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, diarrhea, pain, and bone marrow depression (high dose).[4] LD_{50} (as hydrochloride) (intraperitoneal, mice) 60 mg/kg.[1]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of amsacrine in the spilled solution and to the mixture add household bleach (5% sodium hypochlorite) allowing 10 mL of bleach for each 0.5 mg of amsacrine. Allow to stand overnight, and then decant the liquid into the drain with water and discard the solid as normal refuse.[5]

Waste Disposal

Wear goggles and protective gloves and clothing. Work in the fume hood. Add 10 mL of household bleach (5% sodium hypochlorite) to 0.1 mg of amsacrine. After standing at room temperature for 30 minutes, wash the solution into the drain with water. Immerse vials, syringes, and other glassware that have been in contact with amsacrine in bleach, allow to stand overnight, and then rinse thoroughly with water and discard or reuse.[5]

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ANILINE $C_6H_5NH_2$

GIVES OFF POISONOUS VAPOR

Physical Properties

Colorless to brown liquid; bp, 185°C.[1]

Fire Hazard

Flash point, 70°C; flammable limits, 1.3–11%; ignition temperature, 615°C. Extinguish with water, dry chemical, or carbon dioxide.[2]

Chemical Properties

Immiscible with water; miscible with most organic solvents.[1]

Hazardous Reactions

Benzenediazonium-2-carboxylate. The salt reacts explosively with aniline.[3]

Boron Trichloride. Interaction with aniline is violent.[4]

Dibenzoyl Peroxide. Addition of 1 drop of aniline to 1 g of the peroxide results in mildly explosive decomposition.[5]

Nitromethane. A mixture of nitromethane and aniline can be detonated.[6]

Oxidants. Aniline is oxidized vigorously by perchloric acid, fuming nitric acid, sodium peroxide, and ozone.[7]

Physiological Properties and Health Hazards

Breathing vapor causes headache, drowsiness, cyanosis, mental confusion, and finally convulsions. Liquid damages eyes and if swallowed results in similar effects to breathing vapor. Prolonged breathing of vapor or skin contact disturbs the nervous system and blood. Avoid breathing vapor. Avoid contact with eyes and skin.[8] TLV-TWA 2 ppm (7.6 mg/m^3).[9]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl[10] rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the aniline has been absorbed, scoop the mixture into a plastic pail and add enough water to dissolve the sodium carbonate. Allow the solids to settle and decant the liquid to another container. Discard the solids with normal refuse. To the liquid, slowly (frothing will occur) add 6 M sulfuric acid to pH 2. Stir into the acidified solution sufficient solid potassium permanganate so that the liquid is purple (a drop of the liquid on a filter paper will show a purple ring). Allow the mixture to stand at room temperature for 48 hours, and then neutralize with solid sodium carbonate (frothing will occur), or with a 10% aqueous solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[9,13]

Waste Disposal

Wear eye protection, laboratory coat, and rubber gloves. Dissolve the aniline (1 mL) in 50 mL of 3 M sulfuric acid (prepared by slowly adding 8 mL of concentrated sulfuric acid to 21 mL of water). Weigh 10 g of potassium permanganate and stir small portions of the solid into the aniline solution over a period of about 1 hour. Stir the mixture at room temperature for 48 hours, and then neutralize

the solution by adding solid sodium carbonate or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until solution is colorless. Decant the clear liquid into the drain and discard any brown solid with regular refuse.[11,12]

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ANTIMONY COMPOUNDS Sb^{3+}, Sb^{5+}

POISON

Physical Properties

Most soluble antimony compounds are colorless crystals or powder; the pentachloride is a colorless to yellow fuming liquid with an offensive smell.[1]

Hazardous Reactions

Antimony Compounds (Trivalent)

Perchloric Acid. Trivalent antimony compounds tend to form explosive mixtures with perchloric acid when hot.[2,3]

Antimony Perchlorate $SbOClO_4$

This chemical decrepitates when heated above 60°C.[4]

Antimony Pentafluoride SbF_5

Phosphorus. Phosphorus ignites in contact with antimony pentafluoride.[5]

Antimony Sulfide Sb_2S_3

Air. When the crystalline form of antimony trisulfide is heated in air, it burns with a blue flame.[6]

Chlorates. Antimony sulfide reacts with incandescence with chlorates of cadmium, magnesium, or zinc.[7]

A mixture of chlorates plus antimony sulfide, cyanide, or thiocyanate explodes. The reaction may be caused by heat, shock, or friction.[8]

Antimony Tribromide $SbBr_3$

Potassium or Sodium. A mixture of antimony tribromide with potassium or sodium produces a strong explosion on impact.[9]

Physiological Properties and Health Hazards

All soluble antimony compounds must be considered poisonous when taken by mouth. Some compounds cause skin irritation and dermatitis. If taken by mouth, soluble antimony compounds may cause burning of the mouth and throat, choking, nausea, and vomiting. Stibine, which may be formed by the action of acidic reducing agents on antimony-containing materials, is an extremely poisonous gas, causing blood destruction and damage to liver and kidneys. Insoluble antimony compounds, such as the oxide and sulfide, are not toxic. Avoid contact with skin.[1] TLV-TWA 0.5 mg(Sb)/m^3.[10]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Avoid breathing dust. Brush spill into a container and transport to the fume hood. Follow waste disposal procedure.

Waste Disposal

Work in the fume hood. Wear nitrile rubber gloves, eye protection, and a laboratory coat. Avoid breathing dust. Dissolve the soluble antimony salt (0.04 mol, e.g., 0.8 g of antimony trifluoride) in water (20 mL) and add, while stirring, a solution of 25 g of sodium metasilicate pentahydrate ($Na_2SiO_3 \cdot 5H_2O$, 0.12 mol) in water (200 mL). Adjust pH to 10 with 2 M sulfuric acid. Collect the precipitate by filtration or allow the liquid to evaporate in a large evaporating dish in the fume

hood. Package and label the dry solid for disposal in accord with local regulations.[11] For dilute solutions of antimony salts, add the sodium metasilicate solution until there is no further precipitation. Allow the mixture to stand overnight before collecting the solid by filtration or allowing the liquid to evaporate.[11]

Reactions for Spillage and Waste Disposal

$$2Sb^{3+} + 3Na_2SiO_3 \rightarrow \underset{\substack{\text{antimony silicate}\\ \text{(insoluble)}}}{Sb_2(SiO_3)_3} + 6Na^+$$

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ARSENIC COMPOUNDS As^{2+}, As^{3+}, As^{5+}

SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING, DANGER OF CUMULATIVE EFFECTS, CARCINOGENIC

Physical Properties

Most arsenic compounds are colorless powders or crystals. They include arsenites and arsenates of many metals; syrupy arsenic acid and arsenic trichloride are liquids.[1] All must be considered to be extremely poisonous. The metal itself has not been recognized as a noteworthy hazard.[2]

Hazardous Reactions

Arsenic Sulfides AsS_2, As_2S_3, As_2S_5

Chlorates. Arsenic sulfide reacts with incandescence with chlorates of cadmium, magnesium, or zinc.[3]

Arsenic Trichloride $AsCl_3$.

Potassium or Sodium. A mixture of arsenic trichloride with potassium or sodium produces a strong explosion on impact.[4]

Arsenic Trifluoride AsF_3

Phosphorus Trioxide. This reaction is very violent.[5]

Arsenic Triiodide AsI_3

Potassium or Sodium. A mixture of arsenic triiodide with potassium or sodium produces a strong explosion on impact.[4]

Arsenic Trioxide As_2O_3

Fluorine. Fluorine reacts violently with arsenic trioxide.[6]

Arsenic Trisulfide As_2S_3

Hydrogen Peroxide. Hydrogen peroxide reacts vigorously with arsenic trisulfide.[7]

Arsine AsH_3

Chlorine. When chlorine is bubbled into arsine, each bubble produces a flame.[8]

Nitric Acid. Fuming nitric acid reacts explosively with arsine.[9]

Physiological Properties and Health Hazards

The inhalation of dust or fumes irritates the mucous membranes and leads to arsenical poisoning. Certain compounds, especially the trichloride and arsenic acid, irritate the eyes and skin, and absorption causes poisoning. If swallowed, arsenic compounds irritate the stomach severely and affect the heart, liver, and kidneys; nervousness, thirst, vomiting, diarrhea, cyanosis, and collapse may be symptoms. Prolonged inhalation of small concentrations of dust or fumes over a long period will cause poisoning; skin contact over a long period may cause ulceration.
Do not inhale dust or fumes. Prevent contact with skin and eyes.[2] TLV-TWA 0.01 mg (As)/m^3.[10]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Avoid breathing dust. Brush spill into a container and transport to the fume hood. Follow waste disposal procedure.

Waste Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Avoid breathing dust. In the fume hood, dissolve the arsenic compound in acidified boiling water (for 1 g of arsenic compound, use 100 mL of water containing 6 drops of concentrated hydrochloric acid). Add a solution of thioacetamide (for each 1 g of arsenic salt, use 0.2 g of thioacetamide in 20 mL of water). Boil the mixture for 20 minutes and then basify with 2 M sodium hydroxide (prepared by dissolving 8 g of NaOH in 100 mL of water). Filter the precipitate, dry, and package for disposal in a secure landfill site.[11]

Reactions for Spillage and Waste Disposal

$$CH_3CSNH_2 + HCl + 2H_2O \rightarrow CH_3COOH + H_2S + NH_4Cl$$

$$2As^{3+} + 3H_2S \rightarrow As_2S_3 + 6H^+$$

arsenic
sulfide
(insoluble)

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ARSINE AsH_3

VERY TOXIC

Synonyms

Arsenic trihydride.

Physical Properties

Colorless neutral gas; disagreeable garlic odor; mp, −117°C; bp, −62.5°C.[1]

Fire Hazard

Flammable.[2] Explosive limits, 3.9–77.8%.

Chemical Properties

Soluble in chloroform, benzene.[3] Slightly soluble in water (neutral solution); decomposes when heated to 300°C; on exposure to light, moist arsine decomposes quickly, depositing shiny black arsenic.[1]

Hazardous Reactions

Arsine can be detonated.[4]
Chlorine. Ignites in contact with chlorine.[5]

Nitric Acid. Explosively oxidized by fuming nitric acid.[6]

Physical Properties and Health Hazards

The vapor is extremely toxic and causes pulmonary edema. A few inhalations may be fatal. Symptoms of poisoning include headache, weakness, vertigo, and nausea. Damage is caused to kidneys and liver.[2] TLV-TWA 0.05 ppm (0.16 mg/m^3).[7]

Spillage Disposal

Traces of arsine are best removed by absorption in potassium permanganate solution or bromine water.[1]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus. Remove the leaking cylinder to a safe area outdoors or to an efficient fume hood. Fit the cylinder with an appropriate valve and length of hose. To avoid a violent reaction with air, the oxidation must be carried out under a nitrogen atmosphere in a large three-necked, round-bottom flask equipped with magnetic stirrer, nitrogen inlet tube, and gas inlet tube. Discharge the gas slowly into 15% aqueous sodium hydroxide (forms a water soluble complex) in the 3-necked flask through the gas inlet tube. Neutralize the solution to pH 7 with 1 M sulfuric acid (prepared by slowly adding 10 mL of concentrated acid to 170 mL of cold water). Slowly add 1 M sodium sulfide (78 g of Na_2S dissolved in 1 L of water) to the arsenic solution. Check that the solution is neutral (pHydrion paper) and adjust to neutrality with 1 M sulfuric acid if necessary. The precipitate is separated by filtration and packaged for disposal in a secure landfill. The aqueous solution is flushed down the drain with at least 50 times its volume of water.[8]

Reactions for Spillage and Waste Disposal

$$2As^{3+} + 3Na_2S \rightarrow As_2S_3 + 6Na^+$$

arsenic sulfide (insoluble)

REFERENCES

1. Merck 818.
2. Lux 228.
3. CRC.
4. B 52.
5. B 1005.
6. B 1171.
7. ACGIH 16.
8. PPL 167.

ASBESTOS DUST (native Ca-Mg silicate)

HUMAN CARCINOGEN

Physical Properties

Fine, slender, flaxy fibers.[1]

Chemical Properties

Fire and solvent resistant.[1]

Physiological Properties and Health Hazards

Occupational exposure to the dust may cause cancer after a long latent period.[1]
For fibers greater than 5 micrometers in length:
Amosite. TLV 0.1 fiber/cc.

Chyrsotile. TLV 0.1 fibers/cc.

Crocidolite. TLV 0.1 fiber/cc.

Other forms. TLV 0.1 fibers/cc.[2]

Waste Disposal

Wear impermeable gloves and dust mask.

Place the asbestos in a separate labeled container for disposal by burning. Burn in furnace whose temperature and design are appropriate to asbestos.[3]

REFERENCES

1. Merck 833.
2. ACGIH 16.
3. ITI 064–55.

AZIDES and ACYL AZIDES

See *tert*-Butyl Azidoformate and Ethyl Azidoformate entries.

Azides of low molecular weight (more than 25% nitrogen content) should not be isolated from solution since the concentrated material is likely to be dangerously explosive.[1] The concentration of such solutions (prepared below 10°C) should be below 10%.[2]
Carbonyl azides are explosive compounds, some exceptionally so; suitable handling precautions are necessary.[3]

Metal Azides[4]

See Silver Azide and Sodium Azide entries.
This large and well-documented group of explosive compounds contains some that are widely used in industrial processes.

Organic Azides[5]

See Phenyl Azide entry.
Careful and small-scale handling of organic azides, which are usually heat- or shock-sensitive compounds of varying degrees of stability, is necessary. The presence of more than one azido group, particularly if on the same atom (C or N) greatly reduces stability.

Waste Disposal

Metal azides can be oxidized by ceric ammonium–nitrate solution.[6] Organic azides are smoothly reduced by using tin and hydrochloric acid. See individual entries for details of respective methods.[6]

REFERENCES

1. Smith, P.A.S., *Org. React.*, 3, 373–375, 1946.
2. Houben-Weyl, *Methoden der Organischen Chemie*, 4th ed., Vol. 8, Muller, E., Ed., G. Thieme, Stuttgart, 1952, p. 680.
3. Lieber, E. et al., *Chem. Rev.*, 65, 377, 1965.
4. Mellor, Vol. 8, 344–355; Vol. 8, Suppl. 2, 16–54, 1940.
5. Boyer, J.H. et al., *J. Chem. Eng. Data*, 9, 480, 1964; *Chem. Eng. News*, 42, 6, 1964.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985, Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, 1994, p. 100.

BARIUM COMPOUNDS Ba^{2+}

POISON

Physical Properties

Most barium compounds are colorless crystals or powders.[1]

Hazardous Reactions

Barium Azide $Ba(N_3)_2$

Highly sensitive to friction when dry. It is stored under alcohol. Exposure to acid may generate hydrazoic acid, and exposure to lead drains may generate lead azide.[2]

Barium Chloride $BaCl_2$

Boron Trifluoride. Boron trifluoride rapidly attacks barium chloride.[3]

Furan-2-peroxycarboxylic Acid. This acid explodes at room temperature upon addition of barium chloride.[3]

Barium Chlorite $Ba(ClO_2)_2$

Decomposes explosively on rapid heating to 190°C.[4]

Dimethyl Sulfate. Dimethyl sulfate ignites on contact with barium chlorite.[2]

Barium Hydride BaH_2

The finely divided hydride ignites in air[4] or when heated in oxygen.[5]

Barium Hydroxide $Ba(OH)_2$

Chlorinated Rubber. Chlorinated rubber reacts violently or explosively with barium hydroxide.[6]

Barium Nitrate $Ba(NO_3)_2$

Aluminum, Potassium Nitrate, Potassium Perchlorate, and Water. A mixture with barium nitrate exploded after 24 hours of underwater storage.[7]

Aluminum, Potassium Nitrate, Sulfur, and Water. A paste with barium nitrate explodes.[8]

Aluminum–Magnesium Alloy. A mixture of the metal powder with barium nitrate is extremely sensitive to friction or impact.[9]

Barium Oxide BaO

Dinitrogen Tetroxide. In contact with the gas at 200°C, the oxide suddenly reacts, reaches red heat, and fuses.[10]

Barium Perchlorate $Ba(ClO_4)_2$

Explosive alkyl perchlorates are formed on distillation of mixtures with alcohols.[4]

Barium Peroxide BaO_2

Acetic Anhydride. Formation of acetyl peroxide from mixtures of barium peroxide and acetic anhydride results in a violent explosion.[11]

Hydrogen Sulfide. Hydrogen sulfide may ignite in contact with barium peroxide.[12]

Hydroxylamine. Aqueous hydroxylamine ignites on contact with barium peroxide.[13]

Metals. Powdered aluminum or magnesium ignites on contact with the peroxide.[14]

Nonmetal Oxides. Barium peroxide incandesces when heated in a stream of CO_2 or SO_2.[14]

Performic Acid. Addition of barium peroxide to 90% acid causes violent decomposition.[15]

Propane. Heating barium peroxide under propane results in a violent exothermic reaction.[16]

Wood. Friction of the peroxide between wooden surfaces can cause ignition of the wood.[14]

Barium Sulfide BaS

Oxidants. The sulfide explodes on heating with lead dioxide, potassium chlorate, or potassium nitrate.[17]

Phosphorus Pentoxide. Violent interaction occurs.[18]

Physiological Properties and Health Hazards

All soluble barium compounds (i.e., all barium compounds except barium sulfate) are poisonous if swallowed and cause nausea, vomiting, stomach pains, and diarrhea.[18] TLV-TWA (soluble compounds) 0.5 mg (Ba)/m^3.[19]

Spillage Disposal

The sulfate may be brushed up and treated as normal refuse. Soluble barium salts should be scooped up and treated as for waste disposal. Wash the spill area thoroughly with water.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Dissolve the barium salt in the minimum volume of water. For each gram of barium salt, add 15 mL of 10% sodium sulfate solution. Let stand for 1 week. Test for completeness of precipitation by adding a few drops of 10% sodium sulfate solution. If required, add more sodium sulfate solution until no further precipitation occurs. Decant supernatant liquid or filter solid. Treat solid as normal refuse. Wash liquid into drain.[20]

Reactions for Spillage and Waste Disposal

$$Ba^{2+} + Na_2SO_4 \rightarrow \underset{\substack{\text{barium}\\\text{sulfate}\\\text{(insoluble)}}}{BaSO_4} + 2Na^+$$

REFERENCES

1. Lux 231.
2. B 82.
3. NFPA 491M.
4. B 80.
5. Mellor, Vol. 3, 650, 1941.
6. B 1537.
7. B 28.
8. Anonymous, *Chem. Eng. News*, 32, 258, 1954.
9. Tomlinson, W.R. et al., *J. Chem. Educ.*, 27, 606, 1950.
10. Mellor, Vol. 8, 545, 1940.
11. B 83.
12. Mellor, Vol. 10, 129, 141, 1947.
13. Mellor, Vol. 3, 670, 1941; Vol. 8, 287, 1940.
14. B 84.
15. D'Ans, J. et al., *Chemische Berichte*, 48, 1136, 1915.
16. Hoffmann, A.B. et al., *J. Chem. Educ.*, 51, 419, n. 7, 1974.

17. Mellor, Vol. 3, 745, 1941.
18. B 85.
19. ACGIH 16.
20. Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1996, p. 282.

BENOMYL $C_{14}H_{18}N_4O_3$

SYSTEMIC FUNGICIDE

Synonyms

Benlate, methyl-1-(butylcarbamoyl)-2-benzimidazole carbamate.

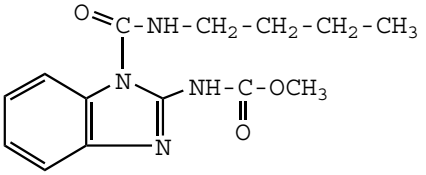
Physical Properties

White crystalline solid; negligible odor; decomposes without melting.[1]

Chemical Properties

Insoluble in water or oil[1]; soluble in chloroform, dimethylformamide, acetone, xylene, ethanol, and heptane.[2]

Structure



Physiological Properties and Health Hazards

LD_{50} (oral, rat), above 10,000 mg/kg; LC_{50} (rainbow trout, 96 hours) 0.17 mg/L.[3] TLV-TWA 10 mg/m^3.[4]

Waste Disposal

Wear eye protection and gloves. To each 1 g of benomyl 50, add 100 mL of 3 M sulfuric acid solution (17 mL of concentrated sulfuric acid added to 83 mL of cold water) and 4.7 g of potassium permanganate. Stir the mixture and allow to remain at room temperature for 2 hours. Add a saturated solution of sodium bisulfite until the solution is colorless. Neutralize solution by the careful addition of soda ash (foaming may occur) or 5% aqueous sodium hydroxide. The clear solution is washed into the drain with water.[5]

REFERENCES

1. Merck 1042.
2. Agro. Handbook, A0031.
3. Agro. Desk Ref., 43.
4. ACGIH 16.
5. Armour, M.A. and Bricker, Y., *Laboratory Disposal of Benomyl*, University of Alberta, Edmonton, 1995.

BENZAL CHLORIDE $C_6H_5CCl_2H$

IRRITANT

Synonyms

Benzylidene chloride.

Physical Properties

Colorless liquid; bp, 205°C; fumes in air.[1]

Chemical Properties

Immiscible with water.[2]

Physiological Properties and Health Hazards

Vapor irritates respiratory system and eyes. Liquid irritates eyes and skin. Both may cause conjunctivitis. Swallowing would cause internal damage. Avoid breathing vapor. Avoid contact with skin and eyes.[2]

Spillage Disposal

Instruct others to keep a safe distance. Wear breathing apparatus, laboratory coat, eye protection, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriate container and package for disposal by burning.[3–5] Area of spillage should be washed thoroughly with soap and water.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[4]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets into a 100-mL, three-necked, round-bottom flask equipped with stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. With brisk stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. Heat the solution to gentle reflux and add the benzal chloride (16.0 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux with stirring (to prevent bumping) for another 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[4]

Reactions for Spillage and Waste Disposal

$$C_6H_5CCl_2H + 2KOH \xrightarrow{C_2H_5OH} \underset{\text{benzoic acid}}{C_6H_5COOH} + \underset{\text{benzyl alcohol}}{C_6H_5CH_2OH} + 2KCl + H_2O$$

REFERENCES

1. Merck 1056.
2. Lux 239–240.

3. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
4. PP 61.
5. *Haz. Mat. Spills Tec.*, 29.5.

BENZALDEHYDE C_7H_6O

TOXIC

Physical Properties

Strongly refractive liquid, becoming yellowish on keeping; characteristic odor of volatile oil of almond; burning aromatic taste. Oxidizes in air to benozic acid; volatile with steam. Density, 1.050 at 25°C[1]; bp, 178–179°C; mp, –26°C.[2,3]

Fire Hazard

Flash point, 62°C; auto-ignition temperature, 192°C. Extinguish fires with water, dry powder, or foam.[3]

Chemical Properties

Soluble in 350 parts water; miscible with alcohol, ether, and oils. It reduces ammoniacal silver nitrate, but not Fehling's solution. Keep container tightly closed and protected from light.[1]

Hazardous Reactions

Oxidizers. Reacts violently with oxidizing agents.

Performic Acid. Violent oxidation with 90% performic acid.[3–5]

Physiological Properties and Health Hazards

A central nervous system depressant in small doses and narcotic in high concentrations. Toxic if inhaled, swallowed, or absorbed through skin.[3,5,6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and self-contained breathing apparatus. Cover the spill with a 1:1:1 by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all liquid has been absorbed, scoop the mixture into a plastic pail and place in the fume hood. Estimate the volume of benzaldehyde that was spilled, and for each 5 mL, add a solution of 6 g of potassium permanganate in 100 mL of water. Swirl the mixture thoroughly and leave to stand at room temperature for 48 hours. Add solid sodium bisulfite while stirring until a colorless solution is formed. Allow the solids to settle and pour the liquid into the drain. Dispose of the solids in regular refuse.[7]

Waste Disposal

Package Lots. Package and label for disposal by burning.[6]

Small Quantities. To each 5 mL of benzaldehyde to be discarded, add 6 g of potassium permanganate in 100 mL of 3 M sulfuric acid (prepared by adding 17 mL of concentrated sulfuric acid to 83 mL of water). Stir the mixture overnight. Add solid sodium bisulfite until the solution is clear; a small quantity of brown precipitate may remain. Neutralize the liquid with 5% aqueous sodium hydroxide and wash into the drain. Discard any brown solid with normal refuse.[7]

Reactions for Spillage and Waste Disposal

$$\underset{\text{benzaldehyde}}{C_6H_5CHO} \xrightarrow{KMnO_4} \underset{\text{benzoic acid}}{C_6H_5COOH}$$

REFERENCES

1. Merck 1057.
2. Aldrich 146.
3. Lux 106.
4. NFPA 491M.
5. Sax 348.
6. Ald 345B.
7. Armour, M.A., *J. Chem. Educ*. 65, A64, 1988; *Haz. Mat. Spills Tec*., 29.5.

BENZ[a]ANTHRACENE $C_{18}H_{12}$

HIGHLY TOXIC, ANIMAL CARCINOGEN

Synonyms

1,2-Benzanthracene, 2,3-benzphenanthrene.

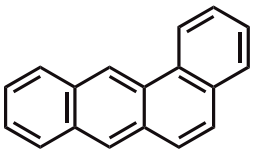
Physical Properties

Plates form from glacial acetic acid or ethanol.[1] Greenish-yellow fluorescence. Sublimes; mp, 159–161°C[2]; bp, 437.6°C.[1]

Chemical Properties

Insoluble in water; slightly soluble in boiling ethanol; soluble in most other organic solvents.[1]

Structure



Physiological Properties and Health Hazards

Carcinogen in mice.[2] Lowest toxic doses (mice) at 2 mg/kg (subcutaneous), 4 mg/kg/15 weeks (oral) and 240 mg/kg/15 weeks (skin).[3,4] Animal carcinogen; reasonably anticipated to be human carcinogen. Exposure by all routes should be carefully controlled to the lowest possible levels.[4]

Spillage Disposal

Wear protective gloves and clothing, goggles, and breathing apparatus if necessary. Turn off all ventilation and isolate spill area. Add enough dimethylformamide to completely wet the contaminated surface, and then pour over the area an excess of *freshly* prepared solution containing 4.7 g of potassium permanganate in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow to react for at least 1 hour. Cover the residual solution with a 1:1:1 of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container in fume hood, and slowly add it to a beaker of cold water. If necessary, neutralize the solution with soda ash. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning.[7]

Small Quantities. Wear goggles and protective gloves and clothing. Work in the fume hood. Dissolve the compound in acetone, allowing 2 mL for each 5 mg of benz[a]anthracene. For each 5 mg of benz[a]anthracene, add 10 mL of solution comprised of 4.7 g of potassium permanganate in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Swirl, and allow to stand at room temperature for 1 hour. Neutralize the mixture by the careful addition of 10% sodium hydroxide solution, and then slowly add a saturated aqueous solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water) until a colorless solution is formed. Wash into the drain with water.[5,6]

REFERENCES

1. Merck 1061.
2. IARC 3, 45.
3. Weiss, G., Ed., *Hazardous Chemicals Data Book*, Noyes Data Corporation, Park Ridge, NJ, 1980, p. 966.
4. NIEHS III–187; ACGIH 16.
5. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Waste: Some Polycyclic Aromatic Hydrocarbons*, No. 49, IARC Publications, Lyon, 1983, p. 17.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
7. Ald 347D.

BENZENE C_6H_6

HUMAN CARCINOGEN, EXTREMELY FLAMMABLE, GIVES OFF VERY POISONOUS VAPOR, DANGER OF CUMULATIVE EFFECTS

Synonyms

Benzol, coal naphtha.

Physical Properties

Colorless, volatile liquid with characteristic odor; bp, 80.1°C.[1]

Fire Hazard

Flash point, −11°C; flammable limits, 1.3–7.1%; ignition temperature, 498°C. Extinguish fire with foam, dry powder, or carbon dioxide.[2]

Chemical Properties

Immiscible with water; miscible with alcohol, chloroform, ether, carbon tetrachloride, carbon disulfide, glacial acetic acid, acetone, and oils.[1,2]

Hazardous Reactions

Arsenic Pentafluoride, Potassium Methoxide. Use of benzene as solvent in the reaction of arsenic pentafluoride with potassium methoxide resulted in explosions.[3]

Diborane, Air. Spontaneously explosive reaction of diborane occurs on contact with benzene vapor in air.[4]

Interhalogens. Benzene ignites on contact with bromine trifluoride,[5] bromine pentafluoride,[6] and iodine heptafluoride[7]; interacts violently with iodine pentafluoride.[8]

Oxidants. The solid complex formed between benzene and silver perchlorate may explode on crushing.[9] Permanganic acid, produced by reaction of permanganates with sulfuric acid, explodes on contact with benzene.[10] Mixtures of nitric acid and benzene containing about 84% acid are highly sensitive to detonation.[11] Benzene ignites on contact with concentrated hydrogen peroxide or with sodium peroxide and water.[12] Mixtures of benzene and liquid oxygen are explosive.[13] The ozonide of benzene is explosive[14]; during the ozonization of rubber dissolved in benzene, an explosion occurred.[15]

Uranium Hexafluoride. Very vigorous reaction with benzene.[16]

Physiological Properties and Health Hazards

Human carcinogen.[1] Vapor irritates eyes and mucous membranes; causes dizziness, headache, restlessness, convulsions, excitement, and depression. High concentrations may cause unconsciousness or death from respiratory failure. Swallowing or absorption through the skin results in severe poisoning. Prolonged breathing of vapor may cause severe or even fatal blood disease. Avoid inhalation of vapor. Prevent contact with skin and eyes.[17] TLV-TWA 0.5 ppm; TLV-STEL 2.5 ppm.[18]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and Viton® rubber gloves.[19] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriate container and package for disposal by burning.[20–22] Spillage site should be washed thoroughly with soap and water.[17]

Waste Disposal

Place in nonhalogenated solvent disposal container for recycling or disposal by burning.[21]

REFERENCES

1. Merck 1066.
2. NFPA 49.
3. Kolditz, L. et al., *Zeitschrift fur Anorganische Chemie*, 341, 88, 1965.
4. B 70.
5. Mellor, Vol. 2, Suppl. 1, 164, 1956.
6. Mellor, Vol. 2, Suppl. 1, 172, 1956.
7. Booth, H.S. et al., *Chem. Rev.*, 41, 428, 1947.
8. Ruff, O. et al., *Zeitschrift fur Anorganische Chemie*, 201, 245, 1931.
9. B 7; Peone, J. et al., *Inorg. Synth.*, 15, 69, 1974.
10. B 1146.
11. B 1165.
12. B 1383.
13. B 1408.
14. Mellor, Vol. 1, 911, 1941.
15. B 1419.
16. B 1126.
17. Lux 232.
18. ACGIH 16.
19. LSS.
20. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
21. ITI 074.
22. *Haz. Mat. Spills Tec.*, 29.7.

BENZENESULFONIC ACID $C_6H_5SO_3H$

CAUSES BURNS

Physical Properties

Anhydrous mp, 50–51°C (also reported as 65–66°C). Sesquihydrate, deliquescent plates, tablets; mp, 43–44°C.[1]

Chemical Properties

Freely soluble in water and alcohol; slightly soluble in benzene; insoluble in ether and carbon disulfide.[1]

Physiological Properties and Health Hazards

Solutions irritate skin and eyes and may cause burns. Swallowing results in internal irritation and damage. Avoid contact with skin and eyes.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse. Wash the area of the spill thoroughly with soap and water.[3,4]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, dilute the benzenesulfonic acid to 10% with water. Slowly, and while stirring, add 5% sodium hydroxide solution until the solution has a pH of 10. Allow to stand for 24 hours. Wash the liquid into the drain.[3]

Reactions for Spillage and Waste Disposal

$$C_6H_5SO_3H + Na_2CO_3 \xrightarrow{H_2O} C_6H_5SO_3^-Na^+ + H_2O + CO_2$$

$$C_6H_5SO_3H + NaOH \xrightarrow{H_2O} C_6H_5SO_3^-Na^+ + H_2O$$

sodium benzenesulfonate

REFERENCES

1. Merck 1070.
2. Ald 352B.
3. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
4. Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 24, 1999.
5. PP 66.

BENZENESULFONYL CHLORIDE $C_6H_5SO_2Cl$

CAUSES BURNS

Physical Properties

Colorless, oily liquid, solidifies at 0°C; mp, 14.5°C; bp, 251–252°C (dec).[1]

Chemical Properties

Insoluble in cold water; soluble in ether and alcohol.[1] Reacts slowly with water to form benzenesulfonic acid and hydrogen chloride.[2]

Hazardous Reactions

Dimethyl Sulfoxide. Mixture reacts violently.[3]

Physiological Properties and Health Hazards

Severely irritates the eyes and causes skin burns. Causes severe internal irritation if taken by mouth.[2]

Spillage Disposal

Wearing eye protection, laboratory coat, and nitrile rubber gloves, cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Add the solid in small portions to a 1-L, three-necked, round-bottom flask containing 2.5 M sodium hydroxide (prepared by dissolving 60 g of NaOH in 600 mL of cold water) and equipped with a stirrer and thermometer (approximately 600 mL of sodium hydroxide per 64 mL of benzenesulfonyl chloride). If a reaction does not occur after the first few portions of the benzenesulfonyl chloride mixture are added, heat the flask on a steam bath to about 90°C. When the initially added material has dissolved, slowly add the remaining mixture. After addition is complete, continue heating until a clear solution has been obtained. Then cool the mixture to room temperature, neutralize with 10% hydrochloric, and wash the solution into the drain with at least 50 times its volume of water. Treat the solid residue as normal refuse.[4–6] Wash the area of the spill thoroughly with soap and water.

Waste Disposal

Package Lots. Place the compound in a separate container labeled for disposal by burning. Burn in an incinerator equipped with afterburner and scrubber.[7]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Sodium hydroxide solution (60 mL of 2.5 M) is added to a 100-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Benzenesulfonyl chloride (0.05 mol, 8.9 g, or 6.5 mL) is added dropwise. If the reaction is sluggish (no dissolution or rise in temperature) at first, heat the mixture on a steam bath to about 90°C. When the initially added benzenesulfonyl chloride has dissolved, add the remainder dropwise. After addition is complete, continue heating until a clear solution is obtained. Cool the mixture is cooled to room temperature, neutralize with 10% hydrochloric acid, and wash into the drain.[5]

Reactions for Spillage and Waste Disposal

$$C_6H_5SO_3Cl + 2NaOH \xrightarrow{H_2O} \underset{\text{sodium benzenesulfonate}}{C_6H_5SO_3^-Na^+} + NaCl + H_2O$$

REFERENCES

1. Merck 1072.
2. Lux 233.
3. Buckley, A., *J. Chem. Educ.*, 42, 674, 1965.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
5. PPL 161.
6. *Haz. Mat. Spills Tec.*, 29.5.
7. Ald 352C.

BENZIDINE AND SALTS $H_2N(C_6H_4)_2NH_2$

HUMAN CARCINOGENS

Physical Properties

Benzidine white or reddish crystalline powder; mp, 115–120°C (slow heating) and 128°C (rapid heating).[1]

Chemical Properties

One gram of amine dissolves in 2500 mL of cold water, and 107 mL of boiling water. Amine and salt are soluble in alcohol and ether.[1]

Physiological Properties and Health Hazards

Breathing the dust or its absorption through the skin may result in bladder tumors.[1–3] LD_{50} (oral, rat) 309 mg/kg; LD_{50} (oral, mouse) 214 mg/kg.[2] Exposure by all routes should be carefully controlled to the lowest possible levels.[4]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus.

On skin and clothing. Immediately wash skin with strong soap solution. Rinse thoroughly. Contaminated clothing should be removed, dried, and washed with strong soap solution or destroyed. It may be necessary to destroy shoes by burning.

Small spills. Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Scoop up the solid and package for disposal by burning. Wash site with a strong soap solution.[2,3,5]

Waste Disposal

Package Lots. Dissolve the benzidine in a flammable solvent and place in a separate labeled container for disposal by burning. Spray into a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Add water (0.7 mL), concentrated hydrochloric acid (2.1 mL), and benzidine (1.5 g, 0.0057 mol) to 50-mL, three-necked, round-bottom flask equipped with stirrer, thermometer, and dropping funnel. Maintain temperature at −5 to 0°C by a cooling bath, while adding dropwise 0.43 g (0.006 mol) of 97% sodium nitrite dissolved in 1 mL of water to the mixture of benzidine hydrochloride. Continue stirring 30 minutes more after addition is complete. While maintaining the temperature at −5 to 0°C, add 11.9 mL (0.11 mol) of 50% hypophosphorous acid (precooled to 0°C) over 10–15 minutes. Continue stirring for 1 hour. Allow the mixture to stand at room temperature for 24 hours, and then extract with 2 × 3 mL portions of toluene. Package the toluene extract of the deamination product, biphenyl, for disposal by burning. Wash the aqueous phase down the drain with at least 50 times its volume of water.[6]

To each 9 mg of benzidine, add 10 mL of 0.1 M hydrochloric acid (prepared by slowly adding 1 mL of concentrated acid to 119 mL of cold water). Mix to dissolve. Add 5 mL of 0.2 M potassium permanganate solution (0.3 g of solid potassium permanganate dissolved in 10 mL of water) and 5 mL of 2.0 M sulfuric acid (prepared by carefully adding 1 mL of concentrated acid to 8 mL of cold water). Mix and let stand overnight (at least 10 hours). Decolorize, if necessary, with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (20 g of NaOH dissolved in 100 mL of cold water). Wash the remaining solution into the drain.[7]

Reactions for Spillage and Waste Disposal

$$H_2N(C_6H_4)NH_2 \cdot 2HCl \xrightarrow[2.\ H_3PO_2]{1.\ NaNO_2} (C_6H_5)_2$$

biphenyl

REFERENCES

1. Merck 1077.
2. ITI 075.
3. Lux 234.
4. ACGIH 16.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *Laboratory Disposal of Benzodine,* University of Alberta, Edmonton, 1995.
6. PP 70.
7. Cas 64, 24; PPL 162.

BENZO[a]PYRENE $C_{20}H_{12}$

ANIMAL CARCINOGEN

Physical Properties

Yellowish plates or needles; mp, 179.3°C[1]; bp_{10}, 310–312°C.[1,2]

Chemical Properties

Practically insoluble in water.[1] Soluble in benzene, toluene, cyclohexane, acetone, dimethylsulfoxide, and dimethyformamide; slightly soluble in methanol and ethanol.[2]

Physiological Properties and Health Hazards

An experimental carcinogen, neoplastic, and mutagen.[1–3] It can induce pulmonary adenomas in mice after administration by any route.[4] Exposure by all routes should be carefully controlled to the lowest possible levels.[5]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Scoop up the solid and package for disposal by burning. Wash the site with strong soap solution.[6,7]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve the benzo[a]pyrene in a flammable solvent and spray into furnace with afterburner.[6,7]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Work in the fume hood. For each 5 mg of benzo[a]pyrene, add 2 mL of acetone and dissolve, while making sure that no solid adheres to walls. Add 10 mL of a freshly prepared solution of 0.3 M potassium permanganate (2.35 g of potassium permanganate/50 mL of acid) in 3 M sulfuric acid (8 mL of concentrated acid slowly and carefully added to 42 mL of cold water). Swirl the mixture and allow to react for at least 1 hour. During this period, additional potassium permanganate solution may be added, if necessary, to maintain the purple color. After color has been maintained for 30 minutes, decolorize with sodium metabisulfite. Neutralize the reaction mixture by careful addition of 5 M sodium hydroxide solution (20 g of sodium hydroxide pellets dissolved in 100 mL of water). Any remaining solid may be filtered off and discarded with normal refuse. The filtrate may be washed into the drain.[8]

REFERENCES

1. Merck 1104.
2. Cas 49, 41.
3. Sax 379.
4. Cas 49, 3.
5. ACGIH 16.
6. Ald 366D.
7. PP 60.
8. Cas 49, 19.

BENZOYL PEROXIDE $(C_6H_5CO_2)_2$

EXTREME RISK OF EXPLOSION BY SHOCK, FRICTION, FIRE, OR OTHER SOURCE OF IGNITION

Synonyms

Dibenzoyl peroxide.

Physical Properties

White granulated crystals, normally supplied as moistened with about 30% water; mp, 103–106°C (dec).[1]

Fire Hazard

Highly flammable when dry; explosive at elevated temperatures.[2]

Chemical Properties

Insoluble in water and alcohol[1–3]; soluble in benzene, chloroform, and ether.[1] Liable to explode when heated above melting point or when subjected to friction or shock when dry.[3] Contact with metal should be avoided.

Hazardous Reactions

Dry material burns readily and is sensitive to heat, shock, friction, or contact with combustible materials. It explodes above the melting point.[4,5] Benzoyl peroxide should not be recrystallized from hot chloroform, but rather precipitated from cold chloroform with methanol.[6] The peroxide should be stored in waxed paper tubs and never in metal or glass containers with screw-type closures.[5]

Aniline. Addition of 1 drop of aniline to 1 g of the peroxide results in a mild explosion.[7]

Carbon Tetrachloride and Ethylene. Explosion can result from heating a mixture with a small quantity of benzoyl peroxide.[8]

N,N-Dimethylaniline. Addition of a drop of the amine to solid peroxide results in an explosion.[9]

Dimethyl Sulfide. Explosive reaction in the absence of solvent.[10]

Lithium Aluminum Hydride. Mixture in ether may explode.[11]

Methyl Methacrylate. Exothermic reaction in the presence of oxidizable or polymerizable materials may cause ignition.[12]

Physiological Properties and Health Hazards

Contact with skin or eyes causes irritation. Avoid contact with skin and eyes.[3] TLV-TWA 5 mg/m^3.[13]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Dampen this mixture thoroughly with water, and then scoop into a beaker using a plastic or cardboard shovel. Treat as per waste disposal procedure. Wash the area of the spill thoroughly with soap and water.[14]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood or behind safety shield. Stir the dampened benzoyl peroxide with 10% sodium hydroxide solution (10 mL for each 1 g of peroxide) for 24 hours. The resultant sodium benzoate may be poured into the drain with at least 50 times its volume of water.[15]

Alternatively, react the dampened benzoyl peroxide with a solution of sodium or potassium iodide in acid. Gradually, and while stirring, add 1.0 g of peroxide to a solution of sodium iodide (1.4 g) or potassium iodide (1.5 g) in 28 mL of glacial acetic acid. The solution darkens rapidly due to formation of iodine. Allow to stand for 30 minutes. Add solid sodium metabisulfite until the solution is colorless. Wash the solution into the drain.[16]

Reactions for Spillage and Waste Disposal

1) $(C_6H_5CO_2)_2 + 2NaOH \xrightarrow{H_2O} 2C_6H_5COO^-Na^+ + O_2$
sodium benzoate

2) $2(C_6H_5CO_2)_2 + 2NaI \rightarrow 2C_6H_5COO^-Na^+ + I_2$
sodium benzoate

REFERENCES

1. Merck 1117.
2. NFPA 49.
3. Lux 321.
4. B 869.
5. Lappin, G.R., *Chem. Eng. News*, 26, 3518, 1948; Taub, D., *Chem. Eng. News*, 27, 46, 1949.
6. Nozaki, K. et al., *J. Am. Chem. Soc.*, 68, 1692, 1946.
7. Bailey, P.S. et al., *J. Chem. Educ.*, 52, 525, 1975.
8. Bolt, R.O. et al., *Chem. Eng. News*, 25, 1866, 1947.
9. Horner et al., *Chemische Berichte,* 86, 1071, 1953.
10. Pyror, W.A. et al., *J. Org. Chem.*, 37, 2885, 1972.
11. Sutton, D.A., *Chem. Ind.*, 272, 1951.
12. B 829.
13. ACGIH 17.
14. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
15. Steere, N.V., Ed., *Safety in the Chemical Laboratory*, Vol. 1, Division of Chemical Education of the American Chemical Society, Easton, PA, 1967, p. 71; ITI 082.
16. PP 75; PPL 163.

BENZYL BROMIDE $C_6H_5CH_2Br$

CAUSES BURNS

Synonyms

α-Bromotoluene.

Physical Properties

Colorless to pale yellow lachrymatory liquid; bp, 199°C.[1,2]

Chemical Properties

Immiscible with water.[2]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system, the skin, and eyes. It is a lachrymator. The liquid burns the skin. Swallowing would cause severe internal irritation and damage.[2] Large doses cause central nervous system depression.[1] Avoid breathing vapor. Prevent contact with skin and eyes.[2]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Depending on the size of the spill, breathing apparatus may be required. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the benzyl bromide has been absorbed, scoop the mixture into a plastic container and package for disposal by burning. Wash site of spillage with soap and water.[2–4]

Waste Disposal

Package Lots. Place in a separate, labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and burn in furnace equipped with afterburner and scrubber.[4,5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. While briskly stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. Heat the solution to gentle reflux and add the benzyl bromide (17.2 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring (to prevent bumping) for another 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[5,6]

Reactions for Spillage and Waste Disposal

$$C_6H_5CH_2Br + KOH \xrightarrow{C_2H_2OH} \underset{\text{benzyl alcohol}}{C_6H_5CH_2OH} + KBr$$

REFERENCES

1. Merck 1130.
2. Lux 238.

3. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
4. Ald 381B.
5. PP 61.
6. *Haz. Mat. Spills Tec.*, 29.5.

BENZYL CHLORIDE $C_6H_5CH_2Cl$

CAUSES BURNS

Synonyms

α-Chlorotoluene.

Physical Properties

Refractive liquid with unpleasant, irritating odor; bp, 179°C.[1]

Fire Hazard

Flash point, 67°C; lower flammable limit, 1.1%.[2]

Chemical Properties

Insoluble in water; miscible with alcohol, chloroform, and ether.[1]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system, the skin, and eyes. It is a lachrymator. The liquid burns the skin. Swallowing would cause severe internal irritation and damage.[3] Large doses can cause central nervous system depression.[1] Avoid breathing vapor. Prevent contact with skin and eyes.[3] TLV-TWA 1 ppm (5.2 mg/m^3).[4]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Depending on the size of the spill, breathing apparatus may be required. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the benzyl chloride has been absorbed, scoop the mixture into a plastic container and package for disposal by burning. Wash site of spillage with soap and water.[3,5,6]

Waste Disposal

Package Lots. Place in a separate, labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and burn in furnace equipped with afterburner and scrubber.[6,7]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. While briskly stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. Heat the solution to gentle reflux and add the benzyl chloride (12.6 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring (to prevent bumping) for another 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[7]

Reactions for Spillage and Waste Disposal

$$C_6H_5CH_2Cl + KOH \xrightarrow{C_2H_5OH} \underset{\text{benzyl alcohol}}{C_6H_5CH_2OH} + KCl$$

REFERENCES

1. Merck 1131.
2. NFPA 325M.
3. Lux 239.
4. ACGIH 17.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
6. Ald 382C.
7. PP 61.
8. *Haz. Mat. Spills Tec.*, 29.5.

BERYLLIUM (METAL) Be

ANIMAL CARCINOGEN

Synonyms

Glucinium.

Physical Properties

Gray metal; mp, 1287°C.[1]

Chemical Properties

Oxide film renders metal resistant to attack by acid.[1]

Hazardous Reactions

Carbon Dioxide, Nitrogen. Powdered beryllium ignites on heating in a mixture of CO_2 and N_2.[2]

Halocarbons. Powdered beryllium in carbon tetrachloride or trichloroethylene will ignite on heavy impact.[2]

Halogens. Beryllium incandesces in fluorine or chlorine when warm.[3]

Phosphorus. Beryllium incandesces when heated with phosphorus.[4]

Physiological Properties and Health Hazards

Death may result from short exposure to very low concentrations of the element and its salts. Short exposures may cause acute pneumonitis or chronic pulmonary granulomatous disease.[1] TLV-TWA 0.002 mg/m^3; TLV-STEL 0.01 mg/m^3.[5]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, self-contained respirator, and laboratory coat (after each wearing, launder clothing separately). Scoop up and place in large wide-mouth bottle with stopper. Save for recovery or disposal. Wash site with soap solution.[6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal in a secure landfill.[7]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve the compound in a minimum of 6 M hydrochloric acid (add concentrated acid to an equal volume of cold water). Filter and treat the filtrate with a slight excess of 6 M ammonium hydroxide (add 42 mL of concentrated ammonium hydroxide to 58 mL of cold water, about pH 8 to pHydrion paper). Boil and allow the coagulated precipitate to settle for about 12 hours. Filter and dry. Recycle, return to supplier, or send for disposal in a secure landfill.[6,7]

Reactions for Spillage and Waste Disposal

$$Be + 2Cl^- \rightarrow \underset{\substack{\text{beryllium}\\ \text{chloride}}}{BeCl_2}$$

$$BeCl_2 + 2OH^- \rightarrow \underset{\substack{\text{beryllium}\\ \text{hydroxide (insoluble)}}}{Be(OH)_2} + 2Cl^-$$

REFERENCES

1. Merck 1164.
2. B 85.
3. B 86.
4. Mellor, Vol. 7, 115, 1941; Vol. 8, 842, 847, 853, 1940; Vol. 8, Suppl. 3, 228, 1971.
5. ACGIH 17.
6. ITI 085.
7. PPL 167.

BLEOMYCIN $(A_2)C_{55}H_{84}N_{17}O_{21}S_2$

ANTINEOPLASTIC

Synonyms

NSC-125066, Bleo.[1]

Physical Properties

Colorless or yellowish powder that becomes bluish depending on Cu content.[1]

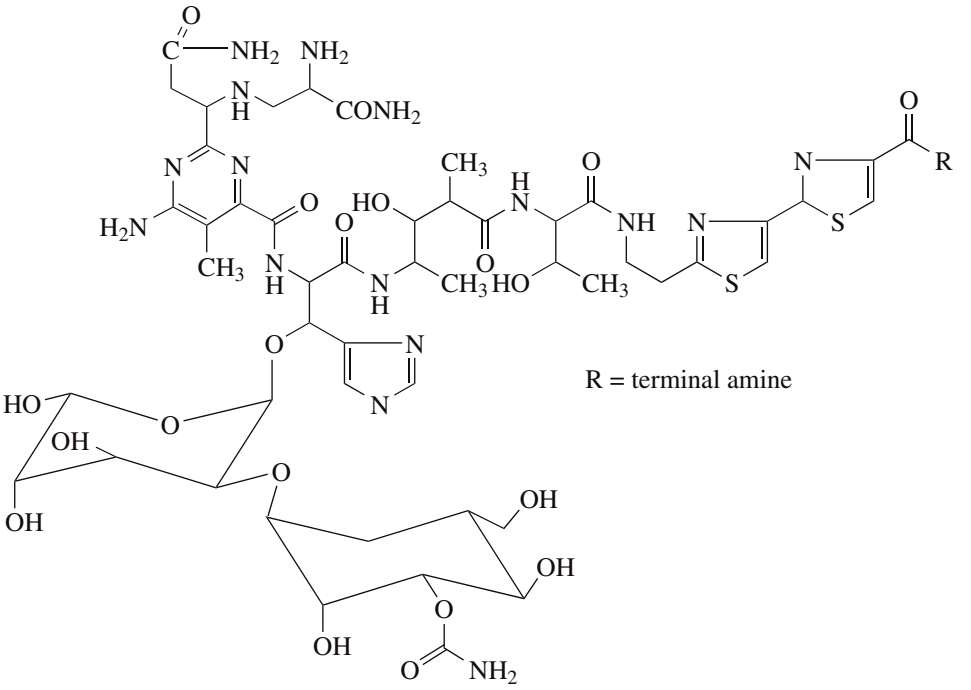
Chemical Properties

Very soluble in water and methanol; slightly soluble in ethanol; practically insoluble in acetone, ethyl acetate, butyl acetate, and ether.[1]

Mode of Action

Bleomycins are thought to react with DNA and cause strand scission; they have also been shown to have a type of oxygen transferase activity.[1]

Structure



Physiological Properties and Health Hazards

Fever (4–6 hours) and chills, anorexia, and vomiting. Patients over age 50 may have pulmonary complications.[2]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a beaker and transport to the fume hood. While stirring, slowly add 50% hydrochloric acid (5 mL of concentrated hydrochloric acid slowly added to 5 mL of cold water), allowing 10 mL of acid for each 1 g of absorbing mixture used. Boil gently for 4 hours, preferably under reflux. Cool, and neutralize the solution by careful addition of soda ash or 10% sodium hydroxide solution. Decant the liquid into the drain with water and discard solid as normal refuse.[3]

Waste Disposal

Wear rubber gloves, protective clothing, and goggles. Work in the fume hood. To 10 mL of an aqueous solution containing 10 mg of bleomycin (in a 100-mL, round-bottom flask) add 10 mL of 50% hydrochloric acid (5 mL of concentrated hydrochloric acid slowly added to 5 mL of cold water). Gently boil the mixture (preferably under reflux) for 4 hours. After cooling, neutralize the solution by slowly adding soda ash or 10% sodium hydroxide solution. Wash liquid into the drain water.[3]

REFERENCES

1. Merck 1311.
2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, 1982, p. 84.
3. *Haz. Mat. Spills Tec.*, 29.7.

BORON TRIBROMIDE BBr_3

TOXIC BY INHALATION, REACTS VIOLENTLY WITH WATER, CAUSES BURNS

Physical Properties

Colorless, fuming liquid with a pungent odor; bp, 90°C.[1]

Chemical Properties

Reacts violently with water.[2] Decomposed by alcohol.[1]

Hazardous Reactions

Sodium. Mixture with sodium metal explodes on impact.[3]

Water. Reacts violently when poured into an excess of water and explosively if water is poured into it. Vessels containing boron tribromide should be cleaned with dry nonpolar solvents, never with water.[4,5]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and the eyes. The liquid burns the skin and eyes. Swallowing would result in severe internal burning. Avoid breathing vapor. Prevent contact with skin and eyes.[2] TLV-STEL-C 1 ppm (10 mg/m^3).[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the boron tribromide has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[7,8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal. Do not place in landfill.[9]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the boron tribromide into a large evaporating dish. Cover with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.[7]

Reactions for Spillage and Waste Disposal

$$2BBr_3 + 3Na_2CO_3 + 3H_2O \rightarrow \underset{\text{boric acid}}{2B(OH)_3} + 6NaBr + 3CO_2$$

REFERENCES

1. Merck 1337.
2. Lux 246.

3. Cueilleron, J., *Bull. Soc. Chem. Fr.*, 12, 88, 1945.
4. B 58.
5. Anonymous, *Lab. Pract.*, 15, 797, 1966.
6. ACGIH 18.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J Chem. Educ.*, 65, A64, 1988.
8. *Haz. Mat. Spills Tec.*, 29.5.
9. PP 91.

BORON TRICHLORIDE BCl_3

TOXIC BY INHALATION, CAUSES BURNS

Physical Properties

Colorless fuming liquid or gas with a pungent odor; bp, 12.5°C.[1]

Chemical Properties

Reacts rapidly with water, forming boric and hydrochloric acids.[2] Decomposed by alcohol.[1]

Hazardous Reactions

Aniline. Reacts violently with aniline.[3]

Phosphine. Reacts violently with phosphine.[2]

Physiological Properties and Health Hazards

The gas irritates the eyes, skin, and respiratory system. The liquid irritates or burns the skin and burns the eyes. Swallowing would result in severe internal burning. Avoid breathing gas. Prevent contact with skin and eyes.[2]

Spillage Disposal

In warm weather, it will exist as a gas, in which case instruct others to keep out of affected area. Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the boron trichloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[2–5]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal. Do not place in landfill.[5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the boron trichloride into a large evaporating dish. Cover the boron trichloride with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.

Reactions for Spillage and Waste Disposal

$$2BCl_3 + 3Na_2CO_3 + 3H_2O \rightarrow \underset{\text{boric acid}}{2B(OH)_3} + 6NaCl + 3CO_2$$

REFERENCES

1. Merck 1338.
2. Lux 246.
3. Jones, R.G., *J. Am. Chem. Soc.*, 61, 1378, 1939.

4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J Chem. Educ.*, 65, A64, 1988.
5. *Haz. Mat. Spills Tec.*, 29.5.
6. PP 91.

BORON TRIFLUORIDE BF_3

TOXIC BY INHALATION

Physical Properties

Colorless, fuming gas with pungent suffocating odor; bp, –100.4°C.[1,2]

Chemical Properties

Soluble in water with some decomposition forming fluoroboric and boric acids; somewhat soluble in concentrated sulfuric acid and nitric acid; soluble in benzene, dichlorobenzene, chloroform, carbon tetrachloride, and carbon disulfide.[1,2]

Hazardous Reactions

Alkali Metals or Alkaline Earth Metals (not Magnesium). Interaction without cooling causes incandescence.[2]

Physiological Properties and Health Hazards

The gas irritates the skin, eyes, and respiratory system; at high concentrations it may burn the skin. Prevent inhalation of gas. Prevent contact with skin and eyes.[2] TLV-STEL-C 1 ppm (2.8 mg/m^3).[3]

Waste Disposal

Seal cylinder and return to supplier. In the fume hood, surplus gas or leaking cylinder can be slowly added to a flask containing water. Precipitate the fluoride in solution by adding calcium chloride solution (10%). Let stand overnight. Filter off precipitate and send to a landfill. Wash the filtrate into the drain.[4]

Boron Trifluoride Complexes

The liquid complex formed between boron trifluoride and acetic acid, diethyl ether, methanol, and propanol all display hazards and toxic effects associated with their constituents. All are readily hydrolyzed by water, corrosive, and, to some degree, flammable.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the boron trifluoride complex has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[5]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the boron trifluoride complex into a large evaporating dish. Cover the boron trifluoride complex with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.[2–5]

Reactions for Spillage and Waste Disposal

$$BF_3 + 3CaCl_2 + 6H_2O \rightarrow \underset{\text{(insoluble)}}{3CaF_2} + \underset{\text{acid}}{2B(OH)_3} + 6HCl$$

REFERENCES

1. Merck 13513391.
2. Lux 246; ITI 090.
3. ACGIH 18.
4. PPL 170.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J Chem. Educ.*, 65, A64, 1988.

BROMINE Br_2

GIVES OFF VERY POISONOUS VAPOR, CAUSES SEVERE BURNS

Physical Properties

Dark reddish-brown fuming liquid with suffocating odor; bp, 59.47°C.[1]

Chemical Properties

Slightly soluble in water; freely soluble in alcohol, ether, chloroform, carbon tetrachloride, carbon disulfide, and concentrated hydrochloric acid.[1]

Hazardous Reactions

Acetone. Excess of bromine causes sudden violent reaction.[2]

Acrylonitrile. Violent polymerization may result.[3]

Ammonia. Explosive red oil formed on cooling to –95°C after interaction at room temperature.[4]

Diethyl Ether. Violent reaction may occur on addition of bromine.[5]

N,N-Dimethylformamide. Highly exothermic reaction to yield hydroxymethylenedimethyl-ammonium bromide.[6]

Ethanol and Phosphorus. Dangerous mixture formed.[7]

Hydrogen. Mixture may be explosive.[8]

Metal Acetylides and Carbides. Mono- and di-alkali metal acetylides and copper acetylides ignite in liquid bromine or its vapor. Alkaline earth, iron, uranium, and zirconium carbides ignite in the vapor.[9]

Metal Azides. Vapor with silver or sodium azide forms explosive bromine azide.[10]

Metals. Impact-sensitive mixtures are formed from lithium or sodium in dry bromine.[11] Potassium, germanium, antimony, and rubidium ignite in bromine vapor.[12] Violent reaction occurs with aluminum, mercury, or titanium.[13]

Methanol. Vigorously exothermic reaction on mixing the liquids.[14]

Nonmetal Hydrides. At room temperature, violent explosion and ignition occur with silane and its homologs[15,16] and with germane.[17]

Rubber. Violent reaction with natural rubber; slower reaction with synthetic rubbers.[18]

Trialkylboranes. Lower homologs tend to ignite in bromine.[18]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and the eyes. The liquid burns the skin and eyes. Swallowing would cause severe burns and internal damage. Prevent vapor inhalation. Prevent contact with eyes and skin. TLV-TWA 0.1 ppm (0.66 mg/m^3); TLV-STEL 0.2 ppm (1.3 mg/m^3).[19]

Spillage Disposal

Instruct others to maintain a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the bromine has been absorbed, quickly scoop into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Add 10% sodium bisulfite until solution turns colorless. Test the pH and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse. Wash the spill area thoroughly with soap and water.[20]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Add bromine (5 mL) to a large quantity of water (1 L). Slowly add 10% sodium bisulfite (about 120 mL) until all color disappears. When reaction subsides, neutralize with sodium carbonate and wash into the drain.[21]

Reactions for Spillage and Waste Disposal

$$Br_2 + 2NaHSO_3 \rightarrow \underset{\text{sodium bromide}}{2NaBr} + 2H^+ + SO_4^{2-} + SO_2$$

REFERENCES

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6. Tayim, H.A. et al., *Chem. Ind.*, 347, 1973.
7. Read, C.W.W., *School Sci. Rev.*, 21, 967, 1940.
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14. Muir, G.D., *Chem. Brit.*, 8, 136, 1972.
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17. Geisler, T.C. et al., *Inorg. Chem.*, 11, 1710, 1972.
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19. ACGIH 18.
20. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A. in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 284; Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 25, 1999; *Haz. Mat. Spills Tec.*, 29.5.
21. Ald 2027B; ITI 091.

N-BROMOSUCCINIMIDE $(-CH_2CO)_2NBr$

CAUSES BURNS, SEVERE IRRITANT

Physical Properties

White to pale buff crystalline solid smelling faintly of bromine[1]; mp, 173–175°C with decomposition.[2]

Chemical Properties

Slightly soluble in water; soluble in acetone and glacial acetic acid.[2]

Hazardous Reactions

Aniline. Extremely violent or explosive reaction.

Diallyl Sulfide. Extremely violent or explosive reaction.

Dibenzoyl Hydrate. A 30-fold increase in scale of a published method for radical-initiated, side-chain bromination of the acid in carbon tetrachloride led to violent reflux and eruption of the flask contents through the condenser.

Hydrazine Hydrate. Extremely violent or explosive reaction.

Propionitrile. After refluxing for 24 hours at 105°C, a mixture exploded, possibly due to dehydrohalogenation of the bromonitrile to acrylonitrile and subsequent polymerization.[3]

Physiological Properties and Health Hazards

Irritates the skin, eyes and respiratory system. Swallowing would result in internal irritation. Avoid breathing dust. Prevent contact with skin or eyes.[1]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Brush the spill into a container and transport to the fume hood. Follow waste disposal procedure.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning in furnace equipped with afterburner and scrubber.[4]

Small Quantities. Wear nitrile rubber gloves, eye protection, and laboratory coat. Work in the fume hood. Prepare solution of sodium sulfite in water (use 1.5 g of sodium sulfite in 10 mL of water for each 1 g of N-bromosuccinimide). Slowly, and while stirring, add N-bromosuccinimide to sodium sulfite solution. When the reaction is complete, wash the solution into the drain.[5]

Reactions for Spillage and Waste Disposal

$$(-CH_2CO)_2NBr + Na_2SO_3 + H_2O \rightarrow \underset{\text{succinimide}}{(-CH_2CO)_2NH} + Na_2SO_4 + HBr$$

REFERENCES

1. Lux 252.
2. Merck 1428.
3. B 423.

4. Ald 580A.
5. Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 279.

tert-BUTYL AZIDOFORMATE $N_3COOC(CH_3)_3$

EXPLOSIVE

Physical Properties

Bp, 73–74°C/70 mm.[1]

Hazardous Reactions

May explode during distillation.[1] Can be prepared without being distilled.[2] Fenlon (Eastman Kodak)[3] warns that this substance is extremely shock-sensitive and unstable between 100° and 135°C. 2-(*tert*-Butoxycarbonyloximino)2-phenylacetonitrile is recommended as a substitute.[4,5]

A serious detonation of undetermined origin during the preparation of the compound has been reported.[6]

Spillage Disposal

Wear leather gloves, eye protection, and laboratory coat. Work from behind body shield. Avoid unnecessary heat, friction, or impact. Absorb the liquid on paper towel. Place paper towel in a beaker containing concentrated hydrochloric acid (about 100 mL of acid for each 1 g of azide). Follow waste disposal procedure. Wash spill site thoroughly with soap and water.[7,8]

Waste Disposal

Keep stock of azide very low. Stamp date of receipt on package.

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood from behind safety shield. Add azide to concentrated hydrochloric acid (100 mL acid/g azide). While stirring, add granular tin (2.5 g/g azide). Allow the mixture to stir for 3 hours. Remove the unused tin by filtration and pour the filtrate into a pail of cold water. Neutralize the solution with sodium carbonate and wash into the drain. Discard or reuse recovered tin.[8]

Reactions for Spillage and Waste Disposal

$$N_3COOC(CH_3)_3 + Sn + 3HCl \rightarrow \underset{\substack{\textit{tert}\text{-butyl aminoformate}\\ \text{hydrochloride}}}{HCl \cdot NH_2COOC(CH_3)_3} + SnCl_2 + N_2$$

REFERENCES

1. Carpino, L.A. et al., *Org. Synth.*, 44, 15, 1964.
2. Sakai, K. et al., *J. Org. Chem.*, 36, 2387, 1971.
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4. Koppel, H.C., *Chem. Eng. News*, 54, 5, 1976.
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7. F & F 1, 84; 2, 44; 3, 36; 4, 54; 6, 77.
8. Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 282.

tert-BUTYL HYDROPEROXIDE $(CH_3)_3COOH$

FLAMMABLE

Physical Properties

Colorless liquid; stable below 75°C; mp, –8°C.[1]

Fire Hazard

Flash point, 27°C. Extinguish fire with water spray, dry chemical, carbon dioxide, or vaporizing liquids.[2]

Chemical Properties

Slightly soluble in water[3]; very soluble in esters and alcohols.[4]

Hazardous Reactions

May explode when distilled.[5,6]

Physiological Properties and Health Hazards

The liquid irritates the eyes and skin. Poisonous if swallowed. Avoid contact with skin, eyes, and clothing.[3]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work from behind body shield. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Carefully transfer to a beaker using a plastic scoop. Follow waste disposal procedure.[7] Wash the spill area thoroughly with soap and water.

Waste Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Work from behind heavy body shield in the fume hood. Cautiously add peroxide to 10% aqueous sodium bisulfite (10 mL/g peroxide). Continue stirring for 24 hours. The resulting solution may be washed into the drain.[8]

Reactions for Spillage and Waste Disposal

$$(CH_3)_3COOH + NaHSO_3 \rightarrow \underset{\textit{tert}\text{-butyl alcohol}}{(CH_3)_3COH} + NaHSO_4$$

REFERENCES

1. Merck 1569.
2. NFPA 325M.
3. Lux 261.
4. Sax 447.
5. Milas, N.A. et al., *J. Am. Chem. Soc.*, 68, 205, 1946.
6. B 488.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985.
8. PPL 170.

tert-BUTYL HYPOCHLORITE $(CH_3)_3COCl$

STORE IN A REFRIGERATOR IN THE DARK

Physical Properties

Pale yellow liquid with irritating odor; bp, 77–78°C.[1]

Hazardous Reactions

Ampoules liable to burst unless stored in a cool, dark place. It should not be heated above its boiling point.[2,3] Violent reaction if exposed to strong light or overheating.[1]
Rubber. Violent reaction.[3]

Physiological Properties and Health Hazards:

Vapor irritates mucous membranes and eyes.[1]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work from behind heavy body shield. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Carefully scoop mixture into a beaker of cold water in the fume hood. Add a 10% solution of sodium bisulfite, allowing 10 mL of bisulfite solution for each 1 mL of hypochlorite spilled. Let stand until solids settle. Decant the liquid into the drain. The solid residue can be discarded as normal refuse.[4] Wash the spill area thoroughly with soap and water.

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

Slowly add the t-butyl hypochlorite to an excess of a stirred, cold 10% solution of sodium bisulfite. Allow 10 mL of the bisulfite solution for each 1 mL of hypochlorite. When reaction has ceased, wash into the drain.[3]

t-Butyl hypochlorite also decomposes with UV light.[2,3,5]

REFERENCES

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3. Mintz, M.J. et al., *Org. Synth.*, Coll. Vol. 5, 185, 1973.
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5. F & F 1, 90.

CADMIUM Cd

EXPERIMENTAL CARCINOGEN, TOXIC

Physical Properties

Silver-white, blue-tinged, lustrous metal; easily cut with a knife; mp, 321°C.[1]

Fire Hazard

Finely divided metal is pyrophoric.[2]

Chemical Properties

Insoluble in water; reacts readily with dilute nitric acid; reacts slowly with hot hydrochloric and hot sulfuric acids but does not react with alkalies.[1]

Hazardous Reactions

Ammonium Nitrate. Violent or explosive reaction between fused ammonium nitrate and cadmium powder.[3]

Nitryl Fluoride. Cadmium incandesces in contact with gas.[4]

Selenium or Tellurium. Exothermic reaction on warming with powdered cadmium.[5]

Physiological Properties and Health Hazards

Inhalation of dust irritates lungs. Ingestion causes abdominal pain and diarrhea. Highly toxic.[1] TLV-TWA 0.01 (Cd)/m^3 (total dust); TLV-TWA 0.002 (Cd)/m^3 (respirable fraction of dust).[6]

Spillage Disposal

Metal Powder: Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Scoop into container for reuse or recycling.

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Package securely, label, and then recycle or reuse.

REFERENCES

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6. ACGIH 19.

CADMIUM COMPOUNDS Cd^{2+}

HARMFUL BY INHALATION, HARMFUL IF TAKEN INTERNALLY

Chemical Properties

Chloride, nitrate, and sulfate soluble in water; oxide and carbonate insoluble.[1]

Hazardous Reactions

Cadmium Amide $Cd(NH_2)_2$.
Rapid heating may result in explosion.[2]
Water. Violent reaction with water.[2]

Cadmium Azide $Cd(N_3)_2$.
The dry solid explodes on heating or with light friction. Contact of cadmium rods with aqueous hydrogen azide caused a violent explosion.[3]
Sodium Azide. A mixture of saturated solutions of cadmium and sodium azides explodes several hours after mixing.[4]

Cadmium Chlorate $Cd(ClO_3)_2$
Sulfides. Explosive interaction with copper sulfide; incandescent interaction with antimony(III), arsenic(III), tin(II), and tin(IV) sulfides.[5]

Cadmium Hydride CdH_2
Sudden decomposition of the hydride occurs at 2°C, forming residue of pyrophoric cadmium.[6]

Cadmium Nitrate $Cd(NO_3)_2$
Nitrate may explode when exposed to shock, heat, or flame, or by spontaneous chemical reaction.[7]

Cadmium Oxide CdO
Magnesium. Cadmium oxide is reduced explosively on heating with magnesium.[8]

Cadmium Phosphide Cd_3P_2
Nitric Acid. Reaction with concentrated acid is explosive.[9]

Cadmium Propionate $Cd(C_2H_5COO)_2$
The salt exploded during drying in an oven at 60–100°C.[10]

Cadmium Selenide CdSe
Explosion may occur on heating cadmium and selenium together unless the particles are smaller than a critical size.[11]

Physiological Properties and Health Hazards

Dust (usually cadmium or cadmium oxide) irritates lungs. Swallowing any cadmium compound causes choking, stomach pains, vomiting, and diarrhea. Prolonged exposure to the dust may result in lung and kidney damage and discoloration of teeth.

Avoid inhaling dust.[12] TLV-TWA 0.01 (Cd)/m^3 (total dust); TLV-TWA 0.002 (Cd/m^3 [respirable fraction of dust]).[13]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Avoid breathing dust. Cadmium compounds are not so toxic as to present serious disposal problems. The insoluble compounds can be mixed with wet sand, swept up, and treated as normal waste. The soluble salts can be mopped

up with water, and a 10% aqueous solution of sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$) added until no further precipitation occurs. Adjust the pH to 11.0 with 2 M sulfuric acid. The solid is collected by filtration or by allowing the filtrate to evaporate in the fume hood. It is dried, packaged, and labeled for disposal in a secure landfill.[14]

Waste Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Work in the fume hood. Dissolve the soluble cadmium salt (0.05 mol, e.g., 9 g of cadmium chloride) in water (50 mL) and add solution of sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$, 25 g, 0.12 mol) in water (200 mL). A white precipitate of cadmium silicate forms immediately. Heat the mixture to 80°C for 15 minutes to complete the reaction. Cool and adjust to pH 11 with 2 M sulfuric acid. Collect the precipitate by filtration or allow the supernatant liquid to evaporate in the fume hood. Allow the solid to dry, and then package and label for disposal in a secure landfill.

For dilute solutions of cadmium salts, add sodium metasilicate solution until there is no further precipitation, and allow the solution to stand overnight before collecting the solid by filtration or allow the filtrate to evaporate in the fume hood.[14]

Reactions for Spillage and Waste Disposal

$$CdCl_2 + Na_2SiO_3 \cdot 5H_2O \rightarrow \underset{\substack{\text{cadmium}\\ \text{silicate}\\ \text{(insoluble)}}}{CdSiO_3} + 2NaCl + 5H_2O$$

REFERENCES

1. Merck 1614–1630.
2. Mellor, Vol. 8, 261, 1940.
3. Mellor, Vol. 8, Suppl. 2.2, 25, 50, 1967.
4. Turney, T.A., *Chem. Ind.*, 1295, 1965.
5. Mellor, Vol. 2, Suppl. 1, 584, 1965.
6. Barbaras, G.D. et al., *J. Am. Chem. Soc.*, 73, 4585, 1951.
7. Sax 654.
8. Mellor, Vol. 3, 138, 378, 1941; Vol. 4, 272, 1940; Vol. 7, 401, 1941.
9. Juza, R. et al., *Zeitschrift fur Anorganische Chemie*, 283, 230, 1956.
10. B 612.
11. Reisman, A. et al., *J. Phys. Chem.*, 67, 22, 1963.
12. Lux 267.
13. ACGIH 18.
14. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.

CALCIUM CARBIDE CaC_2

LIBERATES EXPLOSIVE GAS ON CONTACT WITH WATER

Synonyms

Calcium acetylide.

Physical Properties

Grayish-black, irregular lumps or orthorhombic crystals; mp, 2300°C.[1]

Fire Hazard

Liberates acetylene on contact with moisture. Use dry chemical for fire fighting. Do not use water or carbon dioxide. Fire may also be smothered using dry sand, clay, or ground limestone.[2]

Chemical Properties

Decomposed by water with evolution of acetylene, leaving a residue of lime.[1]

Hazardous Reactions

Nonferrous tools must be used to open containers.[3]

Halogens. Incandesces with Cl_2, Br_2, and I_2 at 245, 350, and 305°C, respectively.[4]

Hydrogen Chloride. Incandesces on warming.[4]

Iron(III) Chloride and Iron(III) Oxide. Powdered mixture burns violently when ignited.[5]

Magnesium. Mixture incandesces on warming.[6]

Methanol. Very vigorous reaction with boiling methanol.[5]

Silver Nitrate. Precipitates the highly sensitive explosive, silver acetylide, from silver nitrate solutions.[5]

Sodium Peroxide. A mixture is explosive.[7]

Tin Dichloride. A mixture can be ignited with a match.[8]

Water. Forms explosive mixture due to generation of acetylene.[9]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container and remove to an open area. Add slowly to a large container of water. After 24 hours, decant the liquid to the drain. Bury the solid in a landfill site.[10,11]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Five grams of calcium carbide are suspended in 60 mL of a hydrocarbon (toluene or cyclohexane) in a 200-mL, three-necked, round-bottom flask equipped with ice bath, stirrer, dropping funnel, nitrogen inlet, and gas outlet leading through plastic tubing to back of the fume hood that the equipment is in. With a moderate flow of nitrogen passing through the flask to carry off the acetylene generated, add dropwise 30 mL of 6 N hydrochloric acid (cautiously add

concentrated acid to an equal volume of cold water) over a period of about 5 hours, and stir mixture for an additional hour. The aqueous and hydrocarbon layers are separated. Package the hydrocarbon layer for disposal by burning. The aqueous layer is neutralized and washed into the drain.[12]

Reactions for Spillage and Waste Disposal

$$CaC_2 + 2HCl \rightarrow \underset{\text{acetylene}}{HC{\equiv}CH} + CaCl_2$$

REFERENCES

1. Merck 1657.
2. NFPA 49.
3. B 204.
4. Mellor, Vol. 5, 862, 1946.
5. B 204.
6. Mellor, Vol. 4, 271, 1940.
7. Mellor, Vol. 2, 490, 1941.
8. Mellor, Vol. 7, 430, 1941.
9. B 205.
10. *Haz. Mat. Spills Tec.*, 29.1.
11. ITI 120.
12. PP 96.

CALCIUM HYDRIDE CaH_2

CONTACT WITH WATER LIBERATES FLAMMABLE GAS

Physical Properties

Orthorhombic crystals or powder; the commercial product is gray; mp, 186°C.[1]

Chemical Properties

Decomposes with water, lower alcohols, and carboxylic acids to form hydrogen; moderately powerful condensing agent with ketones and acid esters; more powerful reducing agent toward metal oxides than lithium or sodium hydrides.[1]

Hazardous Reactions

Manganese Dioxide. Mixture incandesces on warming.[2]

Metal Halogenates. Mixtures with variety of bromates, chlorates, or perchlorates explode on grinding.[3]

Silver Halides. Mixture with silver fluoride incandesces on grinding. Mixture with silver iodide reacts vigorously on heating.[4]

Tetrahydrofuran. The hydride does not prevent peroxide formation during storage of tetrahydrofuran dried over lithium aluminum hydride. Addition of more drying agent to tetrahydrofuran thus stored can result in ignition of liberated hydrogen.[5]

Water. Contact with water liberates highly flammable hydrogen gas.[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a plastic pail and transport to the fume hood. Slowly and while stirring, add the mixture to a beaker of dry butanol or methanol. When reaction has ceased, decant the liquid into a pail of cold water, and then wash into the drain. Treat the solid as normal refuse.[6–8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.[7]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place the hydride in a three-necked, round-bottom flask equipped with nitrogen inlet, dropping funnel, and condenser. Under nitrogen, and while stirring, add methanol dropwise allowing 25 mL for each 1 g of hydride. When the reaction is complete, slowly add an equal volume of water to the slurry of calcium methoxide. Wash the solution into the drain.[9]

Reactions for Spillage and Waste Disposal

$$CaH_2 + 2CH_3OH \rightarrow Ca(OCH_3)_2 + 2H_2$$

$$Ca(OCH_3)_2 + 2H_2O \rightarrow \underset{\substack{\text{calcium}\\\text{hydroxide}\\\text{(insoluble)}}}{Ca(OH)_2} + 2CH_3OH$$

REFERENCES

1. Merck 1674.
2. B 1330.
3. Mellor, Vol. 3, 651, 1946.
4. Mellor, Vol. 3, 389, 651, 1941.
5. Moffett, A.B., *Chem. Eng. News*, 32, 4328, 1954.
6. Lux 276.
7. Ald 667A.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
9. PPL 163.

CALCIUM HYDROXIDE $Ca(OH)_2$

CORROSIVE

Synonyms

Calcium hydrate, slaked lime.[1]

Physical Properties

Crystals or soft, odorless, granules or powder. Slightly bitter, alkaline taste. Readily absorbs CO_2 from air, forming $CaCO_3$.[1]

Chemical Properties

Slightly soluble in water; soluble in glycerol, sugar, or ammonium chloride solutions; soluble in acid with evolution of much heat.[1]

Hazardous Reactions

Violent reaction with maleic anhydride, nitroethane, nitromethane, nitroparaffins, nitropropane, and phosphorous. Reaction with polychlorinated phenols and potassium nitrate forms extremely toxic products.[2,3]

Physiological Properties and Health Hazards

Dust can irritate the skin,[4] eyes, and mucous membranes.[2] TLV-TWA 5 mg/m^3.[5]

Spillage Disposal

Aqueous Solutions of Calcium Hydroxide. Wear nitrile rubber gloves, laboratory coat, and goggles. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite) and sand. When the mixture has been completely absorbed, scoop it into a plastic pail. Add water to dissolve the calcium hydroxide and sodium carbonate. Allow to stand for 24 hours. Test the pH and neutralize with 2 M hydrochloric acid (1 volume of concentrated hydrochloric acid to 5 volumes of water). Decant the solution to the drain with large quantities of water.[6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Dissolve in water to give a 10% solution. Slowly, and while stirring, neutralize by adding 2 M hydrochloric acid (1 volume of concentrated hydrochloric acid added to 5 volumes of water). Wash into the drain.

Reactions for Spillage and Waste Disposal

$$Ca(OH)_2 + 2HCl \rightarrow CaCl_2 + 2H_2O$$

REFERENCES

1. Merck 1675.
2. Sax 668.
3. B 1489.
4. ITI 123.

5. ACGIH 19.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.4.

CAPTAN $C_9H_8Cl_3NO_2S$

FUNGICIDE

Synonyms

cis-N-((Trichloromethyl)thio)-4-cyclohexene-1,2-dicarboximide, Merpan, 1N-trichloromethylthiotetrahydrophthalamide, SR406, orthocide 406.

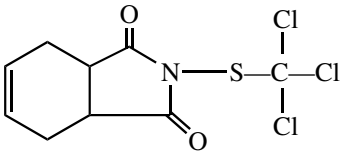
Physical Properties

Odorless crystals from carbon tetrachloride; mp, 178°C.[1]

Chemical Properties

Solubility in water at 25°C, 3.3 mg/L. Practically insoluble in petroleum oils; soluble in xylene and chloroform.[1,2]

Structure



Physiological Properties and Health Hazards

LD_{50} 9,000 mg/kg (oral, rat)[2]; TLV-TWA 5 mg/m^3.[3]

Formulations

Wettable powder, dustable powder.[2]

Waste Disposal

Waste quantities of Captan can be degraded using either chlorine bleach or potassium permanganate.

Chlorine Bleach. Wear gloves and eye protection. For each 100 mg of pure Captan or commercial wettable powder, add 100 mL of chlorine bleach (5% sodium hypochlorite) and stir the mixture at room temperature for 64 hours. The resulting solution is washed into the drain with water.[4]

Potassium Permanganate. Wear gloves and eye protection. For each 100 mg of pure Captan or commercial wettable powder, add 100 mL of acidic potassium permanganate solution (17 mL of concentrated sulfuric acid added to 70 mL of cold water, 4.7 g of potassium permanganate dissolved in the solution, and volume brought to 100 mL with water). The mixture is stirred at room temperature for 30 minutes, and then the excess permanganate is reduced by adding solid sodium metabisulfite until the solution is colorless. The solution is neutralized by the careful and slow addition of sodium carbonate or 5% aqueous sodium hydroxide solution and the resulting liquid is washed into the drain.[4]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family

wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level, and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer before washing other clothes by running the washing machine through a complete wash cycle with detergent (but without clothing).[4]

A 0.12% Captan solution (aqueous suspension) was used to contaminate denim and Goretex fabric.[4] A single wash results in less than 1% Captan residues remaining on the Goretex, regardless of whether a hot (60°C) or warm wash (49°C) is used. For denim fabric, the hot wash leaves less than 1% residues and the warm wash leaves approximately 2% residues.[5]

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CARBON DISULFIDE CS_2

EXTREMELY FLAMMABLE, GIVES OFF VERY POISONOUS VAPOR

Physical Properties

Colorless to yellow liquid with unpleasant odor; bp, 46.5°C.[1]

Fire Hazard

Flash point, –30°C; flammable limits, 1.3–50%; ignition temperature, 90°C. Extinguish fire with foam, dry powder, carbon dioxide, or water spray. Foam and water may be ineffective.[2]

Chemical Properties

Immiscible with water; decomposes on long standing. Miscible with many common organic solvents.[1]

Hazardous Reactions

Many fires and explosions have resulted by ignition of the vapor from liquid poured down laboratory sinks. Highly volatile and highly flammable. The vapor may ignite on contact with steam pipes, especially if rusted.[3]

Air and Rust. A mixture of carbon disulfide and air can explode in the presence of rust.[4]

Halogens. Mixtures of chlorine and carbon disulfide may explode in the presence of iron.[5] Carbon disulfide vapor ignites on contact with fluorine.[6]

Metal Azides. Mixtures of carbon disulfide and aqueous solutions of metal azides form explosive metal azidodithioformates.[7]

Metals. Aluminum powder ignites in carbon disulfide vapor.[8] Mixtures of carbon disulfide with K-Na alloy[9] or with Na itself[10] are powerful explosives. Zinc incandesces in carbon disulfide.[11]

Oxidants. A mixture of carbon disulfide and nitric oxide may explode.[12] Mixtures with nitrogen dioxide are used as explosives.[13]

Physiological Properties and Health Hazards

Vapor irritates the eyes and produces drowsiness, eventually resulting in unconsciousness. Liquid irritates the eyes and is poisonous if swallowed. Prolonged exposure to the vapor results in disruption of nervous system. Avoid breathing vapor.[14] TLV-TWA 10 ppm (31 mg/m^3).[15]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and Viton® rubber gloves.[16] Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Place mixture in an evaporating dish in the fume hood to evaporate. Ventilate the spill area thoroughly to dispel vapor.[17]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear Viton® rubber gloves,[16] laboratory coat, and eye protection. Work in the fume hood. For each 0.05 mol (3.8 g, 3.0 mL) of carbon disulfide to be destroyed, use 670 mL of sodium hypochlorite (bleach) or a mixture of 55 g of 65% calcium hypochlorite in 220 mL of water. Place the hypochlorite in flask equipped with dropping funnel, stirrer, and thermometer,

and add the carbon disulfide dropwise such that the reaction temperature is maintained between 20 and 30°C (to avoid volatilizing of the carbon disulfide). When addition is complete, continue stirring for 2 hours or until a clear, homogeneous solution remains (perhaps containing traces of oily by-products). The reaction mixture can be washed into the drain.[18]

If quantity is large, carbon disulfide may be recovered by distillation and repackaged for use.[18]

Reactions for Spillage and Waste Disposal

$$CS_2 + 8OCl^- + 2H_2O \rightarrow CO_2 + 2H_2SO_4 + 8Cl^-$$

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CARBON TETRACHLORIDE CCl_4

ANIMAL CARCINOGEN, GIVES OFF POISONOUS VAPOR

Synonyms

Tetrachloromethane.

Physical Properties

Heavy, colorless liquid with a characteristic odor; bp, 76.7°C.[1]

Chemical Properties

Immiscible with water.

CAUTION: When used for extinguishing fires, phosgene, which is very poisonous, is liable to be formed.[1]

Hazardous Reactions

Aluminum Chloride and Triethylaluminum. A mixture in carbon tetrachloride exploded when warmed to room temperature.[2]

Benzoyl Peroxide and Ethylene. Mixtures of ethylene and carbon tetrachloride can explode at elevated temperatures on initiation with benzoyl peroxide.[3]

Boranes. Explosions may result from the use of carbon tetrachloride as an eluting solvent in the chromatographic separation of boranes.[4]

Chlorine Trifluoride. Solutions of chlorine trifluoride in carbon tetrachloride can be detonated.[5]

Decaborane. Impact-sensitive mixture formed with carbon tetrachloride.[6]

1,11-Diamino-3,6,9-triazundecane ("Tetraethylenepentamine"). The amine in carbon tetrachloride reacted vigorously 1 hour after mixing.[7,8]

N,N-Dimethylformamide. Iron may catalyze a very exothermic and sometimes violent reaction between carbon tetrachloride and dimethylformamide.[9]

Fluorine. Contact of gaseous fluorine with carbon tetrachloride results in a violent or explosive reaction.[5]

Metals. Heating Al powder with carbon tetrachloride to 152°C may cause an explosion.[10] The cleaning of barium lumps under carbon tetrachloride resulted in a violent reaction.[11] Mixtures of K or K-Na with carbon tetrachloride are extremely shock-sensitive.[12] A paste of Zn and carbon tetrachloride will burn readily after ignition.[13]

Potassium *tert*-Butoxide. Addition of drops of carbon tetrachloride to 1.5 g of potassium *tert*-butoxide resulted in ignition after 1 minute.[14]

Physiological Properties and Health Hazards

Vapor irritates the eyes and may cause headache, mental confusion, nausea, vomiting, and finally coma. Liquid irritates the eyes, and if swallowed, causes nausea, vomiting, and damage to the liver, kidneys, heart, and nervous system. Small doses may result in death. Prolonged exposure to the vapor may cause vomiting, bronchitis, and jaundice. Animal carcinogen; reasonably anticipated to be a human carcinogen.[15] Avoid breathing vapor. Avoid contact with skin and eyes.[16] TLV-TWA 5 ppm (31 mg/m^3); TLV-STEL 10 ppm (63 mg/m^3).[17]

Spillage Disposal

Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and PVA or Viton® gloves.[18] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into appropriate container and label for disposal by burning.[19–21]

Waste Disposal

Wear PVA or Viton® gloves,[18] laboratory coat, and eye protection. Avoid breathing vapor. Recycle by distillation (in a fume hood) or place waste in halogenated solvent–disposal container for disposal by burning. Dissolve the compound in a nonchlorinated flammable solvent and spray into a furnace with afterburner and scrubber.[20,21]

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CARMUSTINE $C_5H_9Cl_2N_3O_2$

ANTINEOPLASTIC

Synonyms

N,N-Bis(2-chloroethyl)-N-nitrosourea, BCNU, BiCNU, NSC 409962, Nitrumon.[1]

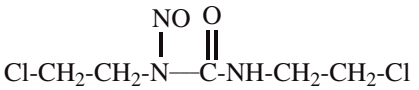
Physical Properties

Light yellow powder that melts to an oily liquid; mp, 30–32°C.[1]

Chemical Properties

Soluble in water (4 mg/mL) and 50% ethanol (150 mg/mL); soluble in ethanol (1 to 2); very soluble in lipids. Both powders and liquids are stable; in aqueous solutions, most stable at pH 4 (half-life, 511 minutes); in acid solutions and in solutions above pH 7, decomposes rapidly[2]; at neutral pH, half-life is 98 minutes.[3]

Structure



Physiological Properties and Health Hazards

LD_{50} orally, intraperitoneal, subcutaneous in mice: 19–25, 26, 24 mg/kg; orally in rats: 30–34 mg/kg.[1] Adverse effects in patients, including nausea and vomiting 4–6 hours after administration; burning pain along vein; facial flushing.[4] Animal carcinogen; reasonably anticipated to be human carcinogen.[5]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of carmustine in the spilled liquid and add 20 mL of detergent solution for each 100 mg of carmustine. The resulting mixture is heated at 55°C for 90 minutes. The solution is washed into the drain with water. The synthetic detergent solution (100 mL) is prepared by dissolving 4.8 g of sodium pyrophosphate in 100 mL of water and adding 1 mL of Triton X100.[6]

Waste Disposal

Wear goggles and protective gloves and clothing. To carmustine (100 mg) contained in a 125-mL Erlenmeyer flask equipped with a magnetic stirrer, slowly add 20 mL of synthetic detergent. Heat the mixture at 55°C for 90 minutes. The cooled solution can be washed into the drain with water.[6]

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CHLORAMBUCIL $C_{14}H_{19}Cl_2NO_2$

ANTINEOPLASTIC, HUMAN CARCINOGEN

Synonyms

4-[Bis(2-chloroethyl)amino]benzenebutanoic acid, Chloroaminophen, CB 1348, Amboclorin, Leukeran.

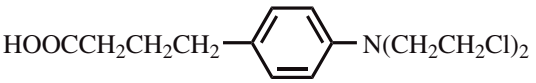
Physical Properties

Flattened needles from petroleum ether; mp, 64–66°C[1]; slight odor.[2]

Chemical Properties

Insoluble in water, but readily soluble in acid or alkali; soluble in benzene and ether. Hydrolysis in aqueous and alkaline solutions leads to replacement of Cl atoms by OH forming the hydroxy form.[2]

Structure



Mode of Action

This drug is an alkylating agent that reacts with proteins and DNA by replacement of one or both chlorine atoms.[3]

Physiological Properties and Health Hazards

Carcinogen in mice and rats.[2] In humans, a single dose of 20 mg produces nausea and vomiting while 6.5 mg/kg may cause irreversible bone marrow depression.[3,4] LD_{50} (i.p., rat), 58.2 μmol/kg.[1] Reasonably anticipated to be a human carcinogen.[3]

Waste Disposal

Wear protective clothing, gloves, and goggles. Work in the fume hood. To a solution of chlorambucil (25 commercial tablets, 50 mg) prepared by stirring at room temperature with 10 mL of water for 30 minutes, add 2 mL of concentrated sulfuric acid and 0.9 g of potassium permanganate. After stirring for 30 minutes, add a second portion of 0.9 g of potassium permanganate. Allow the mixture to stir at room temperature for another 4 hours. After careful neutralization with 10% sodium hydroxide solution, add a saturated solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water) while stirring until a colorless solution is formed. Wash into the drain with a large volume of water.[5]

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CHLORINE Cl_2

TOXIC BY INHALATION, SEVERE IRRITANT

Physical Properties

Greenish-yellow gas with irritating odor; bp, –34.05°C.[1]

Chemical Properties

Soluble in water.[1]

Hazardous Reactions

Acetylene. Mixtures of acetylene and chlorine may explode on exposure to sunlight, other UV source, or high temperature.[2]

Antimony Trichloride and Tetramethylsilane. Chlorination of tetramethylsilane to chlorotrimethylsilane in the presence of antimony trichloride is explosive at 100°C but controllable below 30°C.[3]

Bromine Pentafluoride. Contact results in violent reaction.[4]

tert-Butyl Alcohol. During the preparation of *tert*-butyl hypochlorite by chlorination of *tert*-butyl alcohol, the temperature must be kept below 20°C to prevent explosion.[5] Hypochlorite solution may be used instead of chlorine.[6]

Carbon Disulfide. The presence of traces of iron will catalyze the chlorination of carbon disulfide to carbon tetrachloride, resulting in an explosion.[7]

1-Chloro-2-propyne. Addition of chlorine to 1-chloro-2-propyne at 0°C over 4 hours resulted in an explosion, possibly due to the presence of excess chlorine.[7]

Cobalt(II) Chloride and Methanol. Passage of chlorine into a solution of cobalt chloride, bipyridyl, and lithium chloride in methanol resulted in an explosion and ignition of the methanol, probably due to formation of methyl hypochlorite.[8,9]

Dibutyl Phthalate. Mixture of the ester and chlorine in a bomb exploded at 118°C.[10]

Dichloro(methyl)arsine. Sealed ampoules of a mixture of the arsine with chlorine exploded below 0°C.[11]

Diethyl Ether. Ether may ignite on contact with chlorine.[10]

Diethylzinc. Ignites on contact with chlorine.[10]

Disilyl Oxide. Explodes on contact with chlorine.[10]

Fluorine. Mixture of fluorine and chlorine explodes when ignited by a spark.[12]

Glycerol. Mixture of glycerol and liquid chlorine in a bomb exploded at 70–80°C.[13]

Hydrocarbons. Methane and chlorine explode in the presence of mercury oxide. Mixtures of chlorine and ethylene explode in sunlight or in the presence of mercury or silver oxides.[14] Explosions occur between liquid chlorine and gasoline or a naphtha–sodium hydroxide mixture.[15]

Hydrocarbons and Lewis Acids. In the chlorination of hydrocarbons in the presence of Lewis acid catalysts, large volumes of hydrochloric acid are released if the catalyst is added to the chlorine hydrocarbon mixture. The catalyst should always be mixed with the hydrocarbon before addition of chlorine.[16]

Hydrogen. Mixtures of hydrogen and chlorine over the 5–89% range of hydrogen are explosive and can be initiated by sparks, radiant energy, or catalysts.[17,18]

Iron(III) Chloride and Monomers. Chlorination of styrene in carbon tetrachloride at 50°C resulted in a violent reaction that was catalyzed by ferric chloride.[19]

Metal Acetylides and Carbides. The mono- and di-alkali metal acetylides, copper acetylides, iron, uranium, and zirconium carbides ignite in chlorine.[19]

Metal Hydrides. Potassium, sodium, and copper hydrides ignite in chlorine.[20]

Metals. Aluminum, calcium powder, copper foil, iron wire, manganese powder, and potassium ignite in dry chlorine gas as do powdered antimony, bismuth, and germanium. Magnesium, sodium, and zinc ignite in the moist gas. Thorium, tin, and uranium ignite on warming.[14,21,22]

Methanol. Traces of metallic impurities in methanol can lead to an explosion and ignition of the methanol on introduction of chlorine.[23]

Nitrogen Compounds. Mixtures of ammonia and chlorine are explosive; hydrazine and hydroxylamine ignite in chlorine.[21–24] Formation of the dangerously explosive nitrogen trichloride is possible during chlorination of alkylthiuronium salts.[25] Aziridine readily forms the explosive N-chloro derivative.[26] Chlorination of sulfamic acid or acidic ammonium chloride solutions can give the powerfully explosive oil, nitrogen trichloride.[27]

Nonmetals. Boron, activated carbon, silicon, and phosphorus ignite in gaseous chlorine.[14,21,28]

Phenylmagnesium Bromide. The solid that formed on treatment of the Grignard reagent with chlorine exploded when shaken.[29]

Phosphorus Compounds. Phosphine and phosphorus trioxide ignite on contact with chlorine; ethylphosphine explodes.[30]

Silicon Oils. A mixture of liquid chlorine and silicone oils in a bomb exploded on heating.[31]

Sodium Hydroxide. Addition of liquid chlorine to 20% sodium hydroxide solution resulted in a violent reaction.[31]

Steel. Dry steel wool ignites in chlorine at 50°C.[31]

Sulfides. Diarsenic disulfide, diboron trisulfide, and mercuric sulfide ignite in chlorine.[32]

Synthetic Rubber. Natural and synthetic rubbers burn in liquid chlorine.[33]

Water. Some mixtures of chlorine and water can be ignited by a spark.[31]

Physiological Properties and Health Hazards

Gas irritates eyes and lungs, causing severe damage. Avoid breathing gas.[34] TLV-TWA 0.5 ppm (1.5 mg/m^3); TLV-STEL 1 ppm (2.9 mg/m^3).[35]

Spillage Disposal

Wear butyl[36] rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus. If the valve is leaking because it cannot be closed (a common occurrence), the gas can be bubbled through a reducer (sodium sulfite) and excess sodium carbonate solution. Be sure to include a trap in the line to prevent the solution from being sucked back into the cylinder. If this cannot be done, the cylinder should be placed in, or adjacent to, a fume hood and left to bleed off.

If the leak is in the valve assembly, a plastic bag can be fastened over the head of the cylinder, which can then be taken outdoors or to a fume hood.[37]

Waste Disposal

Package Lots. Seal cylinder and return to supplier.

Small Quantities. Wear butyl[36] rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus. Bubble the gas through a large volume of concentrated solution of reducer (sodium sulfite, sodium bisulfite). When reduction is complete, neutralize and wash into drain with at least 50 times its volume of water.[37]

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CHLOROFORM $CHCl_3$

ANIMAL CARCINOGEN, HARMFUL VAPOR

Synonyms

Trichloromethane.

Physical Properties

Colorless, nonflammable, volatile liquid with a characteristic odor; bp, 61–62°C.[1]

Chemical Properties

Immiscible with water; miscible with most other common organic solvents.[1]

Hazardous Reactions

Acetone and Alkali. In the presence of potassium hydroxide or calcium hydroxide, chloroform and acetone react vigorously to form 1,1,1-trichloro-2-hydroxy-2-methylpropane.[2–4]

Bis(dimethylamino)dimethylstannane. Heating a 1:1 chloroform solution results in a mild explosion.[5]

Fluorine. Contact of gaseous fluorine with chloroform results in a violent or explosive reaction.[6]

Metals. Mixtures of K, Na, or K-Na alloys, in chloroform, form impact-sensitive explosives.[7]

Nitromethane. Forms a detonatable mixture with chloroform.[8]

Potassium *tert*-Butoxide. Contact of potassium *tert*-butoxide with chloroform vapor or liquid results in ignition.[9]

Sodium and Methanol. Addition of sodium to an inadequately cooled mixture of chloroform and methanol results in an explosion.[10]

Sodium Hydroxide and Methanol. A mixture of chloroform and methanol reacts vigorously in the presence of small quantities of sodium hydroxide. A closed container of the mixture will explode.[10]

Sodium Methoxide. A mixture of solid sodium methoxide, methanol and chloroform may explode if the sodium methoxide is added in one portion.[10,11]

Triisopropylphosphine. Reacts vigorously with chloroform.[12]

Physiological Properties and Health Hazards

Vapor irritates the eyes and causes drowsiness, headache, nausea, and unconsciousness. Liquid irritates eyes and causes poisoning if swallowed. Avoid breathing vapor. Avoid contact with eyes.[13] Animal carcinogen; reasonably anticipated to be a human carcinogen.[14] TLV-TWA 10 ppm (49 mg/m^3).[15]

Spillage Disposal

Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and PVA or Viton® gloves.[16] Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriate container and label for disposal by burning in a furnace equipped with afterburner and scrubber.[17,18]

Waste Disposal

Wear PVA or Viton® gloves,[16] laboratory coat, and eye protection. Avoid breathing vapor. Recycle by distillation (in a fume hood) or place waste in halogenated solvent disposal container for disposal

by burning. Dissolve the compound in a nonchlorinated flammable solvent and spray into a furnace with afterburner and scrubber.[18,19]

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CHLOROMETHYL METHYL ETHER $ClCH_2OCH_3$

HUMAN CARCINOGEN

Synonyms

Chloromethoxymethane, CMME, methyl chloromethyl ether.

Physical Properties

Colorless liquid; bp, 59°C.[1]

Fire Hazard

Flash point, –8°C.[2]

Chemical Properties

Decomposes in water to form hydrochloric acid, formaldehyde, and methanol.[3]

Hazardous Reactions

Peroxide Formation. Long-standing ether in contact with air and exposed to light may contain peroxides, especially if stored in clear glass. Explosions have occurred when caps or stoppers were turned.[4]

Physiological Properties and Health Hazards

Avoid inhalation of vapor and contact with eyes or skin. Primary effects probably are due to fairly rapid formation of hydrochloric acid, formaldehyde, and methanol. A high incidence of lung cancer has been observed after chronic exposure.[3] Animal and human carcinogen.[5] Exposure by all routes should be carefully controlled to lowest possible levels.[6]

Spillage Disposal

Wear butyl rubber gloves, eye protection, laboratory coat, large heavy face shield (if in doubt use body shield also), and self-contained breathing apparatus. Eliminate all sources of ignition and flammables. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic shovel, scoop into a plastic pail. In the fume hood, slowly add the solid mixture to a large quantity of water and allow to stand until reaction is complete. Decant solution to drain. The solid residue may be treated as normal refuse.[7]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

Containers of chloromethyl methyl ether that have been opened and stored may contain peroxides. Especially if they have screw caps, these should be handled with great care. Where the container can be opened safely, check for peroxides as follows: in the fume hood, add 1 mL of the ether to a solution of 100 mg of potassium iodide in 1 mL of glacial acetic acid. A pale yellow color indicates a low concentration, and a bright yellow or brown color, a high concentration of peroxides in the sample.[8] If peroxides are present, these may be removed by passing the ether through a column of basic alumina.

Package the ether for disposal by burning.

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m-CHLOROPERBENZOIC ACID $m\text{-}ClC_6H_4CO_3H$

IRRITANT

Physical Properties

Solid; mp, 92–94°C (dec).[1]

Hazardous Reactions

The peracid, when hot and in concentrated solutions may decompose explosively.[2]

2-Bromo-4-Methylpyridine. Solid residue from oxidation of the substituted pyridine with 5% excess of the peracid decomposed violently.[3]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic shovel, scoop into a container. In the fume hood, add the solid mixture to 50% ice-cold sodium metabisulfite solution. Stir mixture for 1 hour. Pour solution into the drain. Treat solid residue as normal refuse.[4,5]

Waste Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Body shield should be available. In the fume hood behind a shield, cautiously add the *m*-chloroperbenzoic acid in portions to a large volume of ice-cold 50% aqueous sodium metabisulfite solution. Use starch iodide paper to test for complete reduction of any peracid. Neutralize and wash into the drain.[5]

Reactions for Spillage and Waste Disposal

$$m\text{-}ClC_6H_4CO_3H + Na_2S_2O_5 \rightarrow m\text{-}ClC_6H_4CO_2H + Na_2SO_4 + SO_2$$

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N-CHLOROSUCCINIMIDE $(\text{-}CH_2CO)_2NCl$

MAY REACT VIOLENTLY, IRRITANT

Physical Properties

Orthorhombic crystals; mp, 150–151°C. Odor of chlorine.[1]

Chemical Properties

Acid to litmus. One gram dissolves in about 70 mL water, 150 mL alcohol, 50 mL benzene. Sparingly soluble in ether, chloroform, and carbon tetrachloride. Liberates iodine from potassium iodide solutions and bromine from sodium bromide solutions.[1]

Hazardous Reactions

Alcohols. Extremely violent reaction with aliphatic alcohols.[2]

Benzylamine. Mixture reacts violently.[2]

Dust. Dust contamination of the stored imide may cause smoldering.[3]

Hydrazine Hydrate. Mixture reacts violently.[2]

Physiological Properties and Health Hazards

Irritant.[4] Avoid contact with skin and breathing dust.[5]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Scoop up and treat as for waste disposal.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a combustible solvent and spray into a furnace equipped with afterburner and scrubber.[4]

Small Quantities. Wear nitrile rubber gloves, eye protection, and laboratory coat. Work in the fume hood. Prepare a solution of sodium sulfite in water (use 1.2 g of sodium sulfite in 10 mL of water for each 1 g of N-chlorosuccinimide). Slowly, while stirring, add the N-chlorosuccinimide to the sodium sulfite solution. When the reaction is complete, wash the solution into the drain.[6]

Reactions for Spillage and Waste Disposal

$$(\text{-}CH_2CO)_2NCl + Na_2SO_3 + H_2O \rightarrow \underset{\text{succinimide}}{(\text{-}CH_2CO)_2NH} + Na_2SO_4 + HCl$$

REFERENCES

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CHLOROSULFONIC ACID $ClSO_3H$

VIOLENT REACTION WITH WATER, CAUSES SEVERE BURNS

Synonyms

Chlorosulfuric acid, sulfuric chlorohydrin.

Physical Properties

Colorless or slightly yellow liquid; fumes in air; pungent odor; mp, –80°C; bp, 151–152°C.[1]

Chemical Properties

Reacts violently with water. Soluble in pyridine, liquid sulfur dioxide, and dichloroethane. Chlorosulfonating and condensing agent in organic syntheses.[1]

Hazardous Reactions

Vigorous to violent reactions with organic matter.[2]

Diphenyl Ether. Vigorous interaction above 40°C.[3]

Hydrogen Peroxide. An explosion occurred 10 minutes after a sample of permonosulfonic acid, which was prepared by reacting chlorosulfonic acid and 90% hydrogen peroxide and then stored overnight at 0°C, was removed to a test-tube rack.[4]

Phosphorus. Explosive reaction with yellow phosphorus above 25°C.[5]

Silver Nitrate. Interaction with chlorosulfonic acid is violent, with nitrosulfuric acid being formed.[6]

Water. Highly exothermic and violent reaction with water.[1,3]

Physiological Properties and Health Hazards

Highly irritating and corrosive to eyes, skin, and mucous membranes.[1] Causes severe burns and is irritating to respiratory system. If taken by mouth, causes severe local and internal irritation and damage.[1,7]

Spillage Disposal

Wear eye protection, nitrile rubber gloves, and laboratory coat (and respirator if required), and work from behind heavy body shield. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop up solid mixture; in the fume hood, add slowly, while stirring, to a pail of cold water. If necessary, neutralize with sodium carbonate. Decant the liquid to the drain with 50 times its volume of water. Discard the solid as normal refuse. Wash spill area thoroughly.[7,8]

Waste Disposal

Work in the fume hood wearing eye protection, laboratory coat, and nitrile rubber gloves. Slowly and carefully pour the chlorosulfonic acid onto sodium carbonate contained in a large plastic tray. When reaction is over, wash into drain.[7,8]

Reactions for Spillage and Waste Disposal

$$ClSO_3H + Na_2CO_3 \rightarrow NaCl + \underset{\text{sodium bisulfate}}{NaHSO_4} + CO_2$$

REFERENCES

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6. Mellor, Vol. 3, 470, 1941.
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CHLOROTRIMETHYLSILANE $(CH_3)_3SiCl$

HIGHLY FLAMMABLE, CAUSES BURNS

Synonyms

Trimethylchlorosilane.

Physical Properties

Colorless, volatile, fuming liquid with pungent odor; bp, 57°C.[1]

Fire Hazard

Highly flammable liquid. Flash point, –20°C.[2]

Chemical Properties

Reacts violently with water.[1]

Hazardous Reactions

Preparation by antimony trichloride catalyzed chlorination of tetramethylsilane may result in explosion if allowed to heat to 100°C.[2]

Physiological Properties and Health Hazards

Vapor irritating to eyes, skin, and respiratory system. Liquid causes burns and severe internal damage if ingested.[1]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and a laboratory coat. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid mixture into a container and transport to the fume hood. Slowly, while stirring, add to a pail of cold water. If necessary, neutralize with sodium carbonate. Decant the liquid into the drain with 50 times its volume of water and treat the residue as normal refuse.[3,4]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

Place 500 mL of water into a 1-L, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Place 10 mL of chlorotrimethylsilane in the funnel and add dropwise to the stirred water. The water temperature should not be allowed to rise above 35°C. Cool in an ice bath if necessary. When addition is complete, neutralize the aqueous solution with sodium carbonate and wash into the drain with at least 50 times its volume of water.[3,4]

Reactions for Spillage and Waste Disposal

$$(CH_3)_2SiCl + H_2O \rightarrow \underset{\text{trimethylsilanol}}{(CH_3)_3SiOH} + HCl$$

REFERENCES

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2. B 996.

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CHLOROZOTOCIN $C_9H_{16}ClN_3O_7$

ANTINEOPLASTIC

Synonyms

2-({[2-Chloroethyl)nitrosamino]carbonyl}amino)-2-deoxy-D-glucose, 2-[3-(2-chloroethyl)-3-nitrosoureido]-2-deoxy-D-glucopyranose.[1]

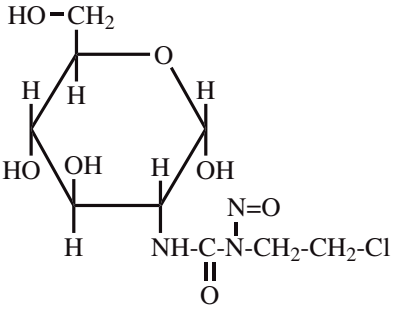
Physical Properties

Ivory-colored crystals; light yellow crystals[2]; mp, 147–148°C (decomposition with evolution of gas).[1]

Chemical Properties

Soluble in water.[1]

Structure



Physiological Properties and Health Hazards

Adverse effects in patients include nausea or vomiting, bone marrow depression, and pancreatic islet-cell toxicity.[5]

Spillage Disposal

(Not appropriate for the treatment of aqueous spills.) Wear protective gloves, laboratory coat, and goggles. Isolate spill area. For solutions in volatile organic solvents, allow solvent to evaporate. Cover area with an excess of 4.5% solution of hydrobromic acid. Allow to react for at least 1 hour. Absorb residual solution with 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and, in the fume hood, slowly add it to a beaker of cold water. If necessary, neutralize the solution with soda ash. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse.[3,4]

Waste Disposal

Small quantities. Wear goggles and protective gloves and clothing. Work in the fume hood. To prepare the 4.5% hydrobromic acid in glacial acetic acid, add 85 mL of glacial acetic acid to 15 mL of commercial 30% hydrobromic acid in glacial acetic acid. Add required volume (10 mL) of the acid mixture to the solid chlorozotocin (100 mg). Allow the mixture to stand at room temperature for at least 15 minutes. When reaction is complete and the mixture has been flushed with nitrogen

for 30 minutes, carefully add 10% sodium hydroxide until a neutral solution is formed. Wash the liquid into the drain with a large volume of water.[3,4] Immerse glassware, including vials, containing solid chlorozotocin residue in glacial acetic acid, 4.5% in hydrobromic acid, and allow to stand overnight. Thoroughly rinse glassware with water and reuse or discard. After careful neutralization with 10% sodium hydroxide solution, wash hydrobromic acid/glacial acetic acid solution into the drain with water.[4]

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3. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 103.
4. Armour, M.A., *Laboratory Disposal of Chlorozotocin,* University of Alberta, Edmonton, 2000.
5. Becker, T.M., *Cancer Chemotherapy: A Manual for Nurses*, 2nd ed., Little, Brown, Boston; New York, 1982, p. 243.

CHLORPYRIFOS $C_9H_{11}Cl_3NO_3PS$

INSECTICIDE

Synonyms

O,O-diethyl-O(3,5,6-trichloro-2-pyridyl)phosphorothioate, Dursban, Lorsban, Pyrinex.

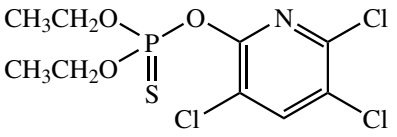
Physical Properties

White crystals with mild mercaptan odor; mp, 41.5–43°C.[1]

Chemical Properties

Solubility in water, 2 ppm at 25°C; soluble in organic solvents.[2]

Structure



Physiological Properties and Health Hazards

LD_{50} (oral rat) 163 mg/kg. LD_{50} (dermal rabbit) 2000 mg/kg. Toxic to shrimp and at high levels to fish.[3,4] LC_{50} (rainbow trout, 96) 3 μg/L.[1] TLV-TWA 0.2 mg/m^3.[5]

Formulations

Emulsifiable concentrate, wettable powder, granules.

Waste Disposal

Wear gloves and eye protection. To decompose chlorpyrifos, add 100 mL of 6% sodium hypochlorite solution (household bleach) to 300 mg of pure insecticide or 1 mL of concentrated commercial solution, and allow the mixture to stand at room temperature for at least 18 hours. The solution can be poured into the drain with water.[6]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer before washing other clothes by running the machine through a complete wash cycle with detergent (but without clothing).

The best laundry procedure for 100% cotton and 65/35 polyester/cotton fabric is to presoak the fabric for 3 hours in the washing machine filled with warm water (70 liters at 50°C) to which 280 mL of chlorine bleach has been added. After the 3 hours, empty the machine, and give the fabric a single warm wash (at 50°C). Less than 0.1% chlorpyrifos residue remains on both cotton and polyester/cotton when the bleach soak pre-treatment is used; however, when a single wash, without the bleach pre-soak is used, approximately 40% of the chlorpyrifos residue remains on the cotton fabric and 28% on the polyester/cotton blend.

NOTE: The bleach/soak procedure results in color loss that is severe for 100% cotton and less for the polyester/cotton blend; in addition, after 25 bleach/soak launderings, a third to half of the original strength of the 100% cotton is lost, whereas there is no significant strength loss for the 65/35 polyester/cotton blend.[7]

Do not wash clothing that has been contaminated with concentrated chlorpyrifos; approximately 38% chlorpyrifos remains in the fabric after the bleach/soak procedure is used. Workwear contaminated with the concentrate should be placed in a plastic bag and taken to an approved landfill site.[8]

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CHROMIC ACID CLEANING SOLUTION H_2CrO_4

HIGHLY CORROSIVE, TOXIC

Physical Properties

Solution of potassium or sodium dichromate in concentrated sulfuric acid (50 g/L).

Hazardous Reactions

See Potassium Dichromate, Sodium Dichromate, and Sulfuric Acid.

Physiological Properties and Health Hazards

See Potassium Dichromate, Sodium Dichromate, and Sulfuric Acid. Very corrosive; toxic.

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a container of water. In the fume hood, carefully acidify this solution with 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water) until a pH of 1 (pHydrion paper) is obtained. Slowly and while stirring, add solid sodium thiosulfate until the solution becomes cloudy and blue colored. Neutralize the solution by adding sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Let the mixture stand for a week or filter immediately through Celite. After standing, much of the supernatant can be decanted. Allow the remaining liquid to evaporate or filter the solid. The liquid in both methods can be washed into the drain. The solid residue should be washed with hot water, dried, packaged, labeled, and sent to a secure landfill site. The spillage site and contaminated clothing should be washed thoroughly with soap and water to remove the oxidant.[1,2]

Waste Disposal

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, slowly and carefully add the cleaning solution (100 mL) to a container of water (about 1:1 dilution). Adjust pH to 1 by the addition of 3 M sulfuric acid or sodium carbonate. While stirring, slowly add solid sodium thiosulfate (about 13.5 g) until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes a blue-gray flocculent precipitate is formed. Let the mixture stand for a week or filter immediately through Celite. After a week, much of the supernatant can be decanted. Allow the remaining liquid to evaporate or filter the solid. The liquid can be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to a secure landfill site.[1]

Reactions for Spillage and Waste Disposal

$$Cr_2O_7^{2-} + 3S_2O_3^{2-} + 2H_3O^+ \rightarrow \underset{\substack{\text{chromium}\\ \text{hydroxide}\\ \text{(insoluble)}}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

REFERENCES

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2. Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 27, 1999; *Haz. Mat. Spills Tec.*, 29.7; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A., et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 287.

CHROMIUM Cr

TOXIC AS DUST, IRRITANT

Physical Properties

Steel-gray lustrous metal; mp, 1900°C.[1]

Chemical Properties

Soluble in dilute hydrochloric acid or sulfuric acid; practically insoluble in nitric acid. Attacked by caustic alkalis and alkali carbonates. Not oxidized by air, even in presence of much moisture.[1]

Hazardous Reactions

Moderate fire hazard in form of dust. Reacts violently with ammonium nitrate, hydrogen peroxide, lithium, nitrous oxide, potassium chlorate, and sulfur dioxide.[2]

Physiological Properties and Health Hazards

Irritant.[3] High pulmonary toxicity. Certain hexavalent chromium compounds have been listed as known human carcinogens.[4] TLV-TWA 0.5 mg/m^3.[5]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. Avoid breathing dust. If in finely divided form, cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriately labeled container for disposal in a poisonous or hazardous waste landfill site.[6,7]

Waste Disposal

Place in separate container labeled for reuse or recycling[3] or package for disposal in accordance with local regulations.[7]

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CHROMIUM DIACETATE $Cr(C_2H_3O_2)_2$

PYROPHORIC IN AIR WHEN ANHYDROUS

Synonyms

Chromous acetate.

Physical Properties

Monohydrate, deep red powder; anhydrous salt: brown.[1]

Chemical Properties

Slightly soluble in cold water; readily soluble in hot. Slightly soluble in alcohol; practically insoluble in ether. Reacts with most acids. Easily oxidized, especially when moist, to chromic acetate.[1]

Hazardous Reactions

Anhydrous salt is pyrophoric in air; in lower oxygen concentrations, it chars slowly.[2]

Physiological Properties and Health Hazards

TLV-TWA 0.5 mg/m^3.[3]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, and laboratory coat. To reduce risk of forming airborne dust, cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Mix and scoop into a container. In the fume hood, add to a pail of water. If effervescent, wait until reaction is complete. Cautiously add calcium or sodium hypochlorite solution (reaction may be vigorous). Add more water, stir and allow to stand for 2 hours. Add 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water) until solution is definitely acid (pH 1 to pHydrion paper). Add sodium thiosulfate crystals until solution becomes blue colored and cloudy. Neutralize with sodium carbonate. A blue-gray flocculent precipitate should form. The mixture can be filtered immediately through Celite or allowed to stand 1 week, when much of the supernatant liquid can be decanted. In either case, the liquid can be washed into the drain with 50 times its volume of water; the solid should be washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to a secure landfill site.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. In a large container, mix with equal volume of sodium carbonate and add water to form a slurry. Cautiously add calcium or sodium hypochlorite. Add more water if necessary and let stand two hours. Add 3 M sulfuric acid until solution is definitely acid (pH 1 to pHydrion paper). Add sodium thiosulfate crystals until solution becomes blue colored and cloudy. Neutralize with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. The mixture can be filtered immediately through Celite or allowed to stand for 1 week, when much of the supernatant liquid can be decanted. In the latter case, the remaining liquid is allowed to evaporate, or the solid filtered through Celite. The supernatant (if colorless) can be washed into the drain with 50 times its volume of water. The solid residue should be washed with hot water to remove sodium sulfate, packaged, labeled, and sent to a secure landfill site.[4]

Because it is difficult to stop the oxidation at the Cr(III) state, a reduction step is necessary.[4]

Reactions for Spillage and Waste Disposal

$$Cr(C_2H_3O_2)_2 + 2NaOCl + H_2O \rightarrow CrO_3 + 2CH_3COOH + 2NaCl$$

$$2CrO_3 + 3S_2O_3^{2-} + 3H_2O \rightarrow \underset{\substack{\text{chromium}\\\text{hydroxide}\\\text{(insoluble)}}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

REFERENCES

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CHROMIUM HEXACARBONYL $Cr(CO)_6$

CANCER SUSPECT AGENT, SEVERE POISON

Synonyms

Chromium carbonyl.

Physical Properties

Orthorhombic, highly refractive crystals; sublimes at room temperature; decomposes at 130°C; explodes at 210°C.[1]

Chemical Properties

Burns with luminous flame; almost insoluble in water, ethanol, methanol; soluble in ether, chloroform, and other organic solvents; solutions and impure solid decompose in light.[1]

Hazardous Reactions

May explode when heated.[2]

Physiological Properties and Health Hazards

Severe poison. May be fatal if inhaled, swallowed, or absorbed through skin. May cause irritation.[2] LD_{50} (intravenous, mouse) 100 mg/kg.[1] TLV-TWA 0.01 mg (Cr)/m^3.[3] Suspected to be animal and human carcinogen.[4]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and protection. To reduce risk of forming airborne dust, cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container and transport to the fume hood. Slowly, while stirring, add the solid to household bleach (50 mL/g of chromium hexacarbonyl). Allow the solid to settle, decant the liquid to the drain with at least 50 times its volume of water. Dry and package the solid for disposal in accordance with local regulations.[5,6]

Waste Disposal

Package Lots. Package securely, label, and send for burial in a site approved for disposal of poisonous wastes.[2]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve the hexacarbonyl in tetrahydrofuran to give a 5% solution. For each 1 g of hexacarbonyl to be destroyed, place 50 mL of household bleach in a three-necked, round-bottom flask equipped with a nitrogen inlet, dropping funnel, and stirrer. Slowly add the hexacarbonyl solution dropwise. After addition is complete, stir for an hour. Collect the solid by filtration and package for disposal in a secure landfill; the liquid is washed into the drain.[5,6]

Reactions for Spillage and Waste Disposal

$$2Cr(CO)_6 + 3NaOCl + 3H_2O \rightarrow \underset{\substack{\text{chromium}\\\text{hydroxide}\\\text{(insoluble)}}}{2Cr(OH)_3} + 12CO + 3NaCl$$

REFERENCES

1. Merck 2253.
2. Ald 881B.
3. ACGIH 23.
4. NIEHS III–22.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 101; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 289.
6. PP 95.

CHROMIUM TRIOXIDE CrO_3

CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE, CAUSES SEVERE BURNS

Synonyms

Chromic acid.

Physical Properties

Dark red crystals or flakes, or granular powder; mp, 197°C; dec, 250°C to chromic oxide and oxygen.[1]

Fire Hazard

Powerful oxidizing agent. Extinguish with water.[2]

Chemical Properties

Soluble in water and sulfuric acid.[1]

Hazardous Reactions

Since the presence of nitrates in chromium trioxide may cause oxidation reactions to accelerate out of control, the oxide should be tested before use. If oxides of nitrogen are evolved on melting a sample of the oxide, it should be discarded.[3]

Acetic Acid. Mixture with glacial acetic acid may explode on heating.[4]

Acetic Anhydride. Mixture may explode if not adequately cooled and stirred.[5–8]

Acetic Anhydride and Tetrahydronaphthalene. Ignition may result on mixing a solution of the trioxide in acetic anhydride with tetrahydronaphthalene in the preparation of tetralone.[9]

Acetone. Ignition occurs on contact at room temperature; thus, the trioxide should not be used to purify acetone.[10]

Acetylene. Violent oxidation occurs.[11]

Alcohols. Ignites on grinding in methanol, ethanol, 2-propanol, butanol, and cyclohexanol.[12] Finely divided trioxide must be used in the oxidation of secondary alcohols in N,N-dimethylformamide to avoid violent local reaction.[13]

Alkali Metals. Incandesces with sodium or potassium.[14]

Ammonia. Incandesces with gaseous ammonia and reacts very exothermally with the liquid.[15]

Arsenic. Mixture incandesces.[16]

Butyric Acid. Mixture incandesces above 100°C.[17]

N,N-Dimethylformamide. Addition of lumps of the trioxide causes ignition even on ice cooling under nitrogen.[18]

Glycerol. Violent reaction of mixture, which may ignite.[18]

Hexamethylphosphoric Triamide. Violent decomposition occurs when crushed trioxide is added to the amide.[19]

Organic Materials and Solvents. Combustible materials may ignite or explode on contact with the trioxide.[20]

Performic Acid. Violent reaction occurs.[21]

Pyridine. The pyridine-trioxide complex is a powerful oxidant. During its preparation, effective stirring must be maintained during the addition of the oxide to pyridine at 10 to 20°C. Pyridine should never be added to the solid oxide.[22–26]

Selenium. Violent reaction occurs.[15]

Sulfur. Mixture ignites on warming.[27]

Physiological Properties and Health Hazards

The dust irritates all parts of the respiratory system.[28] Skin contact can cause primary irritation and ulceration as well as allergic eczema. The solid and its solutions cause severe eye burns. Swallowing would result in severe internal irritation and damage. Pulmonary irritation and bronchogenic carcinoma may result from breathing chromate dust.[1] Avoid inhaling dust. Prevent contact with eyes and skin.[28] TLV-TWA 0.5 mg (Cr)/m^3.[29]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves (and rubber boots or overshoes if spillage is large). To reduce risk of forming airborne dust, cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a beaker of water. In the fume hood, carefully acidify this solution with 3 M sulfuric acid (prepared by cautiously adding 15 mL of concentrated acid to 75 mL of cold water) to pH 1. Slowly add solid sodium thiosulfate until the solution becomes blue colored and cloudy. Neutralize with sodium carbonate; a blue-gray flocculent precipitate is formed after a few minutes. Filter immediately through Celite or let stand for 1 week. Much of the liquid may then be decanted. The remaining liquid can be filtered off or allowed to evaporate. In either case, wash the liquid into the drain. The solid residue is dried, packaged, labeled, and sent to a secure landfill site.[30]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal in hazardous waste landfill.[31]

Small Quantities. Wear nitrile rubber gloves, eye protection, and a laboratory coat. In the fume hood, add the solid chromium trioxide (5 g) to a beaker of water (about 100 mL). Acidify this solution to pH 1 with 3 M sulfuric acid (about 55 mL). While stirring, slowly add solid sodium thiosulfate (about 20 g) until the solution becomes cloudy and blue colored. Neutralize with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Filter immediately through Celite or allow to stand for 1 week, when much of the supernatant can be decanted. In either case, the liquid can be washed into the drain while the solid residue should be washed with hot water to remove sodium sulfate, dried, packaged, labeled, and sent to a secure landfill site.[30]

Reactions for Spillage and Waste Disposal

$$2CrO_3 + 3S_2O_3^{2-} + 3H_2O \rightarrow \underset{\text{chromium hydroxide (insoluble)}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

REFERENCES

1. Merck 2256.
2. NFPA 49.

3. Baker, W., *Chem. Ind.*, 280, 1965.
4. B 1068.
5. Dawber, J.G., *Chem. Ind.*, 973, 1964.
6. B 1068.
7. Baker, W., *Chem. Ind.*, 280, 1956.
8. Eck, C.R. et al., *J. Chem. Soc., Chem. Commun.*, 865, 1974.
9. Peak, D.A., *Chem. Ind.*, 14, 1949.
10. Delhez, R., *Chem. Ind.*, 931, 1956.
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12. Newth, F.H. et al., *Chem. Ind.*, 1482, 1964.
13. Neumann, H., *Chem. Eng. News*, 48, 4, 1970.
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15. Mellor, Vol. 11, 233, 1943.
16. Mellor, Vol. 11, 234, 1943.
17. Wilson, R.D., *Chem. Ind.*, 758, 1957.
18. B 1070.
19. Cardillo, G. et al., *Synthesis*, 6, 394, 1976.
20. B 1071.
21. D'Ans, J. et al., *Chemische Berichte*, 48, 1136, 1915.
22. Dauben, W.G. et al., *J. Org. Chem.*, 34, 3587, 1969.
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26. Stensio, K.E., *Acta Chem. Scand.*, 25, 1125, 1971.
27. Mellor, Vol. 11, 232, 1943.
28. Lux 304.
29. ACGIH 23.
30. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.
31. Ald 882A.

CISPLATIN $Cl_2H_6N_2Pt$

ANTINEOPLASTIC, ANIMAL CARCINOGEN

Synonyms

Platinol, Platinex, Neoplatin, CACP, CPDC, DDP, *cis*-DDP, Cisplatyl, NSC-119875, *cis*-platinum II.

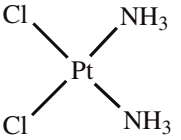
Physical Properties

Deep yellow solid; mp, 270°C (dec).[1]

Chemical Properties

Slightly soluble in water (0.253 g/100 g at 25°C). Insoluble in most common organic solvents except DMF.[1] When heated to decomposition, emits toxic fumes of chlorine and NO_x.[2]

Structure



Physiological Properties and Health Hazards

Cisplatin caused lung adenomas and skin papillomas in female mice and leukemia in rats. Animal carcinogen; reasonably anticipated to be a human carcinogen.[2]

Waste Disposal

Wear goggles and protective gloves and clothing. Dissolve a 50-mg sample in 200 mL of water. Add 4 g of diethyldithiocarbamate and stir for 24 hours. The yellow precipitate that forms is a deactivated complex which can safely be discarded. Remove the precipitate by filtration and wash the filtrate into the drain with a large volume of water.[3–5]

Prepare a solution containing 1 g of diethyldithiocarbamate in 100 mL of 0.1 M sodium hydroxide. For each 10 mg of cisplatin, add 3 mL of the diethyldithiocarbamate solution and 3 mL of a saturated solution of sodium nitrate. After mixing thoroughly, discard the solution into the drain with a large volume of water.[4,5]

Immerse glassware containing residues of cisplatin in a 1:1 diethyldithiocarbamate and saturated sodium nitrate solution. After standing overnight, rinse thoroughly with water and reuse or discard. Discard the solution into the drain with a large volume of water.[4,5]

REFERENCES

1. Merck 2341.
2. NIEHS III–93.
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4. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 97, 1985.
5. Armour, M.A., Chang, M., Crerar, J., Linetsky, A., and Renecker, D., On-site destruction of small quantities of chemical carcinogens, *Proceedings of 16th Annual Waste Management Conference*, Environment Canada, Calgary, Alberta, 1994, p. 378.

CYANIDES (water soluble) CN^-

POISONOUS, SERIOUS RISK OF POISONING BY SWALLOWING OR SKIN CONTACT, CONTACT WITH ACIDS LIBERATES A TOXIC GAS

See individual entries for Potassium Cyanide and Sodium Cyanide.

Physical Properties

Colorless crystals or powders.[1]

Chemical Properties

Soluble in water; react with acids to generate hydrogen cyanide.[1]

Physiological Properties and Health Hazards

Cyanide salts and solutions of cyanides present considerably less risk than does hydrogen cyanide gas because they are dangerous only by ingestion. Swallowing significant quantities is unlikely to occur except by deliberate intention; poisoning by inhalation of these preparations is not a hazard. However, they should be treated with due care. Solutions of over 1% strength should always be kept under strict control; those under 1% constitute, in practice, little danger but always demand careful handling.[1] The early warning symptoms of poisoning are general weakness and heaviness of the arms and legs, increased difficulty in breathing, headache, dizziness, nausea, vomiting, and these may be rapidly followed by pallor, unconsciousness, cessation of breathing, and death.[1,2] If ingestion of a cyanide solution does take place, amyl nitrite should be administered as for the gas and the casualty rapidly moved to hospital.[2] TLV-STEL-C 5 mg/m^3 (skin).[3]

Waste Disposal

Wear nitrile gloves, laboratory coat, and eye protection. Work in the fume hood. Dilute the solution of cyanide with water to a concentration not greater than 2%. For each 50 mL of solution, slowly add, while stirring, 5 mL of 10% sodium hydroxide solution, and 60–70 mL of household bleach. Test the solution for continued presence of cyanide as follows. Remove about 1 mL of the solution and place in a test tube. Add 2 drops of a freshly prepared 5% aqueous ferrous sulfate solution. Boil the mixture for 30 seconds, cool to room temperature, and add 2 drops of 1% ferric chloride solution. Acidify the mixture to litmus with 6 M hydrochloric acid (slowly add concentrated acid to an equal volume of cold water). If cyanide is still present, a deep blue precipitate forms. If the test is positive, more bleach is added to the cyanide solution and the test repeated. When the blue precipitate no longer forms, the solution can be washed down the drain.[4,5]

Reactions for Spillage and Waste Disposal

$$CN^- + ClO^- \rightarrow \underset{\text{cyanate}}{CNO^-} + Cl^-$$

REFERENCES

1. Lux 310.
2. Lux 19.
3. ACGIH 36.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
5. PPL 164.

CYANOGEN $(CN)_2$

EXTREMELY FLAMMABLE, TOXIC BY INHALATION, IRRITANT

Synonyms

Dicyanogen.

Physical Properties

Colorless gas with almond-like odor; bp, –21°C.[1]

Fire Hazard

Extremely flammable. Explosive limits, 6–32%. Because the gas is supplied in a cylinder, turning off the valve will reduce any fire involving it; if possible, cylinders should be removed quickly from an area in which a fire has developed. Fight fire with water spray.[2]

Chemical Properties

One volume of water dissolves about 4 volumes of cyanogen gas; soluble in alcohol and ether. Slowly hydrolyzed in water to give oxalic acid and ammonia.[1]

Hazardous Reactions

Fluorine. Decomposes and ignites in fluorine.[3]

Oxygen (liquid). Mixture of liquid oxygen and liquid cyanogen explodes.[4]

Physiological Properties and Health Hazards

The gas irritates the respiratory system, leading to headache, dizziness, rapid pulse, nausea, vomiting, unconsciousness, convulsions, and death, depending on exposure. The gas irritates the eyes, causing lachrymation. Prevent inhalation of gas.[5] TLV-TWA 10 ppm (21 mg/m^3).[6]

Waste Disposal

Seal cylinder and return to supplier. When the leak cannot be sealed, wear self-contained breathing apparatus and transport the cylinder to a fume hood. Attach a regulator to the cylinder and a piece of flexible rubber hose to the regulator. Dip the hose into a solution of sodium hydroxide and sodium hypochlorite. When the cylinder is empty, allow the basic solution to stand for 24 hours, and then wash into the drain with at least 50 times its volume of water.[7]

Reactions for Spillage and Waste Disposal

$$(CN)_2 + NaOCl + 2NaOH \rightarrow \underset{\text{sodium cyanate}}{2NaCNO} + NaCl + H_2O$$

REFERENCES

1. Merck 2721.
2. NFPA 49.
3. B 1099.
4. B 1409.

5. Lux 310.
6. ACGIH 24.
7. Braker, W. and Mossman, A.L., *Matheson Gas Data Book*, 6th ed., Matheson Gas Products, East Rutherford, NJ, 1980.

CYANOGEN BROMIDE CNBr

POISONOUS, EXPLOSIVE

Physical Properties

Needles, cubes; volatile at ordinary temperatures; mp, 52°C; bp, 61–62°C.[1]

Chemical Properties

Freely soluble in water, alcohol, and ether. Aqueous solutions of alkalies decompose it to alkali cyanide and alkali bromide. Pure cyanogen bromide, completely dried by distillation over sodium, may be stored in a desiccator for several months. Impure material decomposes rapidly and tends to explode.[1]

Physiological Properties and Health Hazards

Vapor is a severe irritant and very poisonous.[1]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, laboratory coat, and self-contained breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Add to a pail of cold water, and then, while stirring, add an alkaline solution of calcium hypochlorite or household bleach. After 24 hours, decant the solution into the drain with water. The solid residue can be treated as normal refuse.[2,3]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood and behind a shield, cautiously, while stirring, add to a large volume of an alkaline solution of calcium hypochlorite or household bleach. After 24 hours, wash into the drain.[3]

Reactions for Spillage and Waste Disposal

$$CNBr + 2NaOH \rightarrow NaCN + NaOBr + 2H_2O$$

$$NaCN + NaOCl \rightarrow \underset{\text{sodium cyanate}}{NaCNO} + NaCl$$

REFERENCES

1. Merck 2723.
2. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
3. ITI 178.

CYANOGEN CHLORIDE CNCl

IRRITANT, VERY POISONOUS

Physical Properties

Colorless gas or liquid; bp, 13.8°C; mp, –6°C.[1,2]

Chemical Properties

Soluble in water, alcohol, and ether.[1]

Physiological Properties and Health Hazards

Highly irritating via inhalation. Severe eye irritant.[3] Toxic effects similar to those of hydrogen cyanide.[1] TLV-STEL-C 0.3 ppm (0.75 mg/m^3).[4]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, laboratory coat, and self-contained breathing apparatus if necessary. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to a beaker of water in the fume hood. Slowly add to an excess of an alkaline solution of calcium hypochlorite or household bleach. After 24 hours, wash into drain with at least 50 times its volume of water. Discard solid residue with normal refuse.[2,5]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, slowly, and while stirring, add the cyanogen chloride to an excess of a strongly alkaline solution of calcium hypochlorite or household bleach. After 24 hours, wash into drain with at least 50 times its volume of water.[2]

Reactions for Spillage and Waste Disposal

$$CNCl + 2NaOH \rightarrow NaCN + NaOCl + H_2O$$

$$NaCN + NaOCl \rightarrow \underset{\text{cyanate}}{\underset{\text{sodium}}{NaCNO}} + NaCl$$

REFERENCES

1. Merck 2724.
2. ITI 178.
3. Sax 979.
4. ACGIH 25.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

CYANOGEN IODIDE CNI

VERY POISONOUS

Physical Properties

White needles with very pungent odor and acrid taste; mp, 146–147°C.[1]

Chemical Properties

Soluble in water, alcohol, ether, and volatile oils.[1]

Hazardous Reactions

Phosphorus. Mixture reacts with incandescence forming phosphorus iodide.[2]

Physiological Properties and Health Hazards

Causes convulsions, paralysis, and death from respiratory failure.[1] Very poisonous.[3]

Spillage Disposal

Wear nitrile rubber gloves, eye protection, laboratory coat, and self-contained breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Add to a pail of water. Cautiously, and while stirring, add to an alkaline solution of calcium hypochlorite or household bleach. After 24 hours, decant solution to the drain with 50 times its volume of water. Discard the solid residue with normal refuse.[4,5]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, add slowly and cautiously, while stirring, to a large volume of alkaline calcium hypochlorite solution or household bleach. After 24 hours, flush down the drain with 50 times its volume of water.[5]

Reactions for Spillage and Waste Disposal

$$CNI + 2NaOH \rightarrow NaCN + NaOI + H_2O$$

$$NaCN + NaOCl \rightarrow \underset{\text{sodium cyanate}}{NaCNO} + NaCl$$

REFERENCES

1. Merck 2725.
2. Wohler, F., *Gilbert's Ann*., 69, 281, 1821; NFPA 491M0.
3. W.S. Spector, Ed., *Handbook of Toxicology*, Vol. 1, Saunders, Philadelphia, 1956, p. 82.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ*., 62, A93, 1985; Armour, M.A., *J. Chem. Educ*., 65, A64, 1988; *Haz. Mat. Spills Tec*., 29.1.
5. ITI 178.

CYCASIN $C_8H_{16}N_2O_7$

ANIMAL CARCINOGEN

Synonyms

(Methyl-ONN-azoxy)methyl β-D-glucopyranoside, methylazoxymethanol β-D-glucoside, β-D-glucosyloxyazoxymethane.

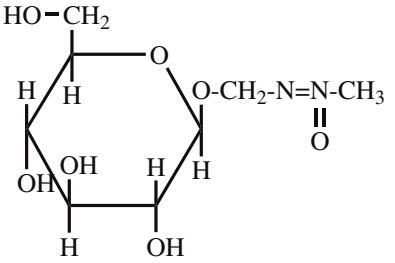
Physical Properties

Needles from water, acetone, and ether; dec, 154°C.[1]

Chemical Properties

Readily soluble in water and dilute ethanol. Insoluble in benzene, chloroform, acetone, and ethyl acetate.[2]

Structure



Physiological Properties and Health Hazards

Animal carcinogen.[2] The acute oral LD_{50} is highly species dependent, due to the extent of *in vivo* decomposition to its biologically active aglycone, methylazoxymethanol. The aglycone, but not cycasin, is mutagenic in the Ames test, and both are teratogenic.[3] LD_{50} (rat) 562 mg/kg.[1] LD_{50} (oral, rabbit) 30 mg/kg.[3]

Waste Disposal

Place in a separate labeled container for disposal by burning. Dissolve or mix material with combustible solvent and burn in a chemical incinerator equipped with afterburner and scrubber.

REFERENCES

1. Merck 2729.
2. IARC 1, 157.
3. Becker, F.F., Ed., *Ethology: Chemical and Physical Carcinogens*, Cancer 1, Plenum Press, New York, 1982, p. 292.

CYCLOHEXANE C_6H_{12}

FLAMMABLE LIQUID, IRRITANT

Synonyms

Hexahydrobenzene, hexamethylene, hexanaphthene.[1]

Physical Properties

Flammable liquid. Solvent odor. Pungent when impure; mp, 6.5°C; bp, 80.7°C. Flash point (closed cup), –18°C.[1]

Fire Hazard

Flammability limits in air, 1.3–8.4% v/v.[1] To fight fire use foam, carbon dioxide, dry chemical spray, and fog.[2]

Chemical Properties

Solubility in water at 23.5°C (w/w) 0.0052%; in methanol at 20°C, 57g in 100 mL; miscible with ethanol, ethyl ether, acetone, benzene, and carbon tetrachloride.[1]

Hazardous Reactions

Dinitrogen Tetraoxide. Addition of liquid dinitrogen tetraoxide to hot cyclohexane caused an explosion.[2–5]

Physiological Properties and Health Hazards

Irritates the eyes, skin, and respiratory system; the inhalation of high concentrations may cause narcosis. Assumed to be irritant and narcotic if taken by mouth.[2,5,6] TLV-TWA 300 ppm (1030 mg/m^3).[7]

Spillage Disposal

Shut off all sources of ignition. Instruct others to keep a safe distance. Wear nitrile rubber gloves, laboratory coat, and self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. Scoop into an appropriate container and package for disposal by burning.[6,8] Spillage site should be washed thoroughly.[6]

Waste Disposal

Waste quantities can be recovered for reuse by distillation. Otherwise, place in a solvent disposal container for disposal by burning in a chemical incinerator equipped with afterburner and scrubber.[6,8]

REFERENCES

1. Merck 2752.
2. Sax 990.
3. B 647.
4. NFPA 491M.
5. Lux 312.
6. Ald 959C.

7. ACGIH 25.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.

CYCLOPHOSPHAMIDE $C_7H_{15}Cl_2N_2O_2P$

HIGHLY TOXIC SUSPECTED CANCER AGENT

Synonyms

N,N-Bis(2-chloroethyl)tetrahydro-2H-1,3,2-oxazaphosphorin-2-amine-2-oxide, cytophosphane, cytoxan, procytox.[1]

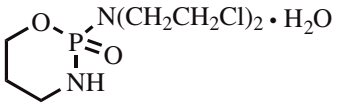
Physical Properties

Fine white, odorless or almost odorless crystalline powder with slightly bitter taste; mp, 41–45°C (monohydrate).[1]

Chemical Properties

Solubility in water, 4 g/100 mL. Slightly soluble in alcohol, benzene, ethylene glycol, carbon tetrachloride, and dioxane; sparingly soluble in ether and acetone. Sensitive to oxidation, moisture, and light; liquefies upon loss of water of crystallization.[2]

Structure



Mode of Action

The drug acts as a purine analogue in the inhibition of DNA synthesis, or as an alkylating agent, or by interaction with sulfhydryl (SH) groups.[3]

Physiological Properties and Health Hazards

LD_{50} orally in mice, rats: 350, 94 mg/kg.[2] Patients suffer anorexia, nausea, and vomiting.[4] Listed as known human carcinogen.[5]

Spillage Disposal

Solutions. Wear goggles, rubber gloves, and protective clothing. Cover the spill with 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate weight of cyclophosphamide, allowing 10 mL of 12% sodium hydroxide to destroy 100 mg of cyclophosphamide in 20 mL of dimethylformamide when refluxed for 4 hours. When reaction is complete, the solution is neutralized by careful addition of 2 M hydrochloric acid (10 mL of concentrated hydrochloric acid added to 50 mL of water), and washed into the drain with a large volume of water.[2,4]

Waste Disposal

Wear protective gloves, clothing, and goggles. To each 2.5 mL of solution containing 50 mg of cyclophosphamide, add 50 mL of 12% solution of sodium hydroxide and 100 mL of dimethylformamide. Allow the mixture to reflux for 4 hours. The cooled solution is neutralized by careful addition of 2 M hydrochloric acid, and washed into the drain with a large volume of water. Thoroughly rinse glassware containing residues of cyclophosphamide with dimethylformamide.

Add rinses to one-half their volume of 12% sodium hydroxide solution and heat the mixture under reflux for 4 hours. Neutralize the cooled solution by careful addition of 2 M hydrochloric acid and wash into the drain with a large volume of water.[3,5]

REFERENCES

1. Merck 2776.
2. Ald 992C.
3. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 125.
4. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, 1982, p. 97.
5. NIEHS III–26.
5. Armour, M.A., Disposal methods for some known or suspect carcinogens, in *Recent Advances of Chemistry and Molecular Biology in Cancer Research*, Dai, Q., Armour, M.A., and Zheng, Q., Eds., Springer-Verlag, Heidelberg, 1993, p. 315.

2,4-D Acid: $C_8H_6Cl_2O_3$

HERBICIDE

Synonyms

Trinoxal, Salvo, Weedone, Chloroxone, 2,4-dichlorophenoxyacetic acid.

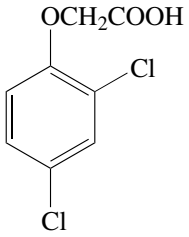
Physical Properties

White powder (or crystals from benzene) having phenolic odor; mp, 138°C[1,2]; amine and ester are liquids.

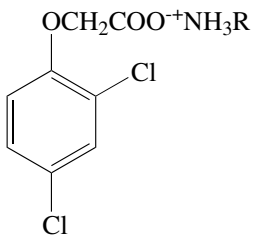
Chemical Properties

The free acid and ester are almost insoluble in water, while the amine is more water soluble; all three forms are soluble in organic solvents.[2] Corrosive.[1]

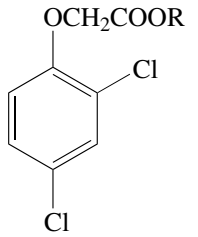
Structure



2,4-D Acid



2,4-D Amine



2,4-D Ester

Physiological Properties and Health Hazards

LD_{50} 368 mg/kg (oral rat); 375 mg/kg (oral, mouse). Causes irritation of eyes and gastrointestinal disturbances.[2] LC_{50} (rainbow trout, 48 hours) 1.1 ppm.[3] TLV-TWA 10 mg/m^3.[4]

Formulations

Emulsifiable concentrate, soluble concentrate, water-soluble powder, and granules.[1,3]

Waste Disposal

2,4-D Acid. Dissolve the 2,4-D (1 g) as far as possible in 200 mL of 10% sodium hydroxide solution; then add 14 g of potassium permanganate and stir the mixture at room temperature for 24 hours. Reduce any excess permanganate remaining at the end of the reaction with sodium bisulfite, and neutralize the solution with dilute hydrochloric acid (1 volume of concentrated acid added to 5 times its volume of water). Pour the solution into the drain and discard any residual brown manganese dioxide with regular refuse.

2,4-D Amine. To each 300 mg of 2,4-D amine, add 100 mL of acidic potassium permanganate solution (17 mL of concentrated sulfuric acid added to 83 mL of water, and then 4.7 g of solid potassium permanganate added). Allow the mixture to stand at room temperature for 24 hours, and then neutralize by the careful addition of 10% sodium hydroxide solution. While stirring, add saturated sodium metabisulfite solution until a colorless solution is formed. Wash this solution into the drain with water.

2,4-D Ester. To each 100 mg of 2,4-D ester, add 50 mL of acidic potassium permanganate solution (17 mL of concentrated sulfuric acid and 4.7 g of potassium permanganate added to 83 mL of water). Stir the solution at room temperature for 4 days, and then carefully neutralize it with 10% sodium hydroxide solution. While stirring, add saturated sodium metabisulfite solution until the mixture is colorless. Wash the liquid into the drain with water.[5]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer before washing other clothes by running the machine through a complete wash cycle with detergent (but without clothing).

2,4-D amine or ester formulation was diluted to field strength (1.25% solution) and used to contaminate cotton denim fabric.[6] The amine formulation is highly water soluble and is readily removed with a single wash; it makes no difference whether a cool (30°C) or hot wash (60°C) is used. At both wash temperatures, less than 1% 2,4-D amine residue remains.

When the ester formulation was used, even a hot (60°C) wash resulted in 55% of the ester residue remaining. A second wash helps to reduce residues; however, the residues remain high.[6]

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DACARBAZINE $C_6H_{10}N_6O$

ANTINEOPLASTIC ANIMAL CARCINOGEN

Synonyms

DIC, DTIC, NSC-45388, DTIC-Dome, Deticene.

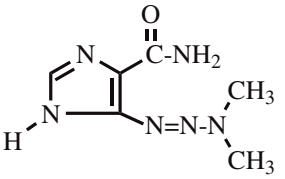
Physical Properties

Ivory microcrystalline substance. Decomposes explosively at 250–255°C; mp also reported as 205°C.[1]

Chemical Properties

Stable in neutral solutions in the absence of light.[1]

Structure



Mode of Action

The drug acts either as a purine analogue in the inhibition of DNA synthesis, or as an alkylating agent, or by interaction with sulfhydryl (SH) groups.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, blood anomalies, alopecia, facial flushing and facial paresthesias, hepatotoxicity, and azotemia.[2] It is carcinogenic in animals. Reasonably anticipated to be a human carcinogen.[3]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover spill area with a 10% sulfuric acid solution. Absorb the liquid by covering with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of the mixture and, very slowly, add it to 10% sulfuric acid, allowing 10 mL of acid for each 1 g of mixture. Allow to stand overnight; neutralize by the careful addition of soda ash and decant the liquid into the drain with water. Discard the solid as normal refuse. Wash the spill area with soap and water.[4]

Waste Disposal

Wear goggles and protective gloves and clothing. Work in the fume hood. Pour waste solutions of dacarbazine into an excess of 10% sulfuric acid and allow to digest overnight. Dilute and wash into the drain with water. Immerse glassware containing residues of dacarbazine in a 10% solution of sulfuric acid and allow to stand overnight. Rinse thoroughly with water before discarding or reusing.[4]

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DACTINOMYCIN $C_{62}H_{86}N_{12}O_{16}$

ANTINEOPLASTIC

Synonyms

Actinomycin D, Cosmegen, Meractinomycin, NSC 3053.

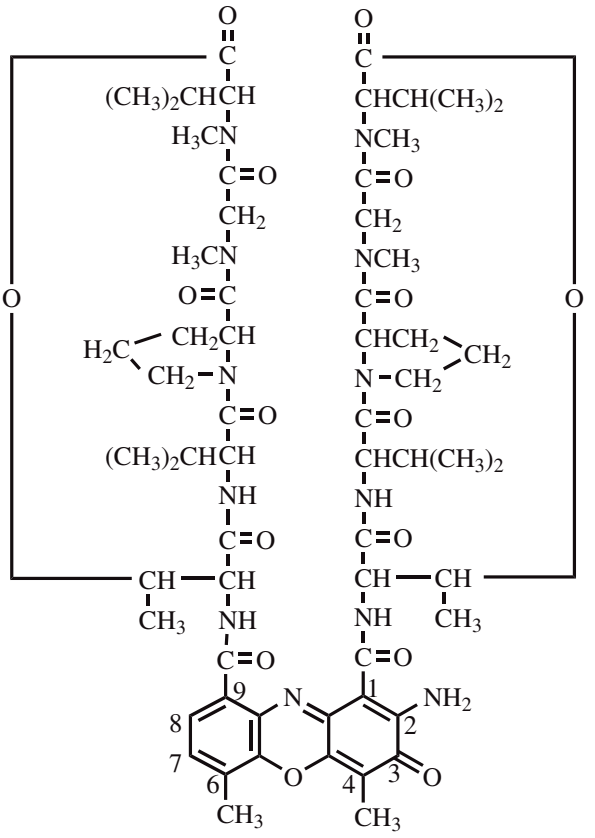
Physical Properties

Bright red prisms from absolute alcohol; dec, 241.5–243°C. Dilute solutions are sensitive to light.[1]

Chemical Properties

Soluble in alcohol, propylene glycol, and water and glycol mixtures.[1]

Structure



Mode of Action

The drug binds to DNA by intercalation between adjacent base pairs of the double helix, thus inhibiting RNA synthesis.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, cramps, diarrhea, bone marrow depression, stomatitis, and skin disorders.[3] LD_{50} (oral, mice and rat) 13.0, 7.2 mg/kg.[1]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of dactinomycin in the spilled solution; to the mixture, add household bleach (5% sodium hypochlorite) allowing 20 mL of bleach for each 0.5 mg of dactinomycin. Allow to stand overnight, and then decant the liquid into the drain with water and discard the solid as normal refuse.[4]

Waste Disposal

Wear goggles and protective gloves and clothing. Work in the fume hood. To 0.25 mg of dactinomycin, add 5 mL of household bleach (5% sodium hypochlorite). After standing at room temperature for 1 hour, wash the solution into the drain with water. Immerse in bleach any vials, syringes, and other glassware that have been in contact with dactinomycin; allow to stand overnight, and then rinse thoroughly with water and discard or reuse.[4]

REFERENCES

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4. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 104; *Haz. Mat. Spills Tec.*, 29.7.

DAUNORUBICIN $C_{27}H_{29}NO_{10}$

ANTINEOPLASTIC

Synonyms

Daunomycin, Cerubidin, Leukaemomycin C, Rubidomycin.

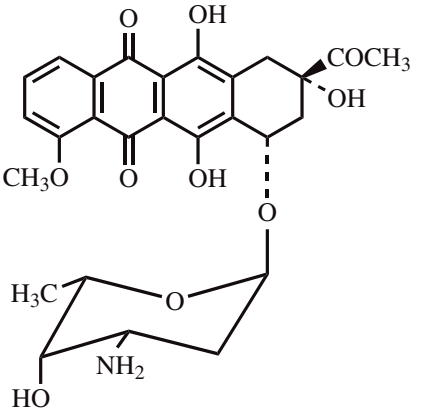
Physical Properties

Thin red needles; mp, 188–190°C (dec).[1]

Chemical Properties

Soluble in water, methanol and aqueous alcohols. Practically insoluble in ether, benzene or chloroform. The color of the aqueous solution changes from pink at acid pH to blue at alkaline pH.[1]

Structure



Mode of Action

The drug intercalates between the base pairs of nucleic acids in DNA, thus inhibiting RNA synthesis.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include vomiting, nausea, anorexia, abdominal constipation, diarrhea, alopecia, and dose-related cardiac toxicity.[2]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Estimate the weight of daunorubicin in the spilled liquid. Pour a solution of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of water) containing 4.7 g of potassium permanganate per 100 mL over the spill, allowing 10 mL of solution per 50 mg of daunorubicin. Allow to react for at least 2 hours. Decolorize with a saturated solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water). Absorb the liquid in a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a pail of cold water. Decant the liquid into the drain with water and discard the solid as normal refuse.[3–5]

Waste Disposal

Wear goggles and protective gloves and clothing. Dissolve daunorubicin (30 mg) in 50 mL of 3 M (8.5 mL of concentrated sulfuric acid slowly added to 41.5 mL of water) sulfuric acid and add 1 g of potassium permanganate while stirring. Continue stirring for 2 hours, neutralize by careful addition of soda ash, and then add a saturated solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water) until a colorless solution is produced. Wash into the drain with water.[3,4]

REFERENCES

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3. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 25.
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2,4-DIAMINOTOLUENE $CH_3C_6H_3(NH_2)_2$

TOXIC, ANIMAL CARCINOGEN

Synonyms

MTD.

Physical Properties

Colorless crystals; mp, 97–99°C; bp, 283–285°C.[1,2]

Chemical Properties

Soluble in water, alcohol, and ether.[2]

Hazardous Reactions

Emits toxic fumes when heated.[3]

Physiological Properties and Health Hazards

Causes conjunctivitis, corneal opacities, irritation of skin and blistering, nausea, vomiting, jaundice, anemia, diarrhea, cyanosis, liver toxicity, and methemoglobinemia.[4,5] Animal carcinogen. Reasonably anticipated to be a human carcinogen.[6] LD_{50} (oral, human) 50 mg/kg; LD_{50} (oral, rat) 500 mg/kg.[3]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl[7] rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the diaminotoluene has been absorbed, scoop the mixture into a plastic pail and add enough water to dissolve the sodium carbonate. Allow the solids to settle and decant the liquid to another container. Discard the solids with normal refuse. To the liquid, add slowly (frothing will occur) 6 M sulfuric acid to pH 2. Stir into the acidified solution sufficient solid potassium permanganate so that the liquid is purple (a drop of the liquid on a filter paper will show a purple ring). Allow the mixture to stand at room temperature for 48 hours, and then neutralize with solid sodium carbonate (frothing will occur), or with a 10% aqueous solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[8,9]

Waste Disposal

Wear eye protection, laboratory coat, and rubber gloves. Dissolve the diaminotoluene (1 mL) in 50 mL of 3 M sulfuric acid (prepared by slowly adding 8 mL of concentrated sulfuric acid to 21 mL of water). Weigh 10 g of potassium permanganate and stir small portions of the solid into the diaminotoluene solution over a period of about 1 hour. Stir the mixture at room temperature for 48 hours, and then neutralize the solution by adding solid sodium carbonate or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until solution is colorless. Decant the clear liquid into the drain and discard any brown solid with regular refuse.[9,10]

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DIAZINON $C_{12}H_{21}N_2O_3PS$

INSECTICIDE

Synonyms

G-2448D, Basudin, Diazol, Garden Tox, Sarodex, Spectracide, O,O-diethyl-O-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorothioate, phosphorothioic acid O,O-diethyl-O-[6-methyl-2-(1-methylethyl)-4-pyrimidinyl] ester, thiophosphoric acid 2-isopropyl-4-methyl-6-pyrimidynyl diethyl ester.

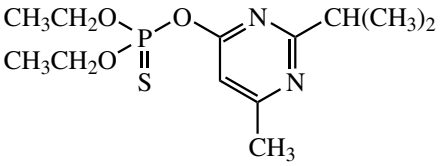
Physical Properties

Colorless liquid, faint ester-like odor; bp, 83–84°C (0.002 mm). Decomposes above 120°C.[1,2]

Chemical Properties

Solubile in water at 20°C, 0.004%; miscible with alcohol, ether, petroleum ether, cyclohexane, benzene, and similar hydrocarbons. More stable in alkaline formulation than when at neutral or acid pH. On storage with traces of water, highly toxic tetraethyl thiopyrophosphate is produced.[1]

Structure



Physiological Properties and Health Hazards

Cholinesterase inhibitor. High acute mammalian toxicity. LD_{50} (oral, rats) 300–850 mg/kg. LC_{50} (rainbow trout, 96 hours) 380 ppb.[3] Toxic to bees, fish and other animals. If taken internally will cause headaches, giddiness, blurred vision, nervousness, weakness, nausea, cramps, diarrhea, discomfort in the chest, sweating, pinpoint pupils, tearing, salivation, and vomiting.[2] TLV-TWA 0.1 mg/m^3.[4]

Formulations

Wettable powder, emulsifiable concentrate, and granules.[1,3]

Waste Disposal

To 200 mg of pure Diazinon, or 0.5 mL of the commercial liquid concentrate, add 100 mL of acidic aqueous potassium permanganate solution (17 mL of concentrated sulfuric acid added to about 70 mL of water, and then 4.7 g of potassium permanganate dissolved in the aqueous acid and the solution made up to 100 mL with water), and stir the mixture at room temperature for 1 hour. If at the end of the reaction time, no excess permanganate remains (determined by placing a drop of the liquid on a piece of filter paper, the presence of a purple ring around the brown spot indicates that permanganate is present), add 1 g of solid potassium permanganate and stir for another 15 minutes. Sodium metabisulfite is then added to the stirred solution until a colorless liquid is formed. The pH of the solution is adjusted to between 6 and 8 with solid sodium carbonate (soda ash; add slowly to avoid excess foaming) or 5% sodium hydroxide solution, and then the liquid is poured into the drain.[5]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer before washing other clothes by running the machine through a complete wash cycle with detergent (but without clothing). A single hot-water (60°C) wash of 100% cotton clothing on which 0.5% diazinon-containing solution has been spilled results in 1% diazinon remaining on the fabric. When concentrated pesticide is spilled, 4% remains on the fabric after washing.[6] When 1% diazinon solution is spilled on 50/50 cotton/polyester-blend clothing, the diazinon residues can be reduced to 1% remaining on the fabric by laundering once with hot water (60°C) or by pretreating with a prewash soil/stain remover and using a warm (50°C) wash.[7]

Emulsifiable concentrate formulation of diazinon, diluted to field strength (0.5% a.i.) was used to contaminate 100% cotton fabric.[7] A single hot-water wash (60°C) results in 1% diazinon remaining on the cotton fabric. When cotton is contaminated with the concentrated (full-strength) diazinon (47.5%), and washed in hot water, 4% pesticide residue remains.

Emulsifiable concentrate formulation of diazinon, diluted to 1.0% a.i. was used to contaminate a 50/50 blend of cotton and polyester. A single warm-water wash (49°C) results in approximately 4% diazinon remaining; however, if the fabric is pretreated with Spray 'n Wash® (a prewash commercial soil and stain remover containing a blend of surfactants and solvents), the pesticide residue is reduced to approximately 1%. Therefore, to reduce diazinon residue to the 1% level, either launder once using hot water (60°C) *or* pretreat with a prewash soil/stain remover and use a warm wash (50°C).[7]

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DIAZOMETHANE CH_2N_2

EXTREME RISK OF EXPLOSION BY SHOCK, FRICTION, FIRE, OR OTHER SOURCES OF IGNITION; TOXIC BY INHALATION

DO NOT STORE SOLUTIONS OF DIAZOMETHANE; FRESHLY PREPARE QUANTITY REQUIRED FOR USE; KEEP COLD

Physical Properties

Yellow gas; bp, –23°C.[1]

Chemical Properties

Soluble in ether and dioxane.[1]

Hazardous Reactions

Gaseous diazomethane may explode on ground glass surfaces and when heated to about 100°C. Safety precautions for the use of diazomethane have been published.[2–7]

Alkali Metals. Explodes on contact.[2]

Calcium Sulfate. Strongly exothermic reaction that may lead to detonation; calcium sulfate should not be used to dry diazomethane. Potassium hydroxide is a suitable drying agent.[8]

Physiological Properties and Health Hazards

Irritates the respiratory system, eyes, and skin. Inhalation may result in chest discomfort, headache, weakness, and, in severe cases, collapse. Powerful allergen. Prevent inhalation of vapor and contact with skin and eyes.[9] TLV-TWA 0.2 ppm (0.34 mg/m^3).[10]

Waste Disposal

Wear leather gloves, heavy face shield, and laboratory coat. Work from behind a body shield. Avoid unnecessary heat, friction, or impact.

In the fume hood, behind a shield, decompose by slowly and cautiously adding a greater-than-stoichiometric amount of acetic acid. Place in solvent disposal container. (Generally, diazomethane has completely reacted when loss of yellow color of the solution is observed.)[11]

Reactions for Spillage and Waste Disposal

$$CH_2 = N^+ = N^- + CH_3COOH \rightarrow CH_3COOCH_3 + N_2$$

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DIBENZ[a,h]ANTHRACENE $C_{22}H_{14}$

ANIMAL CARCINOGEN

Synonyms

1,2,5,6-dibenzanthracene.

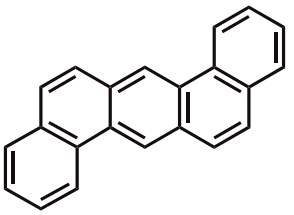
Physical Properties

Monoclinic or orthorhombic crystals. Sublimes; mp, 266°C[1]; bp, 524°C.[2]

Chemical Properties

Soluble in petroleum ether, benzene, toluene, xylene, oils, and other organic solvents; slightly soluble in alcohol and ether; insoluble in water.[1]

Structure



Physiological Properties and Health Hazards

Symptoms of exposure are gastroenteric distress, tremors, convulsions, fever, and dermatitis. Effects of chronic exposure are tumors, cancer, and dermatitis.[3,4] Animal carcinogen. Reasonably anticipated to be a human carcinogen.[5]

Spillage Disposal

Wear protective gloves, laboratory coat, and goggles. Turn off all ventilation and isolate spill area. Add dimethylformamide to completely wet the contaminated surface, and then pour over the area a large amount of a freshly prepared solution containing 4.7 g of potassium permanganate in 100 mL of 3 M sulphuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow to react for at least 1 hour. Absorb the residual solution in a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood, and slowly add it to a beaker of cold water. If necessary, neutralize solution with soda ash. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse.[6,7]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix the material into a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear goggles and protective gloves and clothing. Work in the fume hood. Dissolve the dibenz[a,h]anthracene (5 mg) in acetone (2 mL). Add a freshly prepared solution of 0.5 g of potassium permanganate in 10 mL 3 M sulphuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water). Swirl and allow to stand at room temperature for at

least 1 hour. If necessary add more potassium permanganate solution to maintain the purple color. Cautiously neutralize with 10% aqueous sodium hydroxide and then add, while stirring, a saturated solution of sodium bisulfate (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless solution is formed. Wash into the drain with a large volume of water.[6]

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DIBORANE B_2H_6

FLAMMABLE, FLASH POINT –90°C

Physical Properties

Colorless gas with unpleasant sweet odor; bp, –93°C.[1]

Fire Hazard

Flash point, –90°C; explosive limits, 0.9–98%; auto-ignition temperature, 38–52°C (lowered by moisture).[2] Extinguish from a safe distance with water or liquid nitrogen. Do not use halocarbons.[3]

Chemical Properties

Soluble in carbon disulfide. Decomposes at red heat to boron and hydrogen gas. Hydrolyzes in water to hydrogen and boric acid.[1]

Hazardous Reactions

The addition of anhydrous acids to sodium borohydride to generate borane may result in a dangerously violent reaction.[4]

Air, Benzene, or Moisture. Usually ignites in air and delayed ignition may be followed by violent explosions. The presence of benzene vapor or moisture in air results in an explosive reaction.[5,6]

Chlorine. Mixture reacts explosively at room temperature.[7]

Dimethyl Sulfoxide. Mixture is explosive.[8]

Halocarbons. Halocarbons such as carbon tetrachloride should not be used to extinguish diborane fires because diborane reacts violently with them.[9]

Metals. Forms complex hydrides, which may explode in air, with Li and Al.[9]

Octanal Oxime and Sodium Hydroxide. Very exothermic and sometimes explosive reaction occurs on adding NaOH to a reaction mixture of the oxime and diborane in THF.[9]

Oxygen. Mixture explodes at 105–165°C.[10]

Tetravinyllead. Mixture explodes at room temperature.[9]

Physiological Properties and Health Hazards

Inhalation may produce irritation of lungs and pulmonary edema.[1] TLV-TWA 0.1 ppm (0.11 mg/m^3).[11]

Waste Disposal

Leaking Cylinder. Wear neoprene gloves and self-contained respirator. Transfer the cylinder to a fume hood. Attach a long piece of flexible tubing to the cylinder valve outlet. Adjust the valve to give a moderate flow of diborane. Introduce the gas to the bottom of a tall vessel containing water. Diborane is hydrolyzed by water to hydrogen and boric acid.[12]

Borane-Amine Complexes or Borane Tetrahydrofuran. Deactivate by slow addition of a mixture of the appropriate solvent and butanol. Place in a separate labeled container for disposal by burning. Spray into furnace equipped with afterburner and scrubber.[12,13]

REFERENCES

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2. B 1839.

3. NFPA 49.
4. B 64.
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6. Schlessinger, H.I. et al., *Chem. Rev.*, 31, 8, 1942.
7. Mellor, Vol. 9, 55, 396, 1939; Vol. 8, 65, 1939; Vol. 6, 219, 1940; Vol. 5, 37, 1941.
8. B 300.
9. B 70, 71.
10. Whatley, A.T. et al., *J. Am. Chem. Soc*., 76, 1997, 1954.
11. ACGIH 26.
12. Braker, W., Mossman, A.L., *Matheson Gas Data Book*, 6th ed., Matheson Gas Products, East Rutherford, NJ, 1980, p. 219.
13. Ald 471.

1,2-DIBROMO-3-CHLOROPROPANE $C_3H_5Br_2Cl$

ANIMAL CARCINOGEN

Synonyms

3-Chloro-1,2-dibromopropane, DBCP, OS 1897, Fumazone, Nemafume, Nemagon.

Physical Properties

Brown liquid, pungent odor[1]; mp, 6.7°C[2]; bp, 196°C.[1]

Chemical Properties

Slightly soluble in water; miscible with oils, dichloropropane, and isopropyl alcohol.[1]

Hazardous Reactions

Combustible liquid; flash point (o.c.), 76.7°C.[2]

Physiological Properties and Health Hazards

Irritating to skin and mucous membranes; narcotic in high concentrations.[1] It has been implicated as a cause of male sterility in factory workers.[3] May cause diminished renal function and cirrhosis of the liver.[4] Animal carcinogen. Reasonably anticipated to be a human carcinogen.[3–5] LD_{50} (oral, rat) 0.17 g/kg.[1]

Spillage Disposal

Wear protective gloves, laboratory coat, goggles, and breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container and either label and send for disposal by burning[7] or, in the fume hood, add the mixture to a solution of 85% potassium hydroxide pellets in ethanol, allowing 0.36 mol (23.7 g) of potassium hydroxide and 100 mL of ethanol for each 0.1 mol (23.7 g) of 1, 2-dibromo-3-chloropropane to be destroyed. Heat the mixture under reflux for 8 hours. Decant the solution into the drain with a large volume of water. Treat the solid as normal refuse.[6–8]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Burn in a furnace equipped with afterburner and scrubber.[7]

Small Quantities. Wear protective gloves, clothing, and goggles. Work in the fume hood. Place 23.7 g (0.36 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, and dropping funnel. Add 100 mL of 95% ethanol rapidly. Heat the solution to gentle reflux and add 1,2-dibromo-3-chloropropane (23.7 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring for 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain with a large volume of water.[8]

REFERENCES

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3. Sax 1101.
4. Sittig, M., *Handbook of Toxic and Hazardous Chemicals and Carcinogens*, Noyes Publications, Park Ridge, NJ, 1991, p. 547.
5. NIEHS III–100.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
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8. PP 61.

1,2-DIBROMOETHANE $BrCH_2CH_2Br$

HUMAN CARCINOGEN, HARMFUL VAPOR, TOXIC IN CONTACT WITH SKIN

Synonyms

Ethylene dibromide.

Physical Properties

Colorless liquid with sweetish chloroform-like odor; bp, 131–132°C.[1]

Chemical Properties

Soluble in 250 parts water; miscible with alcohol and ether.[1]

Hazardous Reactions

Magnesium. Reaction to give Grignard compounds may be violent.[2]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and eyes. Liquid irritates the eyes and skin. Swallowing or skin absorption results in nausea, vomiting, and liver and kidney damage.[3] Avoid breathing vapor. Avoid contact with skin and eyes.[3] Animal carcinogen; reasonably anticipated to be a human carcinogen.[4] TLV, no exposure permitted.[5]

Spillage Disposal

Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a suitably labeled container for disposal by burning. Site of spillage should be washed thoroughly with water and soap or detergent.[4,6,7]

Waste Disposal

Place in halogenated solvent disposal container for disposal by burning in a furnace with afterburner and alkali scrubber.[6]

REFERENCES

1. Merck 3830.
2. B 1322.
3. Lux 324.
4. NIEHS III–102.
5. ACGIH 32.
6. ITI 283.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.

DIBUTYL ETHER $(CH_3CH_2CH_2CH_2)_2O$

FLAMMABLE

Synonyms

n-Butyl ether.

Physical Properties

Colorless liquid; bp, 142°C.[1]

Chemical Properties

Insoluble in water; miscible with alcohol or ether.[2] May form explosive peroxides on exposure to light and air. These should be decomposed before the ether is distilled.[1]

Fire Hazard

Flash point, 25°C; explosive limits, 1.5–7.6%; ignition temperature, 194°C.[3] Extinguish fire with dry chemical, carbon dioxide, or foam.[4]

Hazardous Reactions

Peroxides formed during storage can be removed by percolation through alumina.[5]

Chlorine and Ammonium Sulfate. Mixture may explode.[6]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system. The liquid irritates the eyes, and is considered to present some hazard by skin absorption. Avoid breathing vapor. Avoid contact with skin and eyes.[1] LD_{50} (oral, rat) 7.4 mg/kg.[2]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriately labeled container for disposal by burning. Ventilate site of spillage well to evaporate remaining liquid and dispel vapor.[7,8]

Waste Disposal

Containers of dibutyl ether, which have been opened and are more than 1 year old, may contain hazardous quantities of peroxides. Especially if they have screw caps, containers should not be opened, and should be disposed of by the appropriate authorities. When the container can be opened safely, check for peroxides as follows: Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve 100 mg of potassium iodide in 1 mL of glacial acetic acid. Add to 1 mL of the dibutyl ether. A pale yellow color indicates a low concentration (0.001–0.005%) and a bright yellow or brown color a high concentration (above 0.01% and hazardous) of peroxide in the sample.[9]

To remove peroxides, wear butyl rubber gloves, laboratory coat, and eye protection. Pour the ether (100 mL) into a separatory funnel and shake with a freshly prepared 50% aqueous solution of sodium metabisulfite (20 mL) for 3 minutes. Release the pressure in the funnel at 10-second intervals. Separate the aqueous layer and wash the ether layer with water (3×10 mL). Retest the

ether for the continued presence of small amounts of dialkyl peroxides that are not reduced by the metabisulfite treatment. If peroxides are absent, the ether can be dried for reuse or packaged for disposal by burning. If peroxides are still present, in the fume hood place the ether in a 250-mL round-bottom flask equipped with a condenser and add a solution of 100 mg of potassium iodide in 5 mL of glacial acetic acid and 1 drop of concentrated hydrochloric acid. Reflux gently on the steam bath for 1 hour.[8] Package the ether for disposal by burning.[7]

REFERENCES

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2. Merck 1568.
3. B 1849.
4. NFPA 49.
5. B 767.
6. Anonymous, *Chem. Eng. News*, 44, 46, 1966.
7. Ald 631B.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 278.
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DI-*tert*-BUTYL PEROXIDE $(CH_3)_3C\text{-}O\text{-}O\text{-}C(CH_3)_3$

EXPLOSIVE, PREPARATIVE HAZARD

See also Peroxides.

Physical Properties

Colorless flammable liquid[1]; bp, 109°C.[2]

Fire Hazard

Flash point, 18°C.[2]

Chemical Properties

Insoluble in water; soluble in organic solvents.[1]

Hazardous Reactions

Preparation of di-*tert*-butyl peroxide from *tert*-butyl alcohol and 50% hydrogen peroxide: 78% sulfuric acid can result in an explosion.[3] Di-*tert*-butyl peroxide itself is one of the most stable of the dialkyl peroxides.[4]

Physiological Properties and Health Hazards

Vapor irritating to the respiratory system, skin, and eyes.[5]

Spillage Disposal

Eliminate all sources of ignition. Wear nitrile rubber gloves; large, heavy face shield; and laboratory coat. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Sprinkle water on the mixture and, using a soft plastic scoop, transfer to a container and transport to the fume hood. Package the solid for disposal by burning.

Alternatively, add the solid to glacial acetic acid containing sodium iodide (allow 20 mL of acid and 2.5 g of sodium iodide for each 1 g of peroxide). Stir the mixture at room temperature for 24 hours. Neutralize the solution by slowly adding sodium carbonate while stirring. Wash the liquid into the drain. Treat the solid as normal refuse.[6]

Waste Disposal

CAUTION: Keep stock low and date each container as prepared. Never transfer to glass-stoppered containers or screw-cap bottles, which can cause dangerous friction.

Small Quantities. Wear nitrile rubber gloves; eye protection; large, heavy face shield; and laboratory coat. Work in the fume hood behind a heavy body shield. Slowly add the dibutyl peroxide (1 g) to a solution of sodium iodide (2.5 g) in hydrochloric acid (1 mL) and glacial acetic acid (20 mL). Stir at room temperature for 24 hours. Neutralize the solution by the slow addition of sodium carbonate while stirring. Wash into the drain.[6]

REFERENCES

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2. NFPA 325M.
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5. Lux 327.
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3,3'-DICHLOROBENZIDINE $(H_2N\text{-}C_6H_3Cl)_2$

ANIMAL CARCINOGEN

Physical Properties

Needles; mp, 132–133°C.[1]

Chemical Properties

Almost insoluble in water; readily soluble in alcohol, benzene, and glacial acetic acid.[1]

Physiological Properties and Health Hazards

The U.S. Food and Drug Administration has declared this substance and its salts to be carcinogens.[1] Reasonably anticipated to be a human carcinogen.[2] Absorbed through skin. No TLV.[1,3]

Spillage Disposal

Wear butyl rubber gloves, protective laboratory coat, eye protection, self-contained breathing apparatus, and protective shoes.

On skin. Wash with strong soap solution immediately. Rinse well.

Contaminated gloves, clothing, and shoes. Remove and clean at once or destroy by burning. Sweep solid spills into an appropriate container for disposal by burning.[4] Wash the site thoroughly with strong soap solution.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[4]

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To a 50-mL, three-necked, round-bottom flask equipped with stirrer, thermometer, and dropping funnel, add 1 mL of water, 3 mL of concentrated hydrochloric acid, and 2 g (0.008 mol) of 3,3'-dichlorobenzidine. The temperature is maintained at −5 to 0°C by a cooling bath, while 0.2 g (0.0084 mol) of 97% sodium nitrite dissolved in 1.4 mL of water is added dropwise to the solution or slurry of dichlorobenzidine hydrochloride. Stirring is continued an additional 30 minutes after addition is complete. While maintaining the temperature at −5 to 0°C, 16.6 mL (0.16 mol) of 50% hypophosphorous acid (precooled to 0°C) is added over 10–15 minutes. Stirring is continued for 1 hour. The mixture is allowed to stand at room temperature for 24 hours and is then extracted with 2 × 4-mL portions of toluene. The toluene extract of the deaminated dichlorobenzidine is packaged for disposal by incineration. The aqueous phase is washed down the drain.[5]

To each 9 mg of dichlorobenzidine, add 10 mL of 0.1 M hydrochloric acid (slowly add 1 mL of concentrated acid to 119 mL of cold water). Mix to dissolve. Add 5 mL of 0.2 M potassium permanganate solution (0.3 g of solid $KMnO_4$ dissolved in 10 mL of water) and 5 mL of 2.0 M sulfuric acid (prepared by carefully adding 1 mL of concentrated acid to 8 mL of cold water). Mix and let stand overnight (at least 10 hours). Decolorize if necessary with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (20 g of NaOH dissolved in 100 mL of cold water). Discard the remaining solution into the drain with at least 50 times its volume of water.[6]

Reactions for Spillage and Waste Disposal

$$(Cl\text{-}C_6H_3\text{-}NH_2)_2 + 2HCl \rightarrow (Cl\text{-}C_6H_3NH_2)_2 \cdot 2HCl$$

$$(Cl\text{-}C_6H_3\text{-}NH_2) \cdot 2HCl \xrightarrow[2)\ H_3PO_2]{1)\ HNO_2} (C_6H_4Cl)_2$$

3,3'-dichlorobiphenyl

REFERENCES

1. Merck 3084.
2. NIEHS III–105.
3. ACGIH 26.
4. ITI 075.
5. PP 70.
6. Cas 64, 24; PPL 162.

DICHLORODIMETHYLSILANE $(CH_3)_2SiCl_2$

HIGHLY FLAMMABLE

Physical Properties

Colorless fuming liquid; bp, 70.5°C.[1]

Fire Hazard

Highly flammable.[2] Flash point, –9°C. Explosive limits, 3.4–9.5%.[3]

Hazardous Reactions

Water. Violent reaction.[2]

Physiological Properties and Health Hazards

Vapor irritates the eyes and respiratory system. Liquid burns skin and will cause severe internal damage if ingested. Avoid contact with skin, eyes, and clothing.[2]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Slowly add the solid to a pail of cold water. If necessary, neutralize the solution with sodium carbonate. Wash the liquid into the drain. Treat the solid as normal refuse.[4,5]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

Where possible, the compound should be redistilled and reused.

Small Quantities. Place 500 mL of water in a 1-L, three-necked, round-bottom flask equipped with a stirrer, addition funnel, and thermometer. Cool the flask in an ice-water bath. Place 10 mL of dichlorodimethylsilane in the funnel and add it dropwise to the stirred water. Do not allow the temperature to rise above 20°C. When addition is complete, neutralize the solution with sodium carbonate and wash the liquid into the drain.[4,5]

Reactions for Spillage and Waste Disposal

$$(CH_3)_2SiCl_2 + 2H_2O \rightarrow \underset{\text{dimethylsilanol}}{(CH_3)_2Si(OH)_2} + 2HCl$$

REFERENCES

1. Sax 1140.
2. Lux 361.
3. B 1840
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
5. Havill, M.E., Joffe, I., and Post, H.W., *J. Org. Chem.*, 13, 280, 1948.

DICHLOROMETHANE CH_2Cl_2

TOXIC IRRITANT

Synonyms

Methylene chloride, methylene dichloride, methylene bichloride.[1]

Physical Properties

Colorless, volatile liquid with chloroform-like odor[2]; mp, –97°C; bp, 40°C.[2,3]

Chemical Properties

Soluble in about 50 parts water; miscible with alcohol, ether, and dimethylformamide (DMF).[1]

Hazardous Reactions

Previously thought to be nonflammable, it is flammable in the range of 12–19% in ambient air, given a high level of ignition energy.[4]

Air and Methanol. Mixture is flammable in the presence of less than 0.5% volume of methanol.[5]

Metals. Lithium shavings or sodium interact with dichloromethane, becoming shock- or impact-sensitive explosives.[6]

Physiological Properties and Health Hazards

The vapor irritates the eyes and respiratory system and may cause headache and nausea; high concentrations may result in cyanosis and unconsciousness.[2] Harmful if swallowed or absorbed through the skin.[7] TLV-TWA 50 ppm, 174 mg/m^3. Reasonably anticipated to be a human carcinogen.[8]

Spillage Disposal

Turn off all sources of ignition and evacuate the area. Wear breathing apparatus, nitrile rubber gloves, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all the liquid has been absorbed, scoop into an appropriate container and package for disposal by burning.[7,9]

Waste Disposal

Place in a labeled solvent container for disposal by burning.[3]

REFERENCES

1. Merck 6088.
2. Lux 334.
3. Aldrich 551.
4. Downey, J.R., *Chem. Eng. News*, 61, 2, 1983.
5. Coffee, R.D., et al., *J. Chem. Eng. Data*, 17, 89, 1972.
6. B 1315, 1370.
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DICHLOROMETHOTREXATE $C_{20}H_{20}Cl_2N_8O_5$

ANTINEOPLASTIC

Synonyms

N-(3,5-Dichloro-4{[(2,4-diamino-6-pteridinyl)methyl]methylamino}benzoyl)-L-glutamic acid, 4-amino-10-methyl-3',5'-dichloro-pteroyl glutamic acid, NSC 29630.

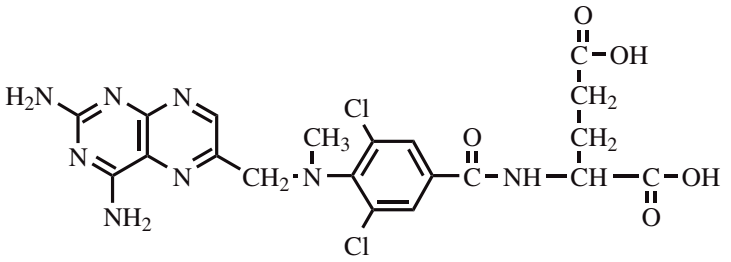
Physical Properties

Square platelets from 50% aqueous alcohol; bright yellow, odorless crystalline powder; mp, 185–204°C (monohydrate).[1]

Chemical Properties

Practically insoluble in water, ethanol, chloroform, and diethyl ether; freely soluble in dilute solutions of alkaline hydroxides and carbonates; soluble in dilute hydrochloric acid.[1]

Structure



Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, diarrhea, damage to intestinal mucosa, depressed bone marrow, and hepatic toxicity (high dose). LD_{50} in rats (intraperitoneal), 30 mg/kg.[2]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Wash the spill area with soap and water. Estimate the weight of absorbing mixture. Add to mixture a 3 M solution of sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7g of potassium permanganate, allowing 12 mL of solution per 10 mg of dichloromethotrexate. Allow the mixture to stand overnight, neutralize by careful addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite until a colorless liquid is formed. Decant the liquid into the drain with water, and discard the residue as normal refuse.[2,3]

Waste Disposal

Wear goggles and protective gloves and clothing. Work in the fume hood. To a solution of chloromethotrexate (10 mg) prepared by stirring at room temperature with 10 mL of water for 30 minutes, add 2 mL of concentrated sulfuric acid and 0.5 g of potassium permanganate. Allow the mixture to stir at room temperature for an additional hour. After careful neutralization with 10%

sodium hydroxide solution, add, while stirring, a saturated solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water) until a colorless solution is formed. Wash the solution into the drain with a large volume of water.[2,3] Immerse glassware, including vials, that contain residues of dichloromethotrexate in a solution prepared by dissolving 4.7 g of potassium permanganate in 83 mL of water and 17 mL of concentrated sulfuric acid. After standing overnight, thoroughly rinse the glassware with water, and then discard or reuse it. Neutralize the permanganate solution by the careful addition of 10% sodium hydroxide, and then stir sodium bisulfite into the mixture until a colorless solution is formed. Wash this solution into the drain with water.[2,3]

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2. Frei, E., III et al., *Clin. Pharmacol. Ther.*, 6, 160, 1965.
3. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, 1994, p. 103; *Haz. Mat. Spills Tec.*, 29.7.

DIEPOXYBUTANE (DL) $C_4H_6O_2$

ANIMAL CARCINOGEN

Synonyms

Erythritol anhydride, bioxiran, butadiene dioxide, butanedione, 1,2,3,4-diepoxybutane.

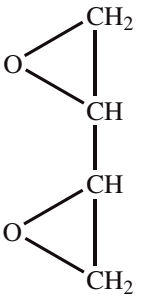
Physical Properties

Colorless liquid; bp, 138°C[1]; mp, 4°C.[2]

Chemical Properties

Miscible in water; hydrolyzes to erythritol.[1]

Structure



Hazardous Reactions

Flammable.[3]

Physiological Properties and Health Hazards

Highly Toxic. Causes cancer in mice.[4,5] Minor exposure causes swelling of the eyelids, painful eye irritation, and upper respiratory tract irritation within 6 hours. Readily absorbed through the skin and respiratory tract.[4] Reasonably anticipated to be a human carcinogen.[5]

Spillage Disposal

Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Wearing protective gloves, goggles, and a laboratory coat, scoop the mixture into a plastic container, label, and either send for disposal by burning, or in the fume hood, add the mixture to a solution of potassium hydroxide in ethanol, allowing 0.24 mol (15.8 g) of 85% potassium hydroxide pellets and 65 mL of ethanol for each 0.1 mol (8.6 g) of diepoxybutane to be destroyed. Heat the mixture under reflux for 8 hours. Decant the cooled solution into the drain with a large volume of water. Treat the solid as normal refuse. Wash the spill area thoroughly with soap and water.[3,6,7]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Burn in a furnace equipped with afterburner and scrubber.[3]

Small Quantities. Wear goggles and protective gloves and clothing. Work in the fume hood. Place 15.8 g (0.24 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, and dropping funnel. Rapidly add 65 mL of 95% ethanol. Heat the solution to gentle reflux and add diepoxybutane (8.6 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring for 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain with a large volume of water.[7]

REFERENCES

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2. Aldrich 284.
3. Ald 597C.
4. IARC 11, 115.
5. NIEHS III–597C.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
7. PP 61.

DIETHYL ETHER $(C_2H_5)_2O$

EXTREMELY FLAMMABLE, MAY FORM EXPLOSIVE PEROXIDES, HARMFUL VAPOR

Synonyms

Ethyl ether, ethoxyethane.

Physical Properties

Colorless liquid with sweet, pungent odor; bp, 34.6°C.[1]

Fire Hazard

Flash point, –45°C; auto-ignition temperature, 160°C.[1] Flammable limits 1.9–36%. Extinguish fire with dry chemical or carbon dioxide.[2]

Chemical Properties

Slightly soluble in water (6.05% at 25°C); miscible with low aliphatic alcohols, benzene, chloroform, petroleum ether, other fat solvents, and many oils. Liable to form explosive peroxides on exposure to air and light.[1]

Hazardous Reactions

Air. Forms explosive peroxides in air and light[3]; may explode when mixed with liquid air.[4] Methods for the control of peroxide formation have been published.[5]

Bromine. Mixture may erupt violently.[6]

Bromine Trifluoride. Explodes on contact.[7]

Chlorine. Ether ignites on contact with chlorine.[8]

Oxidizing Agents. May explode on contact with anhydrous perchloric acid[9]; ignites on contact with chromyl chloride[10]; explodes violently on contact with a mixture of concentrated nitric and sulfuric acids,[11] and may react violently with concentrated nitric acid[12]; simultaneous contact of sodium peroxide with water and diethyl ether causes ignition[13]; and contact with ozone produces explosive diethyl peroxide.[14]

Sulfur. Residue from evaporation of ethereal extract of sulfur may explode violently.[15]

Sulfuryl Chloride. Solution in ether may decompose vigorously.[16]

Uranyl Nitrate. The presence of ether in the crystals may result in explosions when the crystals are disturbed. Solutions of the salt in ether should not be exposed to sunlight to avoid the possibility of explosions.[17,18]

Physiological Properties and Health Hazards

Mildly irritating to skin and mucous membranes.[1] Breathing vapor causes drowsiness and, in high concentration, unconsciousness. Continued exposure leads to symptoms resembling chronic alcoholism. Avoid breathing vapor.[19] TLV-TWA 400 ppm (1210 mg/m^3); TLV-STEL 500 ppm (1520 mg/m^3).[20]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and PVA or silver-shield rubber gloves.[21] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite),

and sand. Transfer to an appropriate container labeled for disposal by burning. Ventilate site of spillage well to evaporate remaining liquid and dispel vapor.[19,22]

Waste Disposal

Containers of diethyl ether that have been opened and are more than 1 year old may contain hazardous quantities of peroxides. Especially if they have screw caps, these containers should not be opened, but should be disposed of by the appropriate authorities. When the container can be opened safely, check for peroxides as follows: Wear PVA or silver shield rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve 100 mg of potassium iodide in 1 mL of glacial acetic acid. Add to 1 mL of the diethyl ether. A pale yellow color indicates a low concentration (0.001–0.005%) and a bright yellow or brown color indicates a high concentration (above 0.01% and hazardous) of peroxide in the sample.[23]

To remove peroxides, wear PVA or silver-shield rubber gloves, laboratory coat, and eye protection. Pour the ether (100 mL) into a separatory funnel and shake with a freshly prepared 50% aqueous solution of sodium metabisulfite (20 mL) for 3 minutes. Release the pressure in the funnel at 10-second intervals. Separate the aqueous layer and wash the ether with water (3×10 mL). Retest the ether for the continued presence of small amounts of dialkly peroxides that are not reduced by the metabisulfite treatment. If peroxides are absent, the ether can be dried for reuse or packaged for disposal by burning. If peroxides are still present, in the fume hood, place the ether in a 250-mL round-bottom flask equipped with a condenser, and add a solution of 100 mg of potassium iodide in 5 mL of glacial acetic acid and 1 drop of concentrated hydrochloric acid. Reflux gently on the steam bath for 1 hour.[22] Package the ether for disposal by burning.

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DIETHYL SULFATE $(C_2H_5)_2SO_4$

HARMFUL VAPOR, HARMFUL IN CONTACT WITH SKIN

Synonyms

Ethyl sulfate.

Physical Properties

Colorless oily liquid with peppermint odor; bp, 209°C with decomposition.[1]

Fire Hazard

Flash point, 104°C; auto-ignition temperature, 436°C. Lower flammable limit, 4.1%. Extinguish with carbon dioxide, dry chemical, foam, or water spray.[2]

Chemical Properties

Insoluble in water; miscible with alcohol and ether.[1]

Hazardous Reactions

2,7-Dinitro-9-phenylphenanthridine, Water. Violent reaction occurs, forming large volume of solid black foam.[3]

Potassium t-Butoxide. Contact of a few drops of liquid sulfate with solid butoxide causes ignition.[4]

Physiological Properties and Health Hazards

Its effect on humans has not been recorded, but authorities consider that animal experiments justify its classification as a dangerous chemical. It is assumed to be poisonous or an irritant by inhalation, eye and skin contact, and ingestion. LD_{50} (oral, rat) 880 mg/kg.[1] Avoid breathing vapor. Avoid contact with skin and eyes.[5]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[6] When the diethyl sulfate has been absorbed, scoop the mixture into a plastic container and package for disposal by burning. Wash spill site with soap and water.[7]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the diethyl sulfate in a flammable solvent and burn in a furnace equipped with afterburner and scrubber.[7]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. With brisk stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. Heat the solution to gentle reflux and add the diethyl sulfate (15.4 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring (to prevent bumping) for another 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[8]

Reactions for Spillage and Waste Disposal

$$C_2H_5OH + (C_2H_5)_2SO_4 + 2KOH \rightarrow 2C_2H_5OH + K_2SO_4$$

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7. Ald 1226C.
8. PP 61.

DI-ISOBUTYLALUMINUM HYDRIDE $[(CH_3)_2CHCH_2]_2AlH$ (and ALKYLALUMINUMS generally)

IGNITES IN AIR

Physical Properties

Clear colorless liquid, usually shipped as a solution in hydrocarbon.[1]

Fire Hazard

Pyrophoric. Extinguish with dry sand, dry chemical, or carbon dioxide. Do not use water; a violent reaction may result.[1]

Chemical Properties

Ignites spontaneously on exposure to air. Reacts violently with water, oxidizing agents, halogenated hydrocarbons, alcohols, and other oxygen-containing compounds.[1]

Hazardous Reactions

Ignites in air except when in less than 25% solution in hydrocarbon solvents.[2]

Physiological Properties and Health Hazards

TLV-TWA 2 mg/m^3.[3]

Spillage Disposal

Eliminate all sources of ignition. Wear butyl rubber gloves, large and heavy face shield, goggles, and laboratory coat. Cover spill with dry sand. Scoop into a container and transfer to the fume hood. Decompose by cautious addition of dry butanol (40 mol butanol to 1 mol hydride or 26 mL/g hydride) until reaction ceases. Slowly add mixture to a pail of cold water. Let stand for 24 hours. Neutralize with 6 M hydrochloric acid (cautiously add concentrated acid to an equal volume of cold water). Decant the solution to the drain. Treat the solid residue as normal refuse.[4,5]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Mix the hydride with dry sand. Slowly and cautiously add dry *n*-butyl alcohol (40 mol butanol to 1 mol hydride, 26 mL/g hydride) until reaction ceases. Carefully add this mixture to a pail of cold water. Let stand for 24 hours. Neutralize with 6 M hydrochloric acid. Decant the solution to the drain. Treat the solid residue as normal refuse.[4,5]

Reactions for Spillage and Waste Disposal

$$(C_4H_9)_2AlH + 3C_3H_7CH_2OH \rightarrow (C_3H_7CH_2O)_3Al + 2C_4H_{10} + H_2$$

$$(C_3H_7CH_2O)_3Al + 3H_2O \rightarrow 3C_3H_7CH_2OH + \underset{\substack{\text{aluminum}\\ \text{hydroxide}\\ \text{(insoluble)}}}{Al(OH)_3}$$

REFERENCES

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DI-ISOPROPYL ETHER $(CH_3)_2CHOCH(CH_3)_2$

EXTREMELY FLAMMABLE, MAY FORM EXPLOSIVE PEROXIDES

Synonyms

Isopropyl ether.

Physical Properties

Colorless liquid; bp, 68–69°C.[1]

Fire Hazard

Flash point, –28°C; ignition temperature, 443°C; flammable limits, 1.4–7.9%.[2] Explosive limits, 1.4–21%.[3] Extinguish with dry chemical or carbon dioxide.[2]

Chemical Properties

Slightly soluble in water (0.2% at 20°C); miscible with alcohol and ether.[1] Readily forms explosive peroxides on exposure to light and air.[4]

Hazardous Reactions

This ether forms peroxides on exposure to air in only a few hours.[4,5] It can be stabilized by the addition of N-benzyl-*p*-aminophenol (16 ppm) or 50 ppm of diethylenetriamine, triethylenetetramine, or tetraethylene pentamine.[4,6] Many examples of explosions of the peroxidized ether have been documented.[4,6,7]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. Liquid irritates the eyes and may cause skin dermatitis. Inhalation or swallowing may result in headache, dizziness, nausea, vomiting, and narcosis. Avoid breathing vapor. Avoid contact with skin and eyes.[5] TLV-TWA 250 ppm (1040 mg/m^3); TLV-STEL 310 ppm (1300 mg/m^3).[8]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to an appropriate labeled container for disposal by burning. Ventilate site of spillage well to evaporate remaining liquid and dispel vapor.[9,10]

Waste Disposal

Containers of di-isopropyl ether that have been opened, or have been stored for more than 3 months, may contain hazardous quantities of peroxides. Especially if crystals are present, the container should be handled only by appropriate authorities such as a bomb squad. Where the container can be opened safely, check for peroxides as follows: Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve 100 mg of potassium iodide in 1 mL of glacial acetic acid. Add to 1 mL of di-isopropyl ether. A pale yellow color indicates a low concentration (0.001–0.005%) and a bright yellow or brown color indicates a high concentration (above 0.01% and hazardous) of peroxide in the sample.[11]

To remove peroxides, wear butyl rubber gloves, laboratory coat, and eye protection. Pour the ether (100 mL) into a separatory funnel and shake with a freshly prepared 50% aqueous solution of sodium metabisulfite (20 mL) for 3 minutes. Release the pressure in the funnel at 10-second intervals. Separate the aqueous layer and wash the ether layer with water (3×10 mL). Retest the ether for the continued presence of small amounts of dialkyl peroxides that are not reduced by the metabisulfite treatment. If peroxides are absent, the ether can be dried for reuse or packaged for disposal by burning. If peroxides are still present, in the fume hood, place the ether in a 250-mL round-bottom flask equipped with a condenser, and add a solution of 100 mg of potassium iodide in 5 mL of glacial acetic acid and 1 drop of concentrated hydrochloric acid. Reflux the mixture gently on a steam bath for 1 hour.[9] Package the ether for disposal by burning.[10]

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4-DIMETHYLAMINOAZOBENZENE $C_{14}H_{15}N_3$

ANIMAL CARCINOGEN

Synonyms

N,N-Dimethyl-4-(phenylazo)benzenimine, butter yellow, methyl yellow, solvent yellow 2, CI 11020.

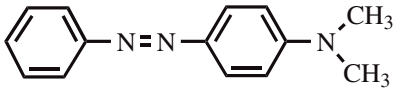
Physical Properties

Yellow crystals; leaflets from ethanol; mp, 114–117°C.[1] Decomposes on exposure to heat or light.[2]

Chemical Properties

Insoluble in water; soluble in alcohol, benzene chloroform, ether, petroleum ether, mineral acids, and oils. Red at pH 2.9, yellow at pH 4.02.[1]

Structure



Physiological Properties and Health Hazards

Animal carcinogen.[2–5] Factory workers may develop contact dermatitis.[4] The characteristic acute toxic effect is methemyglobinemia. LD_{50} (oral, rat) 200 mg/kg[4]; LD_{50} (i.p., mouse) 500 mg/kg; LD_{50} (oral, mouse) 300 mg/kg.[5] Reasonably anticipated to be a human carcinogen.[2]

Spillage Disposal

Wear dust mask, goggles, rubber gloves, and protective clothing. Carefully scoop the solid into a beaker and transport to the fume hood. Dispose of the material using the waste disposal procedure. Wash the spill site with soap and water disposing of cleaning materials in a sealed bag or container for burning.[6,7]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve the compound in a flammable solvent and spray into a furnace equipped with afterburner and scrubber.

Small Quantities. Wear goggles, gloves, and protective clothing. To 2 g of 4-dimethylaminoazobenzene in a 250-mL Erlenmeyer flask, add 40 mL of 6 M hydrochloric acid (20 mL of concentrated hydrochloric acid slowly added to 20 mL of cold water). Stir the deep cherry-red mixture for 15 minutes, and then add 3.0 g of granulated tin and continue stirring for 4 hours. The mixture should be brown in color. Test for complete decomposition of the starting material by removing 1 mL of the solution, neutralizing with 10% sodium hydroxide solution, and adding 1 mL of chloroform. Shake the mixture. If the chloroform develops a bright yellow color, reaction is incomplete and stirring of the tin mixture should be continued until the test yields a colorless chloroform layer. Neutralize the reaction mixture by the slow addition of 10% sodium hydroxide solution. Allow the mixture to stand at room temperature for 1 hour, and then decant the supernatant liquid or remove the precipitate by filtration. Discard the solid as normal refuse; pack and label the liquid for disposal by burning.[7]

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6. Ald 2464D.
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7,12-DIMETHYLBENZ[a]ANTHRACENE $C_{20}H_{16}$

TOXIC, ANIMAL CARCINOGEN

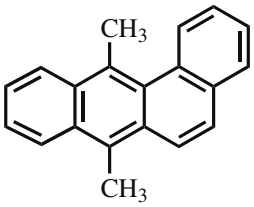
Physical Properties

Plates, leaflets from acetone-alcohol; faint greenish-yellow tinge; mp, 122–123°C.[1]

Chemical Properties

Freely soluble in benzene, moderately soluble in acetone, slightly soluble in alcohol; insoluble in water; may be solubilized in water by purines.[1]

Structure



Physiological Properties and Health Hazards

One of the most potent carcinogenic polycyclic aromatic hydrocarbons. In animals, large single and multiple doses produce tumors of the skin, breast, and stomach or leukemias, regardless of route of administration. It is a strong mutagen after metabolic activation.[2–4]

Spillage Disposal

Wear goggles and protective gloves and clothing. Turn off the ventilation and isolate the spill area. Add enough dimethylformamide to completely wet the contaminated surface, and then pour over the area an excess of a freshly prepared solution of 4.7 g of potassium permanganate in 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow to react for at least 1 hour. Absorb the residual solution on a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and, in the fume hood, slowly add it to a beaker of cold water. If necessary, neutralize the solution with soda ash. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix the material with a combustible solvent and burn in a chemical incinerator equipped with afterburner and scrubber.[7]

Small Quantities. Wear protective gloves, laboratory coat, and goggles. Dissolve the dimethylbenz[a]anthracene (5 mg) in acetone (2 mL). Add a freshly prepared solution of potassium permanganate (0.5 g) in 10 mL of 3 M sulfuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water). Swirl, and allow to stand at room temperature for at least 1 hour. If necessary, add more potassium permanganate solution to maintain the purple color. Cautiously neutralize with 10% aqueous sodium hydroxide, and then add, while stirring, a saturated solution of sodium bisulfite (approximately 1 g per 3.5 mL of water) until a colorless solution is formed. Wash into the drain with water.[5]

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DIMETHYLCARBAMYL CHLORIDE $(CH_3)_2NCOCl$

ANIMAL CARCINOGEN, CORROSIVE, LACHRYMATOR

Synonyms

Dimethylcarbamoyl chloride, (dimethylamino)carbonyl chloride.

Physical Properties

Colorless lachrymatory liquid; bp, 165–167°C.[1]

Chemical Properties

Reacts with water or steam to produce toxic and corrosive fumes.[1]

Physiological Properties and Health Hazards

Lachrymator; severe irritant. Skin exposure causes tumors in animals.[2,3] Very toxic by inhalation and if swallowed. Animal carcinogen; suspected human carcinogen. Exposure by all routes should be carefully controlled to the lowest possible levels.[4]

Spillage Disposal

Wear nitrile rubber gloves, safety glasses, laboratory coat, and self-contained breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to fume hood. Slowly add to a pail of cold water. When reaction has ceased, decant liquid to drain. Treat solid as normal refuse.[5]

Waste Disposal

Package Lots. Place in a an appropriate container labeled for disposal by burning. Dissolve in a combustible solvent and spray into a furnace equipped with afterburner and scrubber.[2]

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4. ACGIH 29.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.

DIMETHYLFORMAMIDE $(CH_3)_2NCHO$

IRRITANT

Physical Properties

Colorless to very slightly yellow liquid with faint amine-like odor; bp, 153°C.[1]

Chemical Properties

Miscible with water and most common organic solvents.[1]

Hazardous Reactions

Bromine. Strongly exothermic or explosive reaction to form hydroxymethylenedimethylammonium bromide.[2]

Carbon Tetrachloride and Iron. Potentially dangerous reaction in the presence of iron.[3]

Chromium Trioxide. Solid lumps of the trioxide ignite on addition to dimethylformamide.[4]

Magnesium Nitrate. Undergoes spontaneous decomposition in dimethylformamide.[5]

Sodium. Vigorous reaction on warming mixture.[6]

Sodium Borohydride. Solution may explode violently.[7]

Physiological Properties and Health Hazards

The vapor from the hot liquid irritates the eyes and respiratory system. The liquid irritates the skin and eyes. Swallowing would result in poisoning. Prolonged inhalation of vapor has resulted in liver damage in experimental animals. Avoid breathing vapor. Avoid contact with skin and eyes.[8] TLV-TWA 10 ppm (30 mg/m^3) (skin).[9]

Spillage Disposal

Wear eye protection, laboratory coat, and butyl rubber gloves.[10] Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Slowly add the mixture to 10% sodium hydroxide solution, allowing at least 10 mL of sodium hydroxide for each 1 mL of dimethylformamide. Allow the solution to stand for 48 hours. Decant the liquid to the drain. Treat the solid as normal refuse.[11]

Waste Disposal

Package Lots. Place in nonhalogenated solvent disposal container for recycling or disposal by burning in a furnace equipped with afterburner and scrubber.

Small Quantities. Wear eye protection, butyl rubber gloves,[10] and laboratory coat. Work in the fume hood. For each 1 mL of dimethylformamide, add 10 mL of 10% sodium hydroxide solution. Heat the mixture under reflux for 30 minutes, or allow to stand at room temperature for 48 hours. Wash the liquid into the drain.[10]

Reactions for Spillage and Waste Disposal

$$HCON(CH_3)_2 + NaOH \rightarrow (CH_3)_2NH + HCOO^-Na^+$$

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12. Ald 1374A.

1,1-DIMETHYLHYDRAZINE $(CH_3)_2NNH_2$

ANIMAL CARCINOGEN, CORROSIVE

Physical Properties

Hygroscopic, mobile liquid. Fumes in air and gradually turns yellow; characteristic ammoniacal odor of aliphatic hydrazines; bp, 63.9°C.[1]

Fire Hazard

Flammable.[1] Flash point, –15°C.

Chemical Properties

Miscible with water with much evolution of heat; also miscible with alcohol, ether, dimethylformamide, and hydrocarbons.[1]

Hazardous Reactions

Oxidants. Ignites violently on contact with nitric acid and hydrogen peroxide, among other oxidants.[2]

Physiological Properties and Health Hazards

Highly corrosive and irritating to skin, eyes, and mucous membranes; toxic also to central nervous system, liver, blood, and kidneys.[3] Convulsant poison.[1] An animal carcinogen; angiosarcomas and tumors in the lungs, kidneys, and livers of mice have been observed.[3] Reasonably anticipated to be a human carcinogen.[4] TLV-TWA 0.01 ppm (skin).[5]

Spillage Disposal:

Wear butyl rubber gloves,[6] laboratory coat, eye protection, and self-contained breathing apparatus. Impervious clothing recommended. Eliminate all sources of ignition and flammables.

On skin or clothing. Wash skin immediately. Remove contaminated clothing at once.

Spills. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container, transport to the fume hood, and slowly add to water, allowing 20 mL of water for each 1 g of dimethylhydrazine. Filter off the clay and sand. For each 1 g of dimethylhydrazine, place 32 mL (approximately 25% excess) of commercial laundry bleach (containing approximately 5% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous dimethylhydrazine dropwise to the stirred hypochlorite solution, monitoring the rate of addition by rise in temperature. The temperature is maintained at 45–50°C and addition takes about 1 hour. Stirring is continued for 2 hours until the temperature gradually falls to room temperature. Filter, and then pour the cooled filtrate down the drain with 50 times its volume of water. Dispose of the solid as normal refuse.[7,8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a large volume of combustible solvent and spray into a furnace equipped with afterburner and scrubber.[9]

Small Quantities. Wear butyl rubber gloves,[6] laboratory coat, and eye protection. Work in the fume hood. Prepare a dilute (5%) aqueous solution of 1,1-dimethylhydrazine by adding slowly to the appropriate volume of water. For each 1 g of dimethylhydrazine, place 32 mL (approximately 25% excess) of commercial laundry bleach (containing approximately 5% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous dimethylhydrazine dropwise to the stirred hypochlorite solution, monitoring the rate of addition by rise in temperature. The temperature is maintained at 45–50°C and addition takes about 1 hour. Continue stirring for 2 hours until the temperature gradually falls to room temperature. The cooled reaction mixture can be flushed down the drain.[8]

Reactions for Spillage and Waste Disposal

$$(CH_3)_2NNH_2 + NaOCl \rightarrow CH_3CH_3 + N_2 + NaCl + H_2O$$

REFERENCES

1. Merck 3273.
2. B 1165.
3. Cas 54, 3.
4. NIEHS III–120.
5. ACGIH 29.
6. LSS.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
8. PPL 161.
9. Ald 1384D.

DIMETHYL PHTHALATE $C_6H_4(COOCH_3)_2$

HARMFUL VAPOR

Physical Properties

Colorless, odorless, viscous liquid; mp, 5.5°C; bp, 283.7°C.[1]

Fire Hazard

Flash point, 146°C.[1]

Chemical Properties

Insoluble in water. Miscible with organic solvents.[1]

Physiological Properties and Health Hazards

Breathing vapor may cause coughing and paralysis. Liquid irritates the skin. Swallowing causes internal irritation, vomiting, diarrhea, and coma.[2] Eye irritant. TLV-TWA 5 mg/m^3.[3]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Add mixture to a beaker containing a solution of potassium hydroxide (3 g of KOH for each 5 g of dimethyl phthalate) in 1:1 mixture of methanol and water (25 mL for each 5 g of ester). Warm the mixture to about 60°C on a steam bath and keep at this temperature for 20 minutes. Swirl frequently. Cool and decant the solution into the drain. Dispose of the solid as normal refuse.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace equipped with afterburner and scrubber.[5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, add the ester to a beaker containing a solution of potassium hydroxide (3 g of KOH for each 5 g of dimethyl phthalate) in 1:1 mixture of methanol and water (25 mL for each 5 g of ester). Warm the mixture to about 60°C on a steam bath and keep at this temperature for 20 minutes, while swirling frequently. Wash the solution into the drain.[4]

Reactions for Spillage and Waste Disposal

$$C_6H_4(COOCH_3)_2 + 2KOH \xrightarrow{CH_3OH/H_2O} C_6H_4(COOK)_2 + 2CH_3OH$$

REFERENCES

1. Merck 3281.
2. ITI 242.
3. ACGIH 29.

4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.
5. Ald 1408C.

DIMETHYL SULFATE $(CH_3)_2SO_4$

HARMFUL VAPOR, CAUSES BURNS, SEVERE IRRITANT, ANIMAL CARCINOGEN

Synonyms

Methyl sulfate, DMS, sulfuric acid dimethyl ester.

Physical Properties

Colorless oily liquid; bp, 188°C with decomposition.[1]

Fire Hazard

Flash point, 83°C; auto-ignition temperature, 188°C. Extinguish with water, dry chemical, foam, or carbon dioxide.[2]

Chemical Properties

Somewhat soluble in water; hydrolysis is rapid at or above 18°C. Soluble in ether, dioxane, acetone, and aromatic hydrocarbons; sparingly soluble in carbon disulfide and aliphatic hydrocarbons.[1]

Hazardous Reactions

Ammonia. Violent reaction with concentrated aqueous ammonia.[3,4]

Tertiary Bases. In absence of solvent, reaction may be explosive.[5]

Physiological Properties and Health Hazards

Vapor, which has no warning characteristics, causes severe irritation of respiratory system, with possible severe lung injury after a latent period. Vapor and liquid irritate or burn the eyes severely, resulting in temporary or permanent dimming of vision. The vapor or liquid may blister the skin, and skin absorption may result in severe poisoning. Extremely poisonous and irritating if taken by mouth. Prevent inhalation of vapor. Prevent contact with skin and eyes.[6] This substance has been listed as a carcinogen by the U.S. Environmental Protection Agency.[1] Reasonably anticipated to be a human carcinogen.[7] TLV-TWA 0.1 ppm (0.52 mg/m^3) (skin).[8]

Spillage Disposal

Instruct others to keep a safe distance. Wear nitrile rubber gloves, laboratory coat, eye protection, and breathing apparatus if required. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Slowly add the mixture to a stirred 10% solution of sodium hydroxide (10 mL for each 1 mL of dimethyl sulfate). Let stand for 24 hours, with continuous stirring, if possible. Decant the liquid into the drain. Discard the solid with normal refuse.[9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[10]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. With brisk stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to

about 55°C. Heat the solution to gentle reflux and add the dimethyl sulfate (12.6 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux with stirring (to prevent bumping) for another 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[11]

Reactions for Spillage and Waste Disposal

$$(CH_3)_2SO_4 + 2KOH \rightarrow \underset{\text{ethanol}}{2CH_3OH} + K_2SO_4$$

REFERENCES

1. Merck 3282.
2. NFPA 49.
3. Lindlar, H., *Angew. Chem.,* 75, 297, 1963.
4. Claesson, P. et al., *Chemische Berichte,* 13, 1700, 1880.
5. B 306.
6. Lux 367.
7. NIEHS III–121.
8. ACGIH 29.
9. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories,* Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 280.
10. ITI 243; Aldrich 588.
11. PP 61.

1,4-DINITROSOPIPERAZINE $C_4N_4O_2H_8$

ANIMAL CARCINOGEN

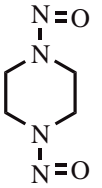
Physical Properties

Liquid; bp, 155–156°C.[1]

Chemical Properties

Slightly soluble in water.[1]

Structure



Physiological Properties and Health Hazards

N-nitrosamines have been shown to produce tumors in 39 animal species, including monkeys, birds, fish, and amphibia.[2] Although there is no direct evidence, for all practical purposes, they possess a carcinogenic risk to humans.[3]

Spillage Disposal

Solutions. Wear protective gloves (some nitrosamine solutions will diffuse through many types of gloves; if these solutions come into contact with gloves, remove gloves immediately, wash hands thoroughly, and put on new gloves), laboratory coat, goggles, and breathing apparatus if necessary. Isolate the area and absorb the spill on paper towels. Place the paper towels in a beaker in the fume hood. Immediately return to the spill area and decontaminate by pouring over it 100 mL of a 3 M sulfuric acid solution (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate. Allow to react overnight, and then cover with soda ash. Scoop the slurry into a pail of cold water and wash into the drain with a large volume of water. Wash the decontaminated area with soap and water. To the beaker in the fume hood containing the paper towels, add acid permanganate and leave overnight. Dilute the mixture with water, and then neutralize the solution by cautiously adding 10% sodium hydroxide solution. Filter the mixture and discard the solids as normal refuse. Wash the filtrate into drain with water.[4]

Solid. Pour a solution of 0.3 M potassium permanganate in 3 M sulfuric acid over the area of the spill. Leave overnight. Absorb the liquid by covering with a 1:1:1 mixture by weight of ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and, in the fume hood, slowly add it to a pail of cold water. If necessary, neutralize the solution with soda ash. Decant or filter the mixture into the drain with a large volume of water. Discard the solid residue with normal refuse.[4,5]

Waste Disposal

Wear protective gloves, laboratory coat, and goggles. Perform all operations in the fume hood. Dissolve the dinitrosopiperazine (300 µg) in 50 mL of 3 M sulfuric acid (8.5 mL of concentrated sulfuric acid slowly added to 41.5 mL of cold water). Add potassium permanganate (2.4 g), swirl until dissolved, and allow to stand at room temperature for at least 8 hours. If necessary, add additional potassium permanganate to maintain the purple color of the solution. Neutralize the solution by cautiously adding 10% aqueous sodium hydroxide solution, and then add, while stirring, a saturated solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless solution is formed. Wash into the drain with water.[4,5]

REFERENCES

1. Lijinsky, W. and Taylor, H.W., *Cancer Res.,* 85, 1270, 1975.
2. Bogooski, P. and Bogooski, S., *Int. J. Cancer,* 27, 471, 1981.
3. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamines,* No. 43, IARC Scientific Publications, Lyon, 1982, p.4.
4. *ibid.*, p. 25.
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DIOXANE $C_4H_8O_2$

HIGHLY FLAMMABLE, MAY FORM EXPLOSIVE PEROXIDES, HARMFUL VAPOR, ANIMAL CARCINOGEN

Synonyms

Diethylene dioxide, diethylene oxide, dioxan.

Physical Properties

Colorless liquid; faint pleasant odor; bp, 101°C.[1]

Fire Hazard

Flash point, 12°C; flammable limits, 2–22%; ignition temperature, 180°C. Extinguish fire with water spray, dry chemical, carbon dioxide, or vaporizing liquid.[2]

Chemical Properties

Soluble in water and the usual organic solvents. On exposure to light and air, liable to form explosive peroxides that should be decomposed before the ether is distilled to small volume.[1,3]

Hazardous Reactions

Air. Like all other ethers, dioxane forms explosive peroxides on exposure to air and these may be hazardous if the dioxane is distilled. Since dioxane is miscible with water, peroxides should be removed by passing the liquid through a column of activated alumina. The alumina should be washed with water or methanol before being discarded.[4]

Nickel. Dioxane reacts almost explosively with Raney nickel above 210°C.[5]

Sulfur Trioxide. The addition complex with sulfur trioxide decomposes violently on storage.[6]

Physiological Properties and Health Hazards

Vapor irritates mucous membranes and may lead to headache, drowsiness, and central nervous system depression. High concentration of vapor or swallowing liquid may cause nausea and vomiting, and injury to the kidney and liver.[3] Animal carcinogen.[1] Reasonably anticipated to be a human carcinogen.[7] Avoid breathing vapor.[3] TLV-TWA 20 ppm (skin).[8]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to keep a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves.[9] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to an appropriate container labeled for disposal by burning. Ventilate spill site well to evaporate remaining liquid and dispel vapor.[3,10,11]

Waste Disposal

Containers of dioxane that have been opened and are more than 1 year old may contain hazardous quantities of peroxides. Especially if they have screw caps, these containers should not be opened, but should be disposed of by the appropriate authorities. When the container can be opened safely, check for peroxides as follows: Wear butyl rubber gloves, laboratory coat, and eye protection. Dissolve 100 mg of potassium iodide in 1 mL of glacial acetic acid. Add to 1 mL of the dioxane. A pale yellow color indicates a low concentration (0.001–0.005%) and a bright

yellow or brown color indicates a high concentration (0.01% and hazardous) of peroxide in the sample.[12]

To remove peroxides, wear butyl rubber gloves, laboratory coat, and eye protection. Pour the dioxane (100 mL) into a separatory funnel and shake with a freshly prepared 50% aqueous solution of sodium metabisulfite (20 mL) for 3 minutes. Release the pressure in the funnel at 10-second intervals. Separate the aqueous layer. Retest the dioxane for the continued presence of small amounts of dialkyl peroxides that are not reduced by the metabisulfite treatment. If peroxides are absent, the dioxane can be dried for reuse or packaged for disposal by burning. If peroxides are still present, in the fume hood, place the ether in a 250-mL round-bottom flask equipped with a condenser, and add a solution of 100 mg of potassium iodide in 5 mL of glacial acetic acid and 1 drop of concentrated hydrochloric acid. Reflux gently for 1 hour.[9] Package the ether for disposal by burning.[13]

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5. Mozingo, R., *Org. Synth., Coll.* Vol. 3, 182, 1955.
6. Sisler, H.H. et al., *Inorg. Synth.*, 2, 174, 1947.
7. NIEHS III–122.
8. ACGIH 29.
9. LSS.
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11. ITI 251.
12. Jackson, H.L. et al., *J. Chem. Educ.,* 3, 114, 1974.
13. Ald 1446D.

DIPHENYLAMINE $(C_6H_5)_2NH$

TOXIC IRRITANT

Synonyms

N-Phenylbenzene amine,[1] phenylaniline, anilinobenzene.[2]

Physical Properties

Crystals, floral odor; mp, 53–54°C; bp, 302°C. Discolors in light.[1]

Fire Hazard

Flash point, 153°C (closed cup); auto-ignition temperature, 634–635°C. Extinguish fires with water, carbon dioxide, or dry chemical powder, or alcohol or polymer foam.[3]

Chemical Properties

One gram dissolves in 2.2 mL of ethanol and 4.5 mL of propanol; freely soluble in benzene, ether, glacial acetic acid, and carbon disulfide. Insoluble in water. Forms salts with strong acids.[1]

Hazardous Reactions

Diphenylamine may react violently with strong acids or with strong oxidizing agents.

Physiological Properties and Health Hazards

Irritant to mucous membranes, eyes, and skin.[2–5] TLV-TWA 10 mg/m^3.[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and goggles. Cover the spill with a 1:1:1 mixture of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all of the liquid has been absorbed, scoop into a plastic pail and place in the fume hood. Add sufficient water to dissolve the sodium carbonate, and then add 3 M sulfuric acid (prepared by adding 17 mL of concentrated sulfuric acid to 83 mL of water) until the pH of the solution is about 1. Estimate the volume of diphenylamine that was spilled and add 10 g of potassium permanganate for each 1 mL of diphenylamine. Stir the mixture vigorously, and then leave overnight. Add solid sodium bisulfite while stirring until the solution is colorless. Neutralize with 5% aqueous sodium hydroxide solution. Allow the solids to settle and pour the liquid into the drain. Dispose of the solids (sand and calcium bentonite) as normal refuse.[4,5,7,8]

Waste Disposal

Package Lots. Label for recycling or disposal by burning.[4,5]

Small Quantities. Quantities of reagents are given for 1 g of diphenylamine. Dissolve the diphenylamine in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid added to 83 mL of water). While stirring, add 10 g of potassium permanganate in small portions to avoid foaming. Stir the mixture overnight. Add solid sodium bisulfite until the solution is colorless. Neutralize with 5% aqueous sodium hydroxide solution and pour the liquid into the drain. Discard any small quantity of brown solid (manganese dioxide) as normal refuse.[9]

REFERENCES

1. Merck 3349.
2. Sax 1457.
3. Lux 376.
4. Ald 1452A.
5. ITI 254.
6. ACGIH 30.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988.
8. *Haz. Mat. Spills Tec.,* 29.6.
9. PPL 162.

DIQUAT $C_{12}H_{12}N_2Br_2$

HERBICIDE

Synonyms

6,7-Dihydrodipyrido[1,2-a:2',1'-c]pyrazinedium dibromide, 1,1'-ethylene-2,2'-dipyridylium dibromide, FB/2, aquacide, Reglone, deiquat.

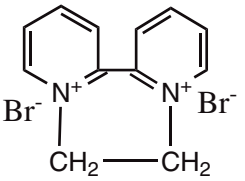
Physical Properties

Yellow crystalline monohydrate, mp, 335–340°C.[1]

Chemical Properties

Soluble in water; insoluble in organic solvents, slightly soluble in alcohol. Stable in acid or neutral solution.[1]

Structure



Physiological Properties and Health Hazards

LD_{50} 231 mg/kg (oral, rat) 231 mg/kg; 101 mg/kg (oral, rabbit).[1,2] LC_{50} (rainbow trout, 96 hours) 11.2 mg/L.[3] TLV-TWA 0.5 mg/m^3 (inhalable particulate mass); TLV-TWA 0.1 mg/m^3 (respirable particulate mass).[4] Minimal absorption from gastrointestinal tract although distension and irritation occur after large doses in experimental animals.[1,2] Absorbed through human skin only after prolonged exposure; shorter exposure can cause irritation and a delay in the healing of cuts and wounds. May cause temporary damage to nails and nose bleeding if inhaled.[2] Symptoms of poisoning include diarrhea and vomiting.

Formulations

Soluble concentrate; gel.

Spillage Disposal

Wear gloves and eye protection. The crystals can be swept up and discarded as for waste disposal. For spills of Diquat solutions, cover with a 1:1:1 mixture of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and add to a pail of water. Estimate the weight of Diquat in the spilled solution, and for each 0.1 g, add 2 g of potassium permanganate. Stir until dissolved and allow to stand at room temperature overnight. Reduce the excess permanganate with solid sodium bisulfite, and then decant the liquid to the drain with water. The residue can be treated as normal refuse.[5]

Waste Disposal

Wear gloves and eye protection. Destroy Diquat (0.1 g) by stirring at room temperature for 48 hours with a solution of 2 g of potassium permanganate in 50 mL of 10% sodium hydroxide solution. At the end of the reaction time, neutralize the solution with dilute hydrochloric acid (concentrated acid added to five times its volume of water) and the excess potassium permanganate and manganese dioxide reduced with sodium bisulfite. If a white precipitate forms, dissolve it by adding water. Pour the clear solution into the drain. Discard any undissolved brown precipitate of manganese dioxide with regular refuse.[5]

REFERENCES

1. Merck 3370.
2. Agro. Handbook, A0121.
3. Agro. Desk Ref., 180.
4. ACGIH 30.
5. Armour, M.A., Decontamination of spills and residues of pesticides and protective clothing worn during their handling, *Proceedings of the Pacific Basin Conference on Hazardous Waste Research*, Kuala Lumpur, Malaysia, 1996, p. 44.

DOXORUBICIN $C_{27}H_{29}NO_{11}$

ANTINEOPLASTIC

Synonyms

14-Hydroxydaunomycin, Adriamycin, NSC-123127, FI-106, Adriablastina.

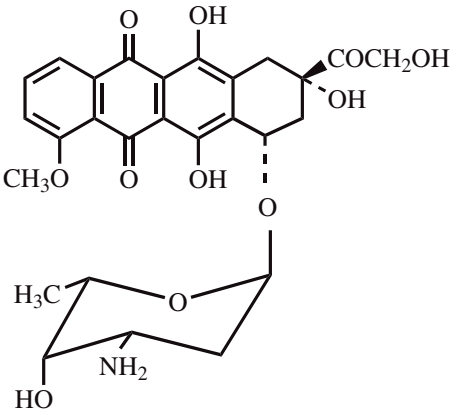
Physical Properties

Orange-red thin needles; mp, 204–205°C (dec).[1]

Chemical Properties

Soluble in water and aqueous alcohols. Practically insoluble in acetone, benzene, and other common organic solvents. Aqueous solutions are yellow-orange at acid pH, orange-red at neutral pH, and violet-blue at pH greater than 9.[1]

Structure



Mode of Action

Doxorubicin intercalates with DNA, causing an inhibition of RNA synthesis.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, bone marrow depression, local phlebitis, alopecia, stomatitis, and cardiac toxicity dependent on cumulative dosage.[2] LD_{50} (i.v., mouse) 21.1 mg/kg.[1] Reasonably anticipated to be a human carcinogen.[3]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Estimate the weight of doxorubicin in the spilled liquid. Pour over the spill 3 M sulfuric acid solution (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate, allowing 100 mL of solution per 50 mg of doxorubicin. Allow to react for at least 2 hours. Decolorize with a saturated solution of sodium bisulfite. Absorb the liquid on a 1:1:1 mixture of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a pail of cold water. Decant the liquid into the drain with water and discard the solid as normal refuse.[4–6]

Waste Disposal

Wear goggles and protective gloves and clothing. Dissolve doxorubicin (50 mg) in 50 mL of 3 M sulfuric acid (8.5 mL of concentrated sulfuric acid slowly added to 41.5 mL of cold water), and add 1 g of potassium permanganate while stirring. Continue stirring for 2 hours, neutralize by careful addition of soda ash, and then add, while stirring, a saturated solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless solution is formed. Wash into the drain with water.[4,5]

REFERENCES

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2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents, Handbook of Clinical Data,* 2nd ed., G.K. Hall Medical Publishers, Boston, 1982, pp. 110–111.
3. NIEHS III–70.
4. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 25.
5. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education,* Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 103.
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ETHIDIUM BROMIDE $C_{21}H_{20}BrN_3$

MUTAGEN

Synonyms

Dromilac.

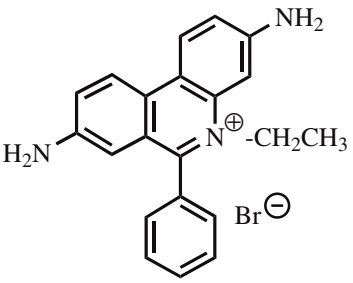
Physical Properties

Bitter tasting, dark red crystals from alcohol; mp, 238–240°C.[1]

Chemical Properties

Soluble in 20 parts water and 750 parts chloroform at 20°C.[1]

Structure



Physiological Properties and Health Hazards

Ethidium bromide is a mutagen which intercalates double-stranded DNA[2] and RNA.[3]

Waste Disposal

Package Lots. Place the compound in a separate labeled container for disposal by burning. Dissolve or mix the material with a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[4]

Small Quantities. Ethidium bromide is normally used in very dilute aqueous solutions and under these conditions, it can be converted to the physiologically inactive product, 2-carboxybenzophenone, with chlorine bleach. Wear goggles and protective gloves and clothing. Work in the fume hood. To a solution of 34 mg of ethidium bromide in 100 mL of water, add 300 mL of household chlorine bleach, and stir the mixture at room temperature for 2 hours. Wash the solution into the drain with water.[5]

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ETHIONINE (DL) $C_6H_{13}NO_2S$

SUSPECTED CANCER AGENT

Synonyms

2-Amino-4-(ethylmercapto)-butyric acid, S-ethyl-L-homocysteine, homocysteine-S-ethyl ester.

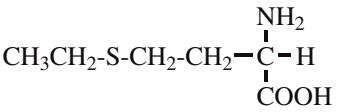
Physical Properties

White crystalline solid; mp (DL form), 257–260°C (dec); (L form) 272–274°C (dec)[1]; sublimes at 196–216°C.[2]

Chemical Properties

Very soluble in water and ethanol; insoluble in nonpolar solvents.[1]

Structure



Physiological Properties and Health Hazards

Produces the following symptoms when methionine is not in the diet: impairment of liver function, proteinuria, hematuria, dermatitis, leucopenia, thrombocytopenia, and diarrhea. Deterioration of mental status may also occur. All symptoms are reversed or prevented by methionine.[3]

Spillage Disposal

To reduce the chance of inhalation of dust, cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Wear protective gloves, goggles, laboratory coat, and a dust mask. Scoop the mixture into a plastic container, label, and send for disposal by burning.[4,5]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix the material with a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[4]

Small Quantities. Wear PVC gloves, laboratory coat, and goggles. Dissolve ethionine (1 g) in 50 mL of water in a 250-mL Erlenmeyer flask. While stirring, slowly add household bleach (60 mL) and continue stirring for 2 hours. Wash the solution into the drain with water.[6]

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6. Armour, M.A. and Renecker, D.M., *Laboratory Disposal Method for Ethionine*, University of Alberta, Edmonton, 1999.

2-ETHOXYETHANOL $CH_3CH_2OCH_2CH_2OH$

TERATOGEN, IRRITANT, FLAMMABLE

Synonyms

Ethylene glycol monoethyl ether, cellosolve, oxitol.[1]

Physical Properties

Colorless, practically odorless liquid; bp, 135°C, mp, –70°C.[1]

Fire Hazard

Flash point, closed cup, 44°C; flash point, open cup, 49°C.[1] Explosive limit, 2.8–18.0%. Autoignition temperature, 240°C. Extinguish fire with water, foam, dry chemical, or carbon dioxide.[2]

Chemical Properties

Miscible with water, alcohol, ether, acetone, and liquid esters. It dissolves many oils, resins, waxes, etc.[1]

Hazardous Reactions

Air and Light. Liable to form explosive peroxides on exposure to light and air that should be decomposed before the ether is distilled to small volume.[2,3] Containers that have been opened and stored may contain peroxides. These should be handled with great care.

Bases, Oxidizing Agents, and Acids. May react violently with bases, acids, and oxidizing agents.[4]

Copper and Alloys. May react violently with copper or its alloys.[4]

Physiological Properties and Health Hazards

Harmful if inhaled, swallowed, or absorbed through skin. May cause irritation. Prolonged exposure can cause narcotic effect. Chronic effects include damage to liver, kidneys, and blood.[2] May cause reproductive disorders.[4] TLV-TWA 5 ppm, 18 mg/m^3.[5]

Spillage Disposal

Shut off all sources of ignition. Evacuate the area. Wear self-contained breathing apparatus, nitrile rubber gloves, and laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all the liquid has been absorbed, scoop into an appropriate container and package for disposal by burning.[4,6,7] Ventilate the spill site to evaporate remaining liquid and dispel vapor.

Waste Disposal

Package and label for disposal by burning.[4]

REFERENCES

1. Merck 3786.
2. Lux 393.
3. B 490, 1206.
4. Ald 1555D.
5. ACGIH 31.

6. *Haz. Mat. Spills Tec.,* 29.5.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.

ETHYL AZIDOFORMATE $N_3COOCH_2CH_3$

EXPLOSIVE

Physical Properties

Liquid; bp, 114°C.[1]

Fire Hazard

It is liable to explode if boiled at atmospheric pressure (at 114°C).[1]

Spillage Disposal

Wear leather gloves, goggles, heavy face shield, and laboratory coat. Work from behind body shield. Avoid unnecessary heat, friction, or impact. Mop up with wet paper towels. In the fume hood and behind a shield, place paper towels in a beaker containing 6 M hydrochloric acid (prepared by adding concentrated acid to an equal volume of cold water; about 150 mL for each 1 g of azide). Follow waste disposal procedure.[2]

Waste Disposal

Keep stock of all azides very low. Stamp date and receipt on package. Wear butyl rubber gloves, eye protection, and laboratory coat. In the fume hood and behind a safety shield, add the azide to 6 M hydrochloric acid (cautiously add a volume of concentrated acid to an equal volume of cold water; 150 mL for each 1 g of azide). While stirring, add granular tin (10 g for each 1 g of azide). Continue stirring at room temperature for 30 minutes. Decant or filter the solution into a pail of cold water. Neutralize with sodium carbonate and pour into the drain with at least 50 times its volume of water. Reuse the excess tin or discard as normal refuse.[2]

Reactions for Spillage and Waste Disposal

$$N_3COOCH_2CH_3 + Sn + 2HCl \rightarrow N_2 + \underset{\text{ethyl aminoformate}}{H_2NCOOCH_2CH_3} + SnCl_2$$

REFERENCES

1. Forster, M.O. et al., *J. Chem. Soc.,* 93, 81, 1908.
2. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 100.

ETHYL DIAZOACETATE $CH_3CH_2OCOCHN_2$

EXPLOSIVE, SUSPECTED CANCER AGENT

Synonyms

Diazoacetic ester, ethyl diazoethanoate.

Physical Properties

Yellow oil, pungent odor, very volatile; bp_5, 42°C; bp_{720}, 140–141°C.[1] Explosive; distillation, even under reduced pressure, is dangerous.

Fire Hazard

Flammable[2]; explosive.[1]

Chemical Properties

Slightly soluble in water. Volatile with steam, ether, and benzene vapors. Miscible with alcohol, benzene, ligroin, and ether.[1]

Hazardous Reactions

Explosive when heated.[2] Distillation may be dangerous, even under reduced pressure.[1,3]

Sulfuric Acid. Explodes on contact with concentrated sulfuric acid.[3]

Physiological Properties and Health Hazards

Experimental carcinogen.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix with flammable solvent and burn in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood behind a shield, cautiously add to dilute ceric ammonium nitrate solution with cooling. Solutions may be discarded upon completion of the reaction. Mixtures should be separated and the organic portion placed in a separate labeled container for disposal by burning.[3]

REFERENCES

1. Merck 3021.
2. Aldrich 765; Ald 1596C.
3. Searle, N.E., *Org. Synth.,* Coll. Vol. 4, 426, 1962.
4. Lux 391.

ETHYLENEDIAMINE $H_2NCH_2CH_2NH_2$

FLAMMABLE LIQUID, CORROSIVE

Synonyms

1,2-Ethanediamine, 1,2-diaminoethane.[1]

Physical Properties

Colorless, clear, viscous liquid; ammonia odor; bp, 116–117°C; mp, 8.5°C. Volatile with steam.[1]

Fire Hazard

Flash point, closed cup, 43°C.[1] Extinguish fire with carbon dioxide, dry chemical, alcohol, or polymer foam.[2,3]

Chemical Properties

Freely soluble in water forming a hydrate, also in alcohol; slightly soluble in ether; soluble in benzene.[1]

Hazardous Reactions

Ethylene may react vigorously with acids, acid chlorides, oxidizing agents, acid anhydrides, phosphorous halides, and perchlorates.[2]

Physiological Properties and Health Hazards

An irritant and allergen. It can cause sensitization leading to asthma. Harmful if swallowed, inhaled, or absorbed through the skin. Exposure can cause damage to the kidneys and liver, stomach pain, vomiting, and diarrhea.[2,4] TLV-TWA 10 ppm (25 mg/m^3).[5]

Spillage Disposal

Shut off all sources of ignition. Evacuate the area. Wear self-contained breathing apparatus, nitrile rubber gloves, and laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all the liquid has been absorbed, scoop the mixture into a plastic pail and place in the fume hood. Add sufficient water to dissolve the sodium carbonate, and then add 3 M sulfuric acid (prepared by adding 17 mL of concentrated sulfuric acid to 83 mL of water) until the pH of the solution is about 1. Estimate the volume of ethylenediamine that was spilled and add 10 g of potassium permanganate for each 1 mL of ethylenediamine. Stir the mixture vigorously, and then leave overnight. Add solid sodium bisulfite while stirring until the solution is colorless. Neutralize with 5% aqueous sodium hydroxide solution. Allow the solids to settle and pour the liquid into the drain. Dispose of the solids (sand and calcium bentonite) as normal refuse.[2,4,6]

Waste Disposal

Package Lots. Label for recycling or disposal by burning.[2,4]

Small Quantities. Quantities of reagents are given for 1 mL of ethylenediamine. Dissolve the ethylenediamine in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid added to 83 mL of water). While stirring, add 10 g of potassium permanganate in small portions to avoid foaming. Stir the mixture overnight. Add solid sodium bisulfite until the solution is colorless. Neutralize with 5% aqueous sodium hydroxide solution and wash the liquid into the drain. Discard any small quantity of brown solid (manganese dioxide) as normal refuse.[6]

REFERENCES

1. Merck 3829.
2. Ald 1606D.
3. Sax 5 658.
4. ITI 282.
5. ACGIH 32.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.

ETHYLENIMINE C_2H_5N

TOXIC, FLAMMABLE

Synonyms

Aziridine, dimethylenimine.

Physical Properties

Colorless liquid; odor of ammonia; strongly alkaline[1]; freezing point, −78°C[2]; bp, 56–57°C.[1]

Fire Hazard

Flash point, −13°C; flammable even at room temperature.[3] Dangerous when exposed to heat, flame, or oxidizers.[4]

Chemical Properties

Polymerizes easily. Miscible with water, soluble in alcohol. Can be stored for some time over a few NaOH pellets.[1]

Structure

Hazardous Reactions

Reacts violently with many chemicals, including acetic acid, acetic anhydride, acrolein, acrylic acid, allyl chloride, carbon disulfide, chloride glyoxal, and vinyl acetate.[4]

Acids. Polymerizes violently.[5]

Silver. May form explosive silver derivatives in contact with silver or its alloys, including silver solder.[5]

Sodium Hypochlorite. Forms explosive 1-chloroaziridine on treatment with sodium hypochlorite.[5]

Physiological Properties and Health Hazards

Exposure may result in nausea, vomiting, shortness of breath, cough, fever, and possibly death when inhaled or absorbed through the skin. Severe blistering agent. Corrosive to the skin, mucous membranes, and eye tissue, although effects may be delayed.[3,6] LD_{50} (oral, rat) below 5 mg/kg.[2] TLV-TWA 0.5 ppm.[7]

Spillage Disposal

Wear protective gloves, laboratory coat, goggles, and breathing apparatus if necessary. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and either pack and send for disposal by burning or, in the fume hood, add the mixture to a solution of 85% potassium hydroxide pellets in ethanol allowing 0.12 mol (7.9 g) of potassium hydroxide and 32 mL of ethanol for each 0.1 mol (4.3 g) of ethylenimine to

be destroyed. Heat the mixture under reflux for 8 hours. Decant the cooled solution into the drain with water. Treat the solid as normal refuse.[8,9]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal. Dissolve the compound in a flammable solvent, and burn in a furnace with afterburner and scrubber.[3]

Small Quantities. Wear protective gloves, laboratory coat, and goggles. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, and dropping funnel. Add 32 mL of 95% ethanol rapidly. Heat the solution to gentle reflux and add ethylenimine (4.3 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring for 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain with water.[9]

REFERENCES

1. Merck 3838.
2. Weiss, G., Ed., *Hazardous Chemicals Data Book*, Noyes Data Corporation, Park Ridge, NJ, 1980, p. 443.
3. ITI 291.
4. Sax 1610.
5. B 285.
6. IARC 9, 37.
7. ACGIH 32.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.
9. PP 61.

ETHYL METHANESULFONATE $CH_3SO_2OCH_2CH_3$

ANIMAL CARCINOGEN

Synonyms

Ethyl mesylate, methanesulfonic acid ethyl ester, NSC-26805.

Physical Properties

Colorless liquid[1]; bp, 213–213.5°C.[2]

Chemical Properties

Moderately soluble in water. Readily soluble in hot water.[3]

Hazardous Reactions

Hydrolyzed in water to the strongly corrosive methanesulfonic acid.[4]

Physiological Properties and Health Hazards

Animal carcinogen in rodents. Insufficient data available to evaluate carcinogenicity in humans.[5]

Spillage Disposal

Wear protective gloves, laboratory coat, goggles, and breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container and either label and send for disposal by burning,[4,6] or, in the fume hood, add the mixture to a solution of potassium hydroxide in ethanol allowing 0.12 mol (7.9 g) of 85% potassium hydroxide pellets and 32 mL of ethanol for each 0.1 mol (12.4 g) of ethyl methanesulfonate to be destroyed. Heat the mixture under reflux for 8 hours. Decant the cooled solution into the drain with water. Treat the solid as normal refuse.[6]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal. Dissolve or mix the material into a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[4]

Small Quantities. Wear protective clothing, gloves, and goggles. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, and dropping funnel. Add 32 mL of ethanol rapidly. Heat the solution to gentle reflux and add ethyl methanesulfonate (12.4 g, 0.1 mol) at such a rate to maintain gentle reflux. Heat under reflux while stirring for 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain with water.[7]

REFERENCES

1. IARC 7, 245.
2. Merck 3859.
3. Beilstein 4, 5.
4. Aldrich 786; Ald 1634B.
5. NIEHS III–130.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.
7. PP 61.

FENBUTATIN OXIDE $C_{60}H_{78}OSn_2$

ACARICIDE

Synonyms

Di[tri-(2-methyl)-2-phenylpropyl)tin] oxide, hexakis-(2-methyl-2-phenylpropyl)-distann-oxane, hexakis (β,β-dimethylphenethyl)-distannane, SD 14114, Torque, Vendex.

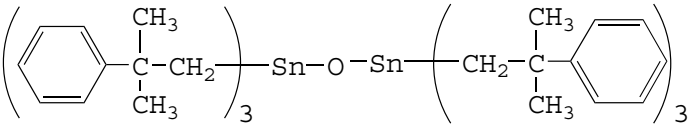
Physical Properties

White crystalline solid, mp, 144–147°C.[1]

Chemical Properties

Insoluble in water. Solubility in g/L at 23°C: acetone 6, benzene 140, and methylene chloride 380.[1]

Structure



Physiological Properties and Health Hazards

Corrosive. Skin and eye irritant.[1] LD_{50} (oral rat) 2631 mg/kg[2]; LC_{50} (rainbow trout, 48 hours) 0.27 mg/L.[3]

Waste Disposal

Wear eye protection and gloves. To each 1 g of Benomyl, add 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid added to 83 mL of water) and 4.7 g of potassium permanganate. Stir the mixture and leave at room temperature for 48 hours. A saturated solution of sodium bisulfite is added until the solution is colorless. The solution is neutralized by the careful addition of soda ash (foaming may occur) or 5% aqueous sodium hydroxide. The clear solution is washed into the drain with water.[4]

REFERENCES

1. Merck 3991.
2. Agro. Handbook, A0196.
3. Agro. Desk Ref., 214.
4. Armour, M.A. and Bricker, Y., *Laboratory Disposal Method for Fenbutatin Oxide*, University of Alberta, 1997.

FLUOROURACIL $C_4H_3FN_2O_2$

ANTINEOPLASTIC

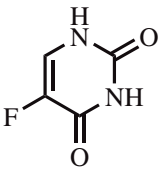
Synonyms

Adrucil, Arumel, Efudex, Fluril, Fluracil, Fluoroplex, Timazin, 5-fluorouracil, 5-FU, RO 2–9757, NSC 19893.

Chemical Properties

Crystals form from water or methanol-ether; dec, 282–283°C; sublimes (0.1 mm), 190–200°C.[1]

Structure



Mode of Action

5-Fluorouracil interferes with nucleic acid biosynthesis, thus inhibiting subsequent DNA synthesis and RNA synthesis.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include stomatitis, bone marrow depression, diarrhea, anorexia, nausea leukopenia, alopecia, and dermatitis.[2]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of fluorouracil in the spilled liquid and add 10% calcium hypochlorite solution (see waste disposal for preparation), allowing 100 mL for each 500 mg of fluorouracil. Allow to stand overnight, and then decant the liquid into the drain with water and discard the solid as normal refuse.[3,4]

Waste Disposal

Wear goggles and protective gloves and clothing. To each 10 mL of solution containing 500 mg of fluorouracil, add 40 mL of an aqueous solution of calcium hypochlorite (prepared by stirring 10 g of calcium hypochlorite in 100 mL of water for 2 hours, and then removing the undissolved material by filtration). Stir the solution at room temperature for 5 hours, remove the precipitated calcium fluoride by filtration, and discard as normal refuse. Wash the filtrate into the drain with water.[3]

REFERENCES

1. Merck 4208.
2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data,* 2nd ed., G.K. Hall Medical Publishers, Boston, 1982, pp. 116–117.

3. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 104.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.

FORMALDEHYDE SOLUTION HCHO

FLAMMABLE, SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING, CAUSES BURNS

Synonyms

Formalin.

Physical Properties

Colorless, sometimes milky solution with pungent odor; bp, 96°C.[1]

Fire Hazard

Flash point, about 60°C.[1]

Chemical Properties

Miscible with water; the solution generally contains 37–41% formaldehyde and 11–14% methanol.[2]

Hazardous Reactions

Hydrogen Peroxide. Mixtures of hydrogen peroxide and formaldehyde explode under initiation.[3]

Magnesium Carbonate-Hydroxide. Shaking of formaldehyde with basic magnesium carbonate to neutralize any formic acid present produces carbon dioxide, which may cause explosion of a sealed container.[4]

Nitromethane. In the presence of alkali, nitromethane and formaldehyde react to produce 2-nitroethanol and di- and tri-condensation products. A flash explosion may result if air is admitted to the vacuum distillation apparatus after removal of the 2-nitroethanol unless the residue is adequately cooled.[5]

Peroxyformic Acid. Violent reaction between formaldehyde and 90% peroxyformic acid.[6]

Phenol. A runaway reaction may be encountered during the preparation of phenol-formaldehyde resins.[4]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. The liquid irritates the eyes severely and hardens the skin. Swallowing results in stomach pain, nausea, vomiting, and finally loss of consciousness. Prolonged exposure to the vapor can result in laryngitis, bronchitis, or bronchial pneumonia. Prolonged skin contact causes skin ulceration. Avoid breathing vapor. Avoid contact with eyes and skin.[2] TLV-STEL-C 0.3 ppm (0.37 mg/m^3).[7]

Spillage Disposal

Shut off all possible sources of ignition. Wear face shield and goggles, laboratory coat, and butyl rubber gloves.[8] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Add the mixture to cold water (about 10 mL for each 1 mL of formaldehyde solution). Slowly add household bleach (25 mL of household bleach for each 1 mL of formaldehyde solution). Allow to stand at room temperature for 20 minutes. Decant liquid to drain. Discard solid residue as normal refuse.[9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[10]

Small Quantities. Wear butyl rubber gloves,[8] laboratory coat, and eye protection. In the fume hood, while stirring, slowly add diluted formaldehyde (about 10 mL water for each 1 mL of formaldehyde) to an excess of household bleach (25 mL household bleach for each 1 mL of formaldehyde). Stir for 20 minutes, and then wash the solution into the drain.[9]

Reactions for Spillage and Waste Disposal

$$HCHO + ClO^- \rightarrow \underset{\text{formic acid}}{HCOOH} + Cl^-$$

REFERENCES

1. Merck 4259.
2. Lux 404.
3. B 1212.
4. B 149.
5. Noland, W.E., *Org. Synth.,* 41, 69, 1961.
6. D'Ans, J. et al., *Chemische Berichte,* 48, 1136, 1915.
7. ACGIH 33.
8. LSS.
9. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.
10. ITI 310; Aldrich 1733C.

HEPTANE $CH_3(CH_2)_5CH_3$

FLAMMABLE LIQUID, IRRITANT

Physical Properties

Volatile, flammable liquid, bp, 98.4°C; mp, –90.7°C.[1]

Fire Hazard

Flash point, open cup, –1°C; flash point, closed cup, –4°C.[1] To fight fire, use foam, carbon dioxide, or dry chemical.[2] Auto-ignition temperature, 204°C. Explosive limits, 1.0–6.7%.[3]

Chemical Properties

Insoluble in water, soluble in alcohol, chloroform, and ether.[1]

Hazardous Reactions

Phosphorous and Chlorine. A violent reaction may occur.[2,4]

Physiological Properties and Health Hazards

Harmful if inhaled or swallowed. Material is irritating to the mucous membranes and upper respiratory tract. Causes eye irritation.[5] Narcotic in high concentrations; central nervous system irritant.[2] TLV-TWA 400 ppm (1640 mg/m^3); TLV-STEL 500 ppm.[6]

Spillage Disposal

Shut off all sources of ignition. Evacuate the area. Wear self-contained breathing apparatus, nitrile rubber gloves, and laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all the liquid has been absorbed, scoop into an appropriate container and package and label for disposal by burning.[5,7]

Waste Disposal

Distill for reuse or package and label for disposal by burning.[5,7]

Small Quantities. Place in an evaporating dish or beaker and allow to evaporate in the fume hood. Ensure that there are no flames or other ignition sources present.

REFERENCES

1. Merck 4679.
2. Sax 1830.
3. Lux 412.
4. NFPA 491M.
5. Ald 1821D.
6. ACGIH 34.
7. ITI 327.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988.
9. *Haz. Mat. Spills Tec.,* 29.5.

HEXACHLOROBUTADIENE $Cl_2C=CCl-CCl=CCl_2$

ANIMAL CARCINOGEN

Synonyms

Perchlorobutadiene.

Physical Properties

Liquid; mp, –21°C; bp, 215°C.[1]

Fire Hazard

Auto-ignition temperature, 610°C.[2]

Chemical Properties

Insoluble in water; soluble in alcohol and ether.[1]

Physiological Properties and Health Hazards

Toxicity high via oral and inhalation routes.[2] TLV-TWA 0.02 ppm (0.21 mg/m^3) (skin).[3]

Waste Disposal

Place in a separate container labeled for disposal by burning. Dissolve or mix with a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[4]

REFERENCES

1. CRC 3–88 3163.
2. Sax 1840.
3. ACGIH 35.
4. Ald 1832C.

HEXAMETHYLPHOSPHORAMIDE $[(CH_3)_2N]_3PO$

ANIMAL CARCINOGEN, TOXIC

Synonyms

Hexamethylphosphoric triamide, Hempa, HMPA, HMPT, ENT-50882.

Physical Properties

Clear, colorless, mobile liquid with spicy odor; mp, 7°C; bp, 235°C.[1]

Chemical Properties

Miscible with water.[2] Soluble in ethanol and ether.[3]

Physiological Properties and Health Hazards

Toxic. An experimental carcinogen by inhalation. Reasonably anticipated to be a human carcinogen.[4] LD_{50} (oral, male and female rat) 2650, 3360 mg/kg.[1] No TLV.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Small quantities of hexamethylphosphoramide can be hydrolyzed with concentrated hydrochloric acid under reflux for at least 5 hours. Add 215 mL of concentrated hydrochloric acid to a 500-mL, three-necked, round-bottom flask equipped with stirrer, heating mantle, water-cooled condenser, and dropping funnel. Over a period of about 30 minutes, add 50 mL (51.5 g, 0.3 mol) of the hexamethylphosphoramide through the dropping funnel while refluxing the reaction mixture. After addition is complete, reflux the mixture for at least 5 hours. Cool the contents of the flask, neutralize carefully with dilute sodium hydroxide or sodium carbonate, and wash the resultant mixture into the drain.[6]

Reactions for Spillage and Waste Disposal

$$[(CH_3)_2N]_3PO + 3HCl + 3H_2O \rightarrow \underset{\text{dimethylamine hydrochloride}}{3(CH_3)_2NH \cdot HCl} + \underset{\text{phosphoric acid}}{H_3PO_4}$$

REFERENCES

1. Merck 4568.
2. Lux 417; ACGIH 35.
3. CRC.
4. NIEHS III–139.
5. Ald 1853D.
6. PP 68.

HEXANE $CH_3(CH_2)_4CH_3$

FLAMMABLE LIQUID IRRITANT

Physical Properties

Colorless, very volatile liquid with faint, peculiar odor; bp, 69°C; mp, –95°C to –100°C.[1] Autoignition temperature, 225°C.[2,3]

Fire Hazard

Flash point, –22°C. Flammable limits: lower 1.1% by volume, upper 7.5% by volume.[2]

Chemical Properties

Insoluble in water; miscible with alcohol, chloroform, and ether.[1]

Hazardous Reactions

Dinitrogen Tetraoxide (Nitrogen Dioxide). An explosive reaction may occur.[4]

Physiological Properties and Health Hazards

The vapor may irritate the respiratory system and, in high concentrations, have narcotic action. Chronic exposure can lead to loss of sensation in hands and feet. Irritant to skin, eyes, and the gastrointestinal tract. Experimental teratogenic, reproductive effects.[3,5,6] TLV-TWA 50 ppm (176 mg/m^3).[7]

Spillage Disposal

Shut off all sources of ignition. Evacuate the area. Wear self-contained breathing apparatus, nitrile rubber gloves, and laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (bentonite), and sand. When all the liquid has been absorbed, scoop into a pail or other wide-mouth container and place in fume hood. If local regulations allow, stand in the fume hood for several days until all of the hexane has evaporated. Discard the solid with normal refuse. Otherwise, package and label the solid for disposal.[8,9]

Waste Disposal

Package Lots. Distill for reuse or package and label for disposal by burning.[5,10]

Small Quantities If local regulations permit, place in an evaporating dish or beaker in the fume hood and allow to evaporate. Ensure that there are no flames or other ignition source present. Otherwise, package and label for disposal by burning.[8]

REFERENCES

1. Merck 4712.
2. NFPA 325M.
3. Lux 417.
4. B 2377.
5. Ald 1855A.
6. Sax 1859.
7. ACGIH 35.

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10. ITI 333.

HYDRAZINE SOLUTIONS $NH_2NH_2 \cdot H_2O$

CAUSES BURNS, ANIMAL CARCINOGEN, TOXIC

Synonyms

Hydrazine hydrate.

Physical Properties

Anhydrous form is colorless, oily, fuming liquid; penetrating odor resembling ammonia; bp, 113.5°C.[1] Hydrate is colorless mobile liquid; bp, 119.4°C; mp, −51.7°C.

Fire Hazard

Flash point: 163°C (100% hydrate); 193°C (85% hydrate).[2]

Chemical Properties

Miscible with water and ethanol; insoluble in chloroform and ether.[2]

Hazardous Reactions

2,4-Dinitrochlorobenzene. Strongly exothermic reaction with 2,4-dinitrochlorobenzene.[3]

Mercuric Oxide. Dropwise addition to mercuric oxide may result in an explosion.[2,4]

Sodium. Dropwise addition to a suspension of Na in ether and heating forms sodium hydrazide, which explodes in air; reacts very exothermally with Na with the liberation of H_2 and NH_3.[5]

Stannous Chloride. Reacts to give stannous dihydrazine chloride, which decomposes explosively when heated.[6]

Physiological Properties and Health Hazards

Very corrosive. The liquid burns the eyes and the skin. Eye irritation may be delayed. Swallowing results in severe internal irritation and damage. Violent poison.[1,7] Animal carcinogen. Reasonably anticipated to be a human carcinogen.[8] Avoid contact with eyes and skin.[7] TLV-TWA (hydrazine) 0.01 ppm (0.13 mg/m^3) (skin).[9]

Spillage Disposal

Wear eye protection, butyl rubber gloves,[10] and laboratory coat. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[11] Scoop the solid into a container, transport to the fume hood, and slowly add to water allowing 20 mL of water for each 1 g of hydrazine. Remove the clay and sand by filtration. For each 1 g of hydrazine, place 120 mL (about 25% excess) of household laundry bleach (containing 5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous hydrazine dropwise to the stirred hypochlorite solution at such a rate that the temperature is maintained at 45–50°C. The addition takes about 1 hour. Stirring is continued overnight (at least 12 hours). The reaction mixture can be flushed down the drain with at least 50 times its volume of water.[12,13]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[14]

Small Quantities. Wear butyl rubber gloves,[10] laboratory coat, and eye protection. Work in the fume hood. Prepare a dilute (5%) aqueous solution of hydrazine by adding slowly to the appropriate volume of water. For each 1 g of hydrazine, place 120 mL (about 25% excess) of household laundry bleach (containing 5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous hydrazine dropwise to the stirred hypochlorite solution at such a rate that the temperature is maintained at 45–50°C. The addition takes about 1 hour. Stirring is continued overnight (at least 12 hours). Wash the reaction mixture into the drain.[12,13]

Reactions for Spillage and Waste Disposal

$$H_2NNH_2 + 2OCl^- \rightarrow N_2 + 2Cl^- + 2H_2O$$

REFERENCES

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7. Lux 419.
8. Cas 54, 3; NIEHS III–140.
9. ACGIH 35.
10. LSS.
11. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.
12. PP 94, 64.
13. Cas 54, 51.
14. Ald 1888D.

HYDROCHLORIC ACID HCl

HARMFUL VAPOR, CAUSES BURNS

Physical Properties

Colorless, fuming liquid with pungent smell, commonly available in reagent grade of 38% hydrochloric acid.[1]

Chemical Properties

Miscible with water.[1]

Hazardous Reactions

Potassium Permanganate. A sharp explosion may be produced on adding concentrated acid to potassium permanganate.[2]

Sodium. Aqueous hydrochloric acid reacts explosively with sodium.[3]

Sulfuric Acid. Addition of concentrated hydrochloric acid to concentrated sulphuric acid dehydrates the hydrochloric acid to release 250 times its volume of gas.[4,5]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system, eyes, and skin. Liquid burns the eyes and skin. Swallowing results in severe irritation and damage. Avoid breathing vapor. Prevent contact with eyes and skin.[6] TLV-STEL-C 5 ppm (7.5 mg/m^3).[7]

Spillage Disposal

Instruct others to keep a safe distance. Wear nitrile rubber gloves,[8] laboratory coat, eye protection, and breathing apparatus if required. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the hydrochloric acid has been absorbed, scoop the mixture into a container and transport to the fume hood. Very slowly add the mixture to a pail of cold water. Neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[9]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus if necessary. In the fume hood, slowly add the hydrochloric acid to a pail of cold water (at least a 1:10 dilution of acid to water is suggested). Soda ash or calcium carbonate is then added slowly until neutralization is complete. Wash the resulting solution into the drain. Any solid residue may be treated as normal refuse.[9]

Reactions for Spillage and Waste Disposal

$$2HCl + Na_2CO_3 \rightarrow 2NaCl + CO_2 + H_2O$$

REFERENCES

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2. B 1297.
3. Mellor, Vol. 2, 469, 1941.

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5. Smith, G.B.L. et al., *Inorg. Synth.*, 3, 132, 1950.
6. Lux 421.
7. ACGIH 36.
8. LSS.
9. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.3; Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety,* 6, 25, 1999.

HYDROFLUORIC ACID HF

SERIOUS RISK OF POISONING BY INHALATION, SWALLOWING, OR SKIN CONTACT; CAUSES SEVERE BURNS

Physical Properties

Colorless, fuming liquid with pungent smell, commonly available in concentrations of 47% and 53% HF.[1,2]

Chemical Properties

Miscible with water.[2]

Fire Hazard

Do not store in glass containers.

Potassium Permanganate. Addition of solid potassium permanganate to concentrated hydrofluoric acid (60–90%) is violently exothermic.[3]

Sodium. Reacts explosively with sodium.[4]

Physiological Properties and Health Hazards

Gas severely irritates the respiratory system and eyes. It irritates the skin and painful burns may develop after an interval. The liquid causes severe, painful burns on contact with all body tissues. Swallowing results in immediate and severe internal irritation and damage. The treatment of hydrofluoric acid burns has been described.[5] Prevent inhalation of gas. Prevent contact with skin, eyes, and clothing.[2] TLV-STEL-C (as F) 3 ppm (2.6 mg/m^3).[6]

Spillage Disposal

NOTE: Handle with extreme caution.

Instruct others to maintain a safe distance. Wear self-contained breathing apparatus, eye protection, butyl rubber gloves, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the hydrofluoric acid has been absorbed, scoop the mixture into a plastic pail and transport to the fume hood. Slowly add to a pail of cold water. Add an excess of calcium carbonate or calcium hydroxide. Let the precipitate settle for 24 hours. Decant the solution to the drain. Allow the solid residue (calcium fluoride and bentonite) to dry and package for disposal in accordance with local regulations.[7,8]

Waste Disposal

Wear butyl rubber gloves, self-contained breathing apparatus, eye protection, and a laboratory coat. In the fume hood, slowly add the hydrofluoric acid to a plastic pail of cold water (at least a 1:10 dilution of acid to water is suggested). Add excess calcium carbonate or calcium hydroxide. Let the precipitate settle for 24 hours. Decant the solution to the drain. Allow the solid residue (calcium fluoride) to dry and package for disposal in accordance with local regulations.[7,8]

Reactions for Spillage and Waste Disposal

$$2HF + Ca(OH)_2 \rightarrow \underset{\substack{\text{calcium}\\\text{fluoride}\\\text{(insoluble)}}}{CaF_2} + 2H_2O$$

REFERENCES

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2. Lux 425.
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4. B 1371.
5. Finkel, A.J., *Adv. Fluorine Chem.*, 7, 199, 1973.
6. ACGIH 36.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.3.
8. PPL 167.

HYDROGENATION CATALYSTS

Many hydrogenation catalysts are sufficiently active to cause rapid interaction of hydrogen and/or solvent vapor with air, causing ignition or explosion. This is particularly so where hydrogen is adsorbed on the catalyst (Raney cobalt, nickel, etc.) either before a hydrogenation or after a hydrogenation during separation of catalyst from the reaction mixture. Exposure to air of such a catalyst should be avoided until complete purging with an inert gas, such as nitrogen, has been effected.

With catalysts of high activity and readily reducible substrates, control of the exotherm may be required to prevent runaway reactions, particularly at high pressures.[1]

Platinum and paladium catalysts are nonpyrophoric as normally manufactured.

Spent catalysts should be purged from hydrogen and washed free from organics with water before storage in the water-wet condition. Under no circumstances should any attempt be made to dry a spent catalyst.[2]

See also entries for Palladium, Platinum, and Nickel.

Waste Disposal

Spent catalysts should be kept underwater and recycled or packaged and labeled for disposal.[3]

REFERENCES

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2. B 1632.
3. ITI 446, 525.

HYDROGEN CYANIDE HCN

EXTREMELY POISONOUS GAS AND LIQUID, POISONOUS BY SKIN ABSORPTION, HIGHLY FLAMMABLE

Physical Properties

Colorless liquid or gas; characteristic odor of bitter almonds; bp, 26°C.[1]

Fire Hazard

Flash point, −18°C; explosive limits, 6–41%; ignition temperature, 538°C. If possible, shut off flow of gas and remove cylinder quickly from area in which a fire has developed. Breathing apparatus must be worn during these operations. Use dry chemical foam or carbon dioxide to extinguish.[2]

Chemical Properties

Very soluble in water, the solution being only weakly acidic (does not redden litmus).[1]

Hazardous Reactions

Stable at or below room temperature in the presence of 0.1% acid; polymerizes explosively above 184°C or in the presence of alkali.[3]

Ammonium Chloride. Electrolysis of mixtures of NH_4Cl and HCN can form explosive nitrogen trichloride.[4]

Heavy Metal Cyanides. Detonates explosively in presence of heavy metal cyanides.[5]

Hydrochloric Acid. Explosive reaction occurs when hydrochloric acid is rapidly passed into alcoholic HCN; cold alcoholic HCN should be rapidly added to cold alcoholic hydrochloric acid.[6]

Physiological Properties and Health Hazards

Inhalation of high concentrations leads to shortness of breath, paralysis, unconsciousness, convulsions, and death by respiratory failure. With lethal concentrations, death is extremely rapid although breathing may continue for some time. With low concentrations, the effects are likely to be headache, vertigo, nausea, and vomiting. Chronic exposure over long periods may induce fatigue and weakness. The average fatal dose is 55 mg, which can also be assimilated by skin contact with the liquid. Prevent inhalation of gas. Prevent contact with skin and eyes.[7] Antidote: Sodium nitrite and sodium thiosulfate.[1] TLV-STEL-C 4.7 ppm (skin).[8]

Waste Disposal

Seal cylinder and return to supplier.

Leaking cylinders. Breathing apparatus must be worn. Wear butyl rubber gloves, laboratory coat, and eye protection. In the fume hood, allow gas to leak into a container of sodium hydroxide solution while stopping leak (avoid possibility of suck-back of alkaline material into liquid hydrogen cyanide). Add excess sodium or calcium hypochlorite to the alkali cyanide. Wash the cyanate into the drain.[9]

Reactions for Spillage and Waste Disposal

$$HCN + NaOH \rightarrow NaCN + H_2O$$

$$NaCN + NaOCl \rightarrow NaCNO + NaCl$$

REFERENCES

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5. Wohler, L. et al., *Chem. Ztg.*, 50, 761, 1926.
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7. Lux 424.
8. ACGIH 36.
9. ITI 342.

HYDROGEN FLUORIDE HF

GIVES OFF VERY POISONOUS VAPOR, CAUSES SEVERE BURNS

Physical Properties

Colorless gas or liquid; fumes in air; bp, 19.5°C.[1]

Chemical Properties

Dissolves in water, readily forming hydrofluoric acid.[1]

Fire Hazard

Mercury(II) Oxide. Passing HF into rapidly stirred suspension of mercury(II) oxide may cause violent reaction.[2]

Phosphorus Pentoxide (P_2O_5). Vigorous reaction with P_2O_5 below 20°C.[3]

Oxides. Arsenic trioxide and calcium oxide incandesce in contact with the liquid.[4]

Physiological Properties and Health Hazards

The gas severely irritates the eyes and respiratory system and may cause burns to the eyes. It irritates the skin and painful burns may develop after an interval. The liquid causes severe, painful burns often delayed on contact with all body tissues. Prevent inhalation of gas. Prevent contact with skin and eyes.[5] TLV-STEL-C (as F) 3 ppm (2.6 mg/m^3).[6]

Spillage Disposal

NOTE: Handle with extreme caution.

Instruct others to maintain a safe distance. Wear butyl rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus. Cover liquid spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the hydrogen fluoride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, slowly add to a plastic pail of cold water. Add an excess of calcium carbonate or calcium hydroxide. Let the precipitate settle for 24 hours. Decant the solution to the drain. Allow the solid calcium fluoride, bentonite, and sand to dry and package and label for disposal in accordance with local regulations.[7]

Waste Disposal

Handling precautions for the gas or anhydrous liquid have been detailed.[8] A polythene condenser for disposal of hydrogen fluoride has been described.[8,9]

Gas. Seal cylinder and return to supplier.

Liquid. Wear butyl rubber gloves, self-contained breathing apparatus, eye protection, and laboratory coat. In the fume hood, slowly add the hydrogen fluoride to a plastic pail of cold water (at least a 1:10 dilution of acid to water is suggested). Add excess calcium hydroxide or calcium carbonate. Let the precipitate settle for 24 hours. Wash the resulting solution into the drain. Allow the solid calcium fluoride residue to dry, and package and label for disposal in accordance with local regulations.[7]

Reactions for Spillage and Waste Disposal

$$2HF + CaCO_3 \rightarrow \underset{\substack{\text{calcium}\\\text{fluoride}\\\text{(insoluble)}}}{CaF_2} + H_2O + CO_2$$

REFERENCES

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2. B 1091.
3. B 1433.
4. Mellor, Vol. 2, Suppl. 1, 122, 1956; Vol. 9, 101, 1939.
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6. ACGIH 36.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.3.
8. Keen, M.J. et al., *Chem. Ind.*, 805, 1957.
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HYDROGEN PEROXIDE H_2O_2

CAUSES BURNS, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

Colorless liquid; bitter taste. Distillable in high vacuum. May decompose violently if trace impurities are present; bp, 152°C; mp, –0.43°C.[1]

Fire Hazard

Strong oxidizer. Drying of concentrated product on clothing or other combustible material may cause fire. Avoid contamination from any source, including metals, dust, etc., as contamination may cause rapid decomposition, generation of large volumes of oxygen, and high pressures. Extinguish fire with water.[2]

Chemical Properties

CAUTION: Strong oxidant.

Marketed as an aqueous solution in concentrations of 3–90% by weight. Solutions of hydrogen peroxide gradually deteriorate and are usually stabilized by organic material. Agitation, contact with rough surfaces, metals, alkalies, and many other substances accelerate decomposition.[1]

Store in original closed container. Be sure that the container vent is working properly. Do not add any other compound to the container. When empty, flush container thoroughly with clean water.[1]

Hazardous Reactions

Hazards associated with the use of H_2O_2 solutions have been published.[3–5] These include the following:

1. Release of enough energy during catalytic decomposition of 65% peroxide to evaporate all water present and formed, and subsequent liability of ignition of combustible materials.
2. Most cellulosic materials contain enough catalyst to cause spontaneous ignition with 90% peroxide.
3. Contamination of concentrated peroxide causes possibility of explosion. Readily oxidizable materials, or alkaline substances containing heavy metals, may react violently.
4. Soluble fuels (acetone, ethanol, and glycerol) will detonate on admixture with peroxide of over 30% concentration, the violence increasing with concentration.
5. Handling systems must exclude fittings of iron, brass, copper, Monel, and screwed joints caulked with red lead.
6. Concentrated peroxide may decompose violently in contact with iron, copper, chromium, and most other metals or their salts, and dust (which frequently contains rust). Absolute cleanliness, suitable equipment (PVC, butyl, or Neoprene rubber), and personal protection are essential for safe handling. During concentration under vacuum of aqueous or of aqueous-alcoholic solutions of hydrogen peroxide, violent explosions occurred when the concentration was sufficiently high (probably above 90%). Detonation of hydrogen peroxide vapor has been studied experimentally.[6]

Acetic Acid. During the addition to acetic acid, the temperature must be controlled so that the reaction proceeds steadily; otherwise, reagent concentrations may increase to the point where reaction proceeds explosively.[7]

Acetic Anhydride. Reaction mixture with acetic anhydride must be kept acidic to prevent separation of the highly explosive diacetyl peroxide.[8]

Acetone. Readily forms explosive dimeric and trimeric peroxides with acetone, which have resulted in explosions during workup of reactions run in acetone[8,9]; acetone should not be used as solvent for peroxide oxidations.[8,10]

Alcohols. Explosion of mixtures with alcohols can be initiated by shock or heat[8]; furfuryl alcohol ignites on contact with 85% peroxide[8]; addition of concentrated acid to alcohol–peroxide mixtures may result in explosion; adequate cooling must accompany initial additions of *tert*-butyl alcohol to H_2O_2:H_2SO_4 (1:2 by weight) mixtures to prevent explosive decomposition.[11]

Carbon. Addition of charcoal to concentrated H_2O_2 results in violent decomposition; if a trace of MnO_2 is present, immediate ignition occurs.[12]

Carboxylic Acids. The peroxyacids formed may be unstable and explosive.[13]

Ketones. Hazardous reaction with acetone and nitric acid; other ketones yield shock- and heat-sensitive explosive peroxides.[14]

Metals. Noble metals, especially when finely divided, cause explosive decomposition; manganese and iron ignite on contact[13]; cobalt, iron, lead, manganese, mercury, nickel, and their oxides (especially rust) promote decomposition and must be excluded when using peroxides; potassium and sodium are oxidized violently.[15]

Nitrogen-Containing Bases. Ammonia in 99.6% H_2O_2 exploded violently[16]; with hydrazine hydrate, initial reaction may be slow, followed by sudden explosive reaction[17]; 1,1-dimethylhydrazine ignites on contact with concentrated H_2O_2.[18]

Organic Compounds. Mixtures of H_2O_2 and organic compounds may be dangerously explosive unless procedures are carefully controlled[18]; destroying organic matter with 50% H_2O_2 and concentrated H_2SO_4 prior to analysis can be hazardous[19]; during epoxidation of unsaturated organic compounds using 70% H_2O_2, reaction must proceed steadily to avoid build-up of reagents; during hydroxylation of vinyl acetate, an explosion occurred when the reaction mixture was being vacuum distilled.[20]

Phosphorus Pentoxide. Violent reaction may be moderated using acetonitrile as diluent.[21]

Tin(II) Chloride. Strongly exothermic reaction is violent when peroxide solution is more than 3% by weight.[22]

Physiological Properties and Health Hazards

Solutions are irritants to the mucous membranes, eyes, and skin. If swallowed, the sudden evolution of oxygen may cause injury by acute distension of the stomach and also nausea, vomiting, and internal bleeding. Avoid contact with eyes and skin.[23] TLV-TWA 1 ppm (1.4 mg/m^3).[24]

Spillage Disposal

Wear face shield and goggles, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Dampen with water. Using a soft plastic scoop, transfer the mix into a container. Transport to the fume hood. Slowly add to a pail of cold water. Gradually add to an excess of aqueous sodium metabisulfite solution. Decant the liquid to the drain. Treat the solid as normal refuse.[25–27]

Waste Disposal

Wear butyl rubber gloves, eye protection, and laboratory coat. A body shield should be available. In the fume hood, prepare a dilute solution (5%) of peroxide by cautiously adding to a large volume of water. Gradually, while stirring, add to a 50% excess of aqueous sodium metabisulfite in a round-bottom flask equipped with a thermometer. An increase in temperature indicates that reaction is taking place. Acidify the reaction if it does not proceed spontaneously. Neutralize the reaction mixture and wash into the drain.[25–27]

Reactions for Spillage and Waste Disposal

$$2H_2O_2 + S_2O_5^{2-} \rightarrow 2SO_4^{2-} + H_2O + 2H^+$$

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22. Vickery, R.C. et al., *Chem. Ind.*, 657, 1949.
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24. ACGIH 36.
25. Armour, M.A., Browne, L.M., and Weir, G.L., *Laboratory Disposal Method for Hydrogen Peroxide,* University of Alberta, Edmonton, 1997; *Haz. Mat. Spills Tec.,* 29.1.
26. ITI 345; Aldrich 782.
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HYDROGEN SELENIDE H_2Se

TOXIC

Synonyms

Selenium hydride.

Physical Properties

Colorless gas; disagreeable odor; bp, –41.3°C; liquid at 0°C under 6.6 atm.[1]

Fire Hazard

High fire potential. Flammable; keep away from heat and open flames.[2]

Chemical Properties

Soluble in water, carbon disulfide, and carbonyl chloride. Unites directly with most metals.[1]

Hazardous Reactions

Air. Forms explosive mixtures.[2]

Oxidizing Agents. Reacts vigorously with strong oxidizing agents such as hydrogen peroxide and nitric acid.[3,4]

Physiological Properties and Health Hazards

Toxic by inhalation. Irritating to the skin, eyes, and mucous membranes. Causes garlic breath, dizziness, and nausea. Can cause damage to the lungs and liver as well as conjunctivitis, and is an allergen.[1,2] TLV-TWA (Se) 0.05 ppm (0.16 mg/m^3).[4.]

Waste Disposal

If possible, seal the cylinder and return to supplier. Otherwise, wear nitrile rubber gloves, face shield, goggles, breathing equipment, and laboratory coat. Remove leaking cylinder to a safe area outdoors or an efficient fume hood. Fit the cylinder with an appropriate control valve and a length of hose. Discharge the gas slowly into 15% aqueous sodium hydroxide. Cautiously make the sodium hydroxide solution strongly acidic with concentrated hydrochloric acid, and then add sodium sulfite until reaction ceases. After standing for 24 hours, the dark brown precipitate of selenium metal can be filtered, dried, and sent to a secure landfill or recycled. The colorless filtrate can be washed into the drain.[5,6]

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HYDROGEN SULFIDE H_2S

EXTREMELY FLAMMABLE, TOXIC BY INHALATION

Physical Properties

Colorless gas with an offensive odor characteristic of rotten eggs; bp, −60.33°C.[1]

Fire Hazard

Explosive limits, 4.3–46%; ignition temperature, 260°C. Shut off the gas flow and extinguish fire with water, dry chemical, or carbon dioxide. If possible, cylinders should be removed quickly from an area in which a fire has developed.[2]

Chemical Properties

One gram dissolves in 242 mL of water, 94.3 mL of absolute ethanol, and 48.5 mL of ether at 20°C; also soluble in glycerol.[1] Aqueous solutions are not stable; absorbed oxygen causes formation of elemental sulfur.[2]

Hazardous Reactions

4-Bromobenzenediazonium Chloride. Interaction with hydrogen sulfide produces a solid product that explodes under water at 0°C.[3]

Copper and Oxygen. A 1:2 mixture of hydrogen sulfide and oxygen ignites in the presence of copper powder.[4]

Metal Oxides. Hydrogen sulfide may ignite in contact with barium dioxide, chromium trioxide, copper oxide, lead dioxide, manganese dioxide, nickel oxide, silver mono- and dioxides, sodium peroxide, and thallium trioxide. It may explode in the presence of air and mixtures of calcium or barium oxide with mercury or nickel oxide.[5]

Metals. Copper powder reaches red heat in a mixture of air and hydrogen sulfide, while finely divided tungsten glows in hydrogen sulfide.[6] Sodium melts and then ignites in the presence of moist hydrogen sulfide.[7]

Oxidants. Hydrogen sulfide ignites on contact with silver bromate,[8] bromine pentafluoride,[9] mercury(I) bromate,[10] lead hypochlorite,[11] copper chromate,[12] fluorine,[13] fuming nitric acid,[14] solid sodium peroxide,[15] and dry or moist lead oxide.[5,16] Dichlorine oxide[17] or chlorine trifluoride[18] explodes on contact with hydrogen sulfide. Heated chromium trioxide incandesces in a stream of hydrogen sulfide.[19]

Oxygen. Mixtures of hydrogen sulfide and oxygen are explosive at temperatures between 280 and 360°C.[20]

Rust. Hydrogen sulfide may ignite if passed through rusty iron pipes.[21]

Silver Fulminate. Explodes violently at room temperature in contact with hydrogen sulfide.[22]

Soda-Lime. There is an exothermic reaction between granular soda-lime and hydrogen sulfide that results in a violent explosion in the presence of oxygen. Barium hydroxide and sodium or potassium hydroxides behave similarly.[6]

Physiological Properties and Health Hazards

Gas at high concentration results in immediate unconsciousness, and then cessation of breathing. At lower concentrations, gas irritates the respiratory system and eyes and causes headache, dizziness, and weakness. Avoid inhaling gas.[23] TLV-TWA 10 ppm (14 mg/m^3); TLV-STEL 15 ppm (21 mg/m^3).[23]

Spillage Disposal

See waste disposal.

Waste Disposal

Wear butyl rubber gloves, eye protection, and laboratory coat. Work in fume hood or wear self-contained breathing apparatus. If a leak of hydrogen sulfide occurs, seal the cylinder and return to the supplier. If the valve is leaking, the gas can be bubbled through a ferric chloride solution. Be sure to include a trap in the line to prevent the solution being sucked back into the cylinder. If this cannot be done, the cylinder should be placed in or adjacent to a fume hood and left to bleed off.[25] Surplus gas or a leaking cylinder can be vented slowly into a water-fed scrubbing tower or column in a fume cupboard, or into a fume cupboard served by such a tower.[23]

Small Quantities. Small amounts of hydrogen sulfide can be destroyed with hypochlorite. Work in a good fume hood. To keep the reaction under control, first absorb the hydrogen sulfide in excess aqueous sodium hydroxide and place in dropping funnel. Place 25% excess of commercial laundry bleach (e.g., Chlorox) containing 5.25% sodium hypochlorite into a large, three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous sulfide solution dropwise to the stirred hypochlorite solution. Monitor the reaction by temperature change. After addition is complete, allow the reaction mixture to stand for several hours. Flush the reaction mixture down the drain.

> ***CAUTION***: Hydrogen sulfide is an acute poison and is one of the more dangerous chemicals in the laboratory.[26]

Reactions for Spillage and Waste Disposal

$$H_2S + 2NaOH \rightarrow Na_2S + 2H_2O$$

$$Na_2S + 4NaOCl \rightarrow Na_2SO_4 + 4HCl$$

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HYDROGEN TELLURIDE H_2Te

TOXIC

Synonyms

Tellurium dihydride.

Physical Properties

Colorless gas; offensive garlic-like odor; mp, –49°C; bp, –2°C.[1]

Chemical Properties

Liquid, decomposed immediately by light. Dry gas is stable to light but decomposes in presence of dust, moisture, rubber, cork, etc.[1,2] Soluble in water; decomposes in alcohol.[2]

Fire Hazard

Nitric Acid. Cold fuming nitric acid ignites hydrogen telluride, sometimes explosively.[3]

Physiological Properties and Health Hazards

Highly toxic.[4] Irritating to the eyes and mucous membranes. Imparts offensive odor to breath. Causes dizziness and nausea.[1]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, face shield, and self-contained breathing apparatus. Remove the leaking cylinder to a safe area outdoors or to an efficient fume hood. Fit the cylinder with an appropriate control valve and length of hose. Discharge the gas slowly into 15% aqueous sodium hydroxide (forms a water-soluble complex). Neutralize the solution to pH 7 with 1 M sulfuric acid (prepared by cautiously adding 5 mL of concentrated acid to 85 mL of cold water). Slowly add 1 M sodium sulfide (prepared by adding 24 g of sodium sulfide monohydrate to 100 mL of water) to the tellurium solution. Check that the solution is neutral (pHydrion paper) and adjust to neutrality with 1 M sulfuric acid if necessary. The precipitate is separated by filtration and packaged for disposal in a secure landfill. The aqueous solution is washed into the drain.[5]

Reactions for Spillage and Waste Disposal

$$Te^{4+} + 2Na_2S \rightarrow \underset{\substack{\text{tellurium}\\ \text{sulfide}\\ \text{(insoluble)}}}{TeS_2} + 4Na^+$$

REFERENCES

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N-HYDROXY-2-ACETYLAMINOFLUORENE $C_{15}H_{13}O_2N$

ANIMAL CARCINOGEN

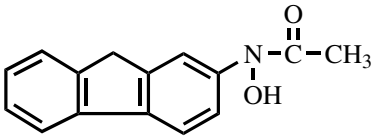
Physical Properties

White crystals; mp, 144–146°C.[1]

Chemical Properties

Dissolves in hot water; low solubility in cold water; soluble in dilute alkali, dimethylsulfoxide, acetone, and other organic solvents; unstable to oxygen.[1]

Structure



Physiological Properties and Health Hazards

RNA synthesis is inhibited by this compound. Carcinogen in rodents.[2]

Spillage Disposal

Wear a dust mask, goggles, rubber gloves, and protective clothing. Carefully scoop the solid into a beaker and transport to the fume hood. Destroy the material using the waste disposal procedure. Wash the spill site with soap and water, disposing of the cleaning materials in a sealed bag or container that can be packaged and labeled for disposal by burning.[3]

Waste Disposal

Small Quantities. Wear goggles, rubber gloves, and protective clothing. Work in the fume hood. To 0.25 g of N-hydroxy-2-acetylaminofluorene in a 50-mL round-bottom flask, add 10 mL of concentrated hydrochloric acid. Fit the flask with a condenser and heat under reflux for at least 10 hours. Cool the contents of the flask to 0°C in an ice-salt bath and, over a period of 5 minutes, add dropwise a solution of 0.13 g of sodium nitrite in 0.3 mL of water. Stir the mixture for 30 minutes, and then slowly add 2.7 mL of ice–cold 50% hypophosphorus acid. After stirring at room temperature for 16 hours, the mixture is filtered. Wash the filtrate into the drain with water and discard the solid (fluorene) with normal refuse or dispose of by burning.[3–5]

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IFOSPHAMIDE $C_7H_{15}Cl_2N_2O_2P$

ANIMAL CARCINOGEN

Synonyms

N,3-Bis(2-chloroethyl)tetrahydro-2H-1,3,2-oxazaphosphorin-2-amine-2-oxide, 3-(2-chloro-ethyl)-2-[(2-chloroethyl)amino]tetrahydro-2H-1,3,2-oxazaphosphorin-2-oxide, iphosphamide, NSC-109724.[1]

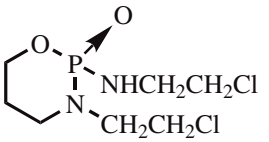
Physical Properties

Crystals from anhydrous ether[1]; mp, 39–41°C.

Chemical Properties

Soluble in water and carbon disulfide; very soluble in dichloromethane; sensitive to hydrolysis, oxidation, and heat.[2]

Structure



Physiological Properties and Health Hazards

Most common and serious toxicity to patients includes low blood count, as well as vomiting, nausea, loss of hair, water retention, and cardiac damage.[3] LD_{50} in rats, 160 mg/kg.[1]

Spillage Disposal

Solutions: Wear goggles, rubber gloves and protective clothing. Cover the spill with 1:1:1 mixture by weight soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of ifosphamide, allowing 1 mL of 12% sodium hydroxide solution to degrade 10 mg of ifosphamide in 2 mL of dimethylformamide when refluxed for 4 hours. Neutralize the solution by carefully adding 2 M hydrochloric acid (10 mL of concentrated hydrochloric acid added to 50 mL of water) and washing into the drain with water.[2,4]

Waste Disposal

Wear protective gloves and clothing, and goggles. To each 2.5 mL of solution containing 10 mg of ifosphamide, add 10 mL of 12% of sodium hydroxide solution and 20 mL of dimethylformamide. Allow the mixture to reflux for 4 hours. Neutralize the cooled solution by carefully adding 2 M hydrochloric acid, and washing into the drain with a large volume of water. For glassware containing residues of ifosphamide, rinse thoroughly with dimethylformamide. The rinses are added to one-half their volume of 12% sodium hydroxide solution and the mixture heated under reflux for 4 hours. Neutralize the cooled solution by carefully adding 2 M hydrochloric acid and then washing it into the drain with water.[2,4]

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IODINE I_2

HARMFUL VAPOR, CAUSES BURNS

Physical Properties

Bluish-black crystalline scales or plates with a characteristic odor and sharp acrid taste; violet corrosive vapor; mp, 113.6°C; bp, 185.2°C.[1]

Chemical Properties

Almost insoluble in water; freely soluble in aqueous solutions of iodide. Soluble in benzene, carbon disulfide, ethanol, ethyl ether, cycohexane, carbon tetrachloride, chloroform, glacial acetic acid, and glycerol oils.[1]

Hazardous Reactions

Acetaldehyde. Interaction may be violent.[2]

Ammonia. Highly explosive addition compounds formed with ammonia solutions.[3]

Ammonia and Potassium. In the presence of potassium, iodine in liquid ammonia may form the highly explosive nitrogen triiodide.[4]

Ethanol and Phosphorus. Interaction of ethanol, phosphorus, and iodine to form iodomethane is considered too dangerous for a school experiment.[2]

Formamide. Bottles containing a modified Karl Fischer reagent when stored for several months may explode.[5]

Halogens or Interhalogens. Bromine trifluoride incandesces in contact with I_2.[6] Bromine pentafluoride ignites in contact with I_2,[7] as do chlorine trifluoride[8] and fluorine.[9]

Metal Acetylides or Carbides. Many metal acetylides react very exothermically with I_2.[10]

Metals. Antimony powder reacts violently; mixture of potassium and iodine explodes weakly on impact; and potassium ignites in contact with molten I_2.[11]

Metals and Water. Flash ignition occurs when mixtures of iodine with powdered aluminum, magnesium, or zinc are moistened with a drop of water.[10]

Nonmetals. Powdered boron reacts spontaneously with iodine at 700°C.[12] Interaction of phosphorus and I_2 in carbon disulfide is rapid.[13–15]

Silver Azide. Silver azide is converted by ethereal I_2 to the highly explosive compound iodine azide.[16]

Sodium Phosphinate. Violently exothermic reaction of anhydrous salt with I_2.[17]

Tetramminecopper(II) Sulfate. Addition of ethanol to a mixture with I_2 led to explosion involving the formation of N-iodine derivatives.[18]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. Solid irritates the eyes and burns the skin. Swallowing causes severe internal irritation and damage. Avoid breathing vapor. Avoid contact with eyes and skin.[19] TLV-STEL-C 0.1 ppm (1.0 mg/m^3).[20]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. If the spillage is large and in a confined area, breathing apparatus should be worn. Large quantities are best disposed of by sweeping up with sand and burying in open wasteland. Small quantities can be dealt with by

dissolving in sodium thiosulfate or sodium metabisulfite solution and washing the resulting solution into the drain. Iodine stains on flooring can be cleared by mopping with thiosulfate or metabisulfite solution.[19]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or reuse.[21]

Small Quantities. Wear nitrile rubber gloves, eye protection, and laboratory coat. In the fume hood, cautiously add iodine (5 g) to a solution of sodium thiosulfate (300 mL of 4%) containing sodium carbonate (0.1 g). Stir until all of the iodine has dissolved (solution is colorless), and then neutralize with sodium carbonate. When reduction is complete, add sodium carbonate or dilute hydrochloric acid to neutralize the solution. Wash into the drain.[19]

Reactions for Spillage and Waste Disposal

$$I_2 + Na_2S_2O_3 + Na_2CO_3 \rightarrow 2NaI + Na_2SO_4 + S + CO_2$$

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20. ACGIH 36.
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IODOMETHANE CH_3I

TOXIC IN CONTACT WITH SKIN, GIVES OFF POISONOUS VAPOR, CAUSES BURNS

Synonyms

Methyl iodide.

Physical Properties

Colorless liquid that turns brown on exposure to light; bp, 42.5°C.[1]

Chemical Properties

Sparingly soluble in water. Miscible with alcohol and ether. Protect from light.[1]

Hazardous Reactions

Sodium. Vigorous reaction with Na dispersed in either toluene[2] or benzene.[3]

Physiological Properties and Health Hazards

Inhalation of vapor may cause dizziness, drowsiness, mental confusion, muscular twitching, and delirium. The vapor and liquid irritate the eyes and distort the vision. The liquid irritates the skin and may cause blistering. The liquid must be assumed to be an irritant and poisonous if taken by mouth. Avoid breathing vapor. Prevent contact with skin and eyes.[4] An experimental neoplastic.[5] TLV-TWA 2 ppm (12 mg/m^3) (skin).[6]

Spillage Disposal

Instruct others to maintain a safe distance. Wear eye protection, breathing apparatus, laboratory coat, and nitrile rubber gloves. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand, and transport to fume hood for atmospheric evaporation. Thoroughly wash spill site with water and soap or detergent.[4,7]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the methyl iodide in a flammable solvent and burn in a furnace equipped with afterburner and scrubber.[8]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place 6.3 g (0.1 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, dropping funnel, and heating mantle. While briskly stirring, rapidly add 25 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. The solution is heated to gentle reflux and iodomethane (5 mL, 11.4 g, 0.08 mol) is added dropwise at such a rate to maintain gentle reflux. Continue heating under reflux, while stirring, for another 2 hours. Cool the mixture, dilute it with 24 mL of water, neutralize it, and wash down the drain with 50 times its volume of water.[9]

Reactions for Spillage and Waste Disposal

$$CH_3I + KOH \rightarrow \underset{\text{methanol}}{CH_3OH} + KI$$

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IPRODIONE $C_{13}H_{13}Cl_2N_3O_3$

FUNGICIDE

Synonyms

Rovral®, 3-(3,5-dichlorophenyl)-N-(1-methylethyl)-2,4-dioxo-1-imidazolinecarboxamide.

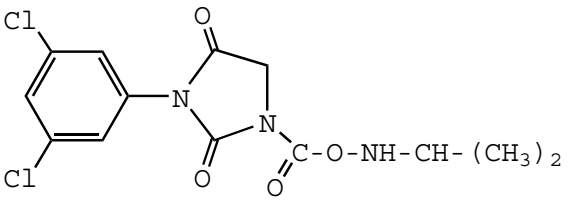
Physical Properties

White, odorless, nonhygroscopic crystals; mp, 136°C.[1]

Chemical Properties

Solubility at 20°C: water 13 mg/L, ethanol 25 mg/L, methanol 25 mg/L, acetone 300 mg/L, dichloromethane 500 mg/L, and DMF 500 mg/L.[2]

Structure



Iprodione

Physiological Properties and Health Hazards

LD_{50} (oral mice) 3500 mg/kg.[2] LD_{50} (oral rat) 4000 mg/kg.[2] LC_{50} (rainbow trout, 96 hours) 6.7 mg/L.[3] Very low, acute mammalian toxicity. Avoid inhaling mist. A mild eye irritant.[1]

Formulations

Wettable powder, flowable.

Spillage Disposal

Wear gloves and eye protection. The powder can be swept up and disposed as for waste disposal. For spills of diluted formulation, cover with a 1:1:1 mixture of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and add to a pail of water. Estimate the weight of iprodione in the solution spilled, and for each 1 g, add 120 mL of household bleach to the solution in the pail. Allow it to stand overnight and decant the solution into the drain. Discard solid residue of sand and bentonite as normal refuse.[4]

Waste Disposal

Concentrated household bleach can be used to degrade iprodione. To each 1 g of commercial iprodione, add 120 mL of undiluted household bleach. The fungicide is completely degraded after 2 days at room temperature or 4 hours at 50°C. Wash the resulting solution into the drain with a large volume of water.[4]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer by running the machine through a complete wash cycle with detergent (but without clothing) before washing other clothes.

When wettable powder formulation of iprodione was diluted to field strength (0.05% a.i.) and used to contaminate 100% cotton and 65/35 polyester/cotton fabric, it was found that a single warm water wash (50°C) left approximately 1% iprodione residues on the fabric.[5]

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IRON PENTACARBONYL $Fe(CO)_5$

PYROPHORIC IN AIR, SEVERE POISON

Physical Properties

Colorless to yellow oily liquid; mp, –20°C; bp, 103°C.[1]

Fire Hazard

Flash point, –15°C; ignition temperature, ambient.[2] Flammable.[3]

Chemical Properties

Insoluble in water; soluble in most organic solvents. Slightly soluble in alcohol; decomposed by light.[1]

Hazardous Reactions

Acetic Acid and Water. Forms brown pyrophoric powder in acetic acid containing more than 5% water.[4]

Nitrogen Oxide. Explodes when heated above 50°C in an autoclave.[5]

Physiological Properties and Health Hazards

Severe poison.[3] Decomposes readily to carbon monoxide; lung irritant; affects central nervous system; causes liver and kidney damage. Inhalation may cause dizziness, nausea, vomiting; delayed reaction may occur with chest pain, cough, and difficult breathing, possibly even cyanosis and circulatory collapse.[6] TLV-TWA 0.1 ppm (0.23 mg/m^3); TLV-STEL 0.2 ppm (0.45 mg/m^3).[7]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[8] For each 1 mL of iron pentacarbonyl, place 45–50 mL of household bleach (about 5% sodium hypochlorite solution) in a plastic bucket equipped with a stirrer and located in a fume hood. Dilute the bleach with three times its volume of water and, to this solution, slowly add the contaminated spill mix while stirring. Continue the stirring for a period of 48 hours, and then filter the slurry using vacuum filtration. The solids may be treated as ordinary refuse and the filtrate, after neutralization, may be washed into the drain.[8,9]

Waste Disposal

Small Quantities. Work in the fume hood. Wear eye protection, butyl rubber gloves, and laboratory coat. The reaction is carried out in a three-necked, round-bottom flask fitted with gas inlet and exit tubes, a dropping funnel, and a magnetic stirrer. The flask is flushed with nitrogen and sodium hypochlorite solution (13 mL) (household bleach) added. Gradually add a 5% solution of the iron pentacarbonyl (1 g) in a hydrocarbon solvent from the dropping funnel to the stirred hypochlorite solution. After completion of the addition, continue stirring for 1 hour. Filter the flask contents by vacuum, and treat the solids as ordinary refuse. Place the filtrate in a separatory funnel, and after neutralization, wash the aqueous layer into the drain. The organic layer is either recycled or placed in a suitable solvent container for disposal by burning.[3,8,9]

Reactions for Spillage and Waste Disposal

$$2Fe(CO)_5 + 3NaOCl + 3H_2O \rightarrow \underset{\text{ferric hydroxide}}{2Fe(OH)_3} + 5CO + 3NaCl$$

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LEAD SALTS Pb salts

HARMFUL IF TAKEN INTERNALLY, DANGER OF CUMULATIVE EFFECTS

Physical Properties

White or colored crystals or powders.[1]

Fire Hazard

Lead Acetate $Pb(OCOCH_3)_2$

Potassium Bromate and Acetic Acid. The double salt, lead acetate-lead bromate, may be formed in the preparation of lead bromate from lead acetate and potassium bromate in acetic acid. It is explosive and very sensitive to friction.[2]

Lead(II) Azide $Pb(N_3)_2$

Used widely as a detonator.[3] A vacuum-desiccated sample exploded violently when touched with a metal spatula.[4]

Copper or Zinc. Prolonged contact of the azide with copper, zinc, or their alloys forms traces of extremely explosive copper or zinc azides.[4]

Lead Bromate $Pb(BrO_3)_2$

Explosive salt.[5]

Lead Carbonate $PbCO_3$

Lead carbonate ignites and burns fiercely in fluorine.[6]

Lead Chlorate $Pb(ClO_3)_2$

Thermal decomposition may be explosive.[7]

Lead Chloride $PbCl_2$

Calcium. Explosive interaction on warming.[8]

Lead Chromate $PbCrO_4$

Sulfur. The mixture with sulfur is pyrophoric.[9]

Lead Nitrate $Pb(NO_3)_2$

Cyclopentadienylsodium. Use of lead nitrate to prepare cyclopentadienyllead may result in explosion during purification of the crude product by high-vacuum sublimation at 108–130°C.[10]

Potassium Acetate. A heated mixture may explode violently.[10]

Lead(II) Oxide PbO

Dichlorodimethylsilane. The pure silane ignites in presence of lead oxide.[11]

Metals. Mixtures with aluminum and titanium give violent reaction on heating; mixture with zirconium explodes on heating; and mixture with finely divided sodium ignites.[12,13]

Nonmetals. Mixture with boron incandesces on heating; and mixture with silicon gives vigorous reaction on heating.[13]

Performic Acid. Violent or explosive decomposition of the acid occurs.[14]

Lead(IV) Oxide PbO_2

Hydrogen Sulfide. Ignition occurs on contact of the gas with the metal oxide.[15]

Metals. Potassium reacts explosively and magnesium violently.[16]

Nitroalkanes. Ignition temperature of nitromethane, nitroethane, and 1-nitropropane, and sensitivity of nitromethane to detonation lowered by contact with lead oxide.[17]

Nitrogen Compounds. Hydroxylamine ignites and phenylhydrazine reacts vigorously on contact with the oxide.[18]

Nonmetals. Boron or yellow phosphorus explodes violently on grinding with the oxide; and sulfur or red phosphorus ignites on grinding with the oxide.[19]

Lead Perchlorate $Pb(ClO_4)_2$

Methanol. Saturated solution in methanol exploded violently when disturbed, possibly due to the formation of methyl perchlorate.[20]

Lead Picrate $Pb[OC_6H_2(NO_2)_3]_2$

Qualitative inorganic analysis of compounds containing lead and salicylate radicals can lead to the formation of explosive lead picrate. This occurs due to conversion of salicylic acid to picric acid during evaporation with nitric acid.[21]

Lead Sulfate $PbSO_4$

Potassium. Violent or explosive interaction.[16]

Lead Thiocyanate $Pb(SCN)_2$

The salt is explosive.[22]

Physiological Properties and Health Hazards

Inhaling the dust or swallowing the solid may result in vomiting, diarrhea, and collapse with severe internal damage. Prolonged exposure results in loss of appetite, pallor, anemia, constipation, colic, and a blue line on the gums. Avoid breathing dust.[1] TLV-TWA 0.05 mg/m^3.[23]

Spillage Disposal

Aqueous Solutions. Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture of sodium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a pail and add water to dissolve the sodium carbonate. Allow to stand until the solids have settled. Decant the liquid to another container and discard the solid as normal refuse. Add sodium metasilicate solution to the liquid following the procedure described under waste disposal for dilute solutions.

Package Lots. Place in a separate labeled container for recycling or disposal of insoluble salts in a secure landfill.[24]

Small Quantities of Soluble Salts. Wear nitrile rubber gloves, laboratory coat, and eye protection. Dissolve the soluble lead salt (0.04 mol, e.g., 11 g of lead chloride) in water (200 mL) and add, while stirring, a solution of sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$, 25 g, 0.12 mol) in water (200 mL). Adjust the pH to about 9 by adding 2 M sulfuric acid. Collect the precipitate by filtration, or allow the supernatant liquid to evaporate in a large evaporating dish in the fume hood. Package and label the dry solid for disposal in accordance with local regulations. For dilute solutions of lead salts, add the sodium metasilicate solution until there is no further precipitation, adjust to pH 9, and allow the solution to stand overnight before collecting the solid by precipitation or allowing the liquid to evaporate.[25]

Reactions for Spillage and Waste Disposal

$$Pb^{+} + Na_2SiO_3 \rightarrow PbSiO_3 + 2Na^{+}$$
lead
silicate
(insoluble)

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LITHIUM Li

CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GASES

Physical Properties

Silver-white metal, becoming yellowish on exposure to moist air; mp, 180.5°C.[1]

Fire Hazard

Extinguish fire with special extinguishant (class D fire extinguisher); powdered graphite, LiCl, KCl, or zirconium silicate may also be used.[2]

WARNING: Possible violent reaction with water.

Chemical Properties

Reacts with water, forming lithium hydroxide and hydrogen. Keep under mineral oil or other liquid free from oxygen or water.[1]

Hazardous Reactions

Atmospheric Gases. Finely divided metal may ignite in air at room temperature; burns in O_2, N_2, or CO_2, and is difficult to extinguish once it is alight.[2]

Bromobenzene. Vigorous stirring of finely powdered lithium in bromobenzene resulted in an explosion.[3]

Halocarbons. Forms explosive mixtures with bromoform, carbon tetra-bromide, -chloride, and -iodide, chloroform, dichloro- and diiodomethane, fluorotrichloromethane, tetrachloroethylene, trichloroethylene, and 1,1,2-trichlorotrifluoroethane[3–5]; may explode when washed with carbon tetrachloride, and then cut with a knife; hexane should be used for washing.[3]

Halogens. Mixtures with bromine explode only on heavy impact; mixtures with iodine are highly exothermic above 200°C.[4,6]

Mercury. Formation of lithium amalgam may be explosive.[7]

Metal Chlorides. Ignites on contact with chromium trichloride or zirconium tetrachloride.[8]

Metal Oxides. Reduction of metal oxides such as chromium(III) oxide, molybdenum trioxide, niobium pentoxide, tungsten trioxide, and vanadium pentoxide is violently exothermic and rapid.[9]

Nitric Acid. Ignites on contact.[8]

Nonmetal Oxides. Molten lithium burns vigorously in CO_2; lithium carbonyl is formed with CO in liquid ammonia and reacts explosively with water or air; silica or glass is rapidly attacked at 250°C.[10]

Sodium Carbonate and Sodium Chloride. These cannot be used as fire extinguishers because sodium is liberated.[11]

Sulfur. Violent reaction if either is molten; vigorous reaction in liquid ammonia.[12]

Water. Powdered metal reacts explosively; shavings react violently with hot water, and moderately vigorously with cold water.[13]

Physiological Properties and Health Hazards

Lithium ion may injure kidneys, especially if sodium intake is limited.[1] Lithium reacts with moisture, forming lithium hydroxide, which irritates the skin and mucous surfaces and may cause burns. Avoid contact with skin, eyes, and clothing.[14]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a pail and transport to the fume hood. Slowly, while stirring, add to a large quantity of 95% ethanol. Mix and let stand for 24 hours. Dilute the solution with water and wash into the drain. The solid residue may be treated as normal refuse.[15,16]

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Burn in an open furnace. Use caution[2] and limit to 100-g packages.[16]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Equip a three-necked, round-bottom flask with a stirrer, dropping funnel, condenser, and heating mantle. Flush the flask with nitrogen and place the lithium (cut in small pieces) in it. Add 30 mL of 95% ethanol per gram of lithium at a rate that causes rapid refluxing. Stirring is started as soon as enough ethanol has been added to make it feasible. The mixture is stirred and heated under reflux until the lithium is dissolved. The heat is turned off, and an equal volume of water is added at a rate that causes no more than mild refluxing. The solution is then cooled, neutralized with 6 M hydrochloric acid (cautiously add a volume of concentrated acid to an equal volume of cold water), and washed into the drain.[16]

Reactions for Spillage and Waste Disposal

$$Li + CH_3CH_2OH \rightarrow LiOCH_2CH_3 + H^+$$

$$LiOCH_2CH_3 + H_2O \rightarrow \underset{\substack{\text{lithium}\\\text{hydroxide}\\\text{(insoluble)}}}{LiOH} + CH_3CH_2OH$$

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LITHIUM ALUMINUM HYDRIDE $LiAlH_4$

CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GASES

Physical Properties

Microcrystalline white powder when pure, gray when aluminum impurity present; decomposes above 125°C.[1]

Fire Hazard

Catches fire on contact with small quantities of water or by heating. Such a fire is best extinguished using a class D fire extinguisher or by smothering with sand and disposing of residue in the manner given under spillage disposal. Do not use water.[2]

Chemical Properties

Keep container tightly closed. Strong reductant; reacts rapidly with water with evolution of hydrogen. Reacts rapidly with alcohols.[1]

Hazardous Reactions

Air. May ignite on grinding in a dry mortar; ignites in moist air or oxygen; spilled ether mixture may ignite after ether has evaporated.[3]

Alkyl Benzoates. Reduction of benzaldehyde with four-fold excess of $LiAlH_4$ may be dangerous.[4]

Bis(2-methoxyethyl) Ether. Violent explosion occurred when the ether was distilled from $LiAlH_4$ at 200°C.[5]

Boron Trifluoride Diethyl Etherate. Addition of lumps of $LiAlH_4$ to etherate-containing peroxide caused explosion.[6]

1,2-Dimethoxyethane. Explosion resulted during addition of 1,2-dimethoxyethane to $LiAlH_4$; caution is required during distillation of solvent from $LiAlH_4$.[7,8]

Dioxane. Explosion occurred during distillation of this solvent from $LiAlH_4$; nitrogen atmosphere is necessary for this reaction.[7]

Ethyl Acetate. Explosion occurred when ethyl acetate was used to destroy excess $LiAlH_4$; either a solution of ethyl acetate in an inert solvent or a moist unreactive solvent should be used.[9]

Fluoroamides. Reduction with $LiAlH_4$ is hazardous at all stages.[10,11]

Pyridine. Reaction is highly exothermic.[12]

Tetrahydrofuran. Addition of $LiAlH_4$ to dried tetrahydrofuran, which had been stored over calcium hydride, caused explosion.[13]

Water. Vigorous reaction with ignition of liberated H_2.[12]

Physiological Properties and Health Hazards

Reaction with moisture forms corrosive lithium hydroxide, which irritates the skin and eyes. Avoid contact with skin, eyes, and clothing.[14] TLV-TWA 2 mg(Al)/m^3.[15]

Spillage Disposal

Instruct others to maintain a safe distance. Wear face shield and goggles, laboratory coat, and butyl rubber gloves. Cover the spill with dry sand. Scoop the resulting mixture into a pail and, in the fume hood, slowly add to an excess of butanol (10 mol:1 mol, 24 mL/g hydride). When the reaction

is complete, dilute with water, and allow the solids to settle. Flush the solution down the drain with at least 50 times its volume of water. The solid residue may be discarded with normal refuse.[16]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear face shield and goggles, laboratory coat, and butyl rubber gloves. In the fume hood, slowly and cautiously add the lithium aluminum hydride to a large excess of butanol in a pail (about 24 mL/g of lithium aluminum hydride). When the reaction is complete, dilute the mixture with water and allow the solids to settle. Wash the liquid into the drain. The solid residue may be discarded with normal refuse.[16]

Reaction Mixtures. Wear butyl rubber gloves, laboratory coat, and eye protection. In the fume hood behind a shield, cautiously and slowly carry out the following procedure. Treat the stirred reaction mixture from n grams of lithium aluminum hydride by successive dropwise addition of n mL of H_2O, n mL of 15% sodium hydroxide solution and 3n mL of H_2O. This produces a granular precipitate that is removed by filtration and treated as normal refuse. Wash the filtrate into drain.[17]

Reactions for Spillage and Waste Disposal

$$LiAlH_4 + 4CH_3(CH_2)_3OH \rightarrow CH_3(CH_2)_3OLi + Al(O(CH_2)_3CH_3)_3 + 4H_2$$

$$LiOCH_2CH_2CH_2CH_3 + H_2O \rightarrow LiOH + CH_3CH_2CH_2CH_2OH$$

$$Al(O(CH_2)_3CH_3)_3 + 3H_2O \rightarrow \underset{\substack{\text{aluminum}\\ \text{hydroxide}\\ \text{(insoluble)}}}{Al(OH)_3} + 3CH_3(CH_3)_3OH$$

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LITHIUM HYDRIDE LiH

FLAMMABLE, CORROSIVE

Physical Properties

White to blue-gray powder or crystals; darkens rapidly on exposure to light; mp, 680°C.[1,2] Decomposes at about 850°C.[2]

Fire Hazard

Flammable; severe fire hazard; may spontaneously ignite with moisture in air.[2] Powdered material burns readily on exposure to air.[3]

Chemical Properties

Rapidly decomposes in water to form lithium hydroxide and hydrogen. Reacts with the lower alcohols, carboxylic acids, chlorine, and ammonia at 400°C to liberate hydrogen. Reducing agent. Condensing agent with ketones and acid esters. Dessicant.[1] Insoluble in benzene and toluene.[2]

Hazardous Reactions

Ignites in moist air.[2]

Dinitrogen Oxide. Spontaneous ignition occurs when nitrous oxide and lithium hydride are mixed.[4]

Oxygen (gas). Ignites in moist air or oxygen.[5]

Oxygen (liquid). Mixtures of powder with liquid oxygen are detonatable explosives of greater power than TNT.[6]

Water. Violently decomposes with water.[2]

Physiological Properties and Health Hazards

Corrosive to the eyes, skin, and mucous membranes.[7] TLV-TWA 0.025 mg/m^3.[8]

Spillage Disposal

Wearing butyl rubber gloves, fireproof clothing, face shield, and goggles; cover spill with sand.[8] Transfer mixture into a dry plastic bag filled in advance with an inert gas. Package and label for disposal by incineration.[2] Alternatively, in the fume hood, add butanol slowly to the solid mixture until the reaction ceases. Then carefully add water until all the hydride is destroyed. Let stand until solid settles. Wash the liquid into the drain. Discard solid residue with normal refuse.[8,9]

Waste Disposal

1. Package and label for disposal by burning.[2]
2. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Mix with sand. Slowly and cautiously add to butanol until the reaction ceases. Then carefully add water until all of the hydride is destroyed. Let stand until solids settle. Decant liquid into drain. Discard solid residue with normal refuse.[8,9]

Reactions for Spillage and Waste Disposal

$$LiH + CH_3CH_2CH_2CH_2OH \rightarrow \underset{\text{lithium butoxide}}{LiOCH_2CH_2CH_2CH_3} + H_2$$

$$LiOCH_2CH_2CH_2CH_3 + H_2O \rightarrow \underset{\substack{\text{lithium}\\\text{hydroxide}\\\text{(insoluble)}}}{LiOH} + CH_3CH_2CH_2CH_2OH$$

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LOMUSTINE $C_9H_{16}ClN_3O_2$

ANTINEOPLASTIC

Synonyms

N-(2-Chloroethyl)-N'-cyclohexyl-N-nitrosourea, 1-(2-chloroethyl)-3-cyclohexyl-nitrosourea.[1]

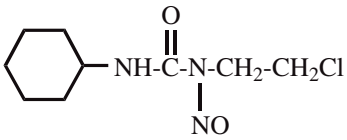
Physical Properties

Yellow powder; mp, 90°C.

Chemical Properties

Sensitive to oxidation and hydrolysis; forms alkylating and carbamoylating intermediates. Half-life of 117 minutes at 25°C and neutral pH.[2] Solubility: below 0.05 mg/mL in water, 0.1 N sodium hydroxide, 0.1 N hydrochloric acid or 10% ethanol; 70 mg/mL in absolute ethanol.[1]

Structure



Physiological Properties and Health Hazards

LD_{50} orally, intraperitoneal, subcutaneous in male mice: 51, 56, 61 mg/kg, respectively.[1] Adverse effects in patients including nausea and vomiting, stomatitis, renal toxicity, and impaired bone marrow functions.[3] Reasonably anticipated to be a human carcinogen.[4]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of lomustine in the spilled liquid and add 20 mL of detergent solution for each 100 mg of lomustine. The resulting mixture is heated at 55°C for 90 minutes. Wash the solution into the drain with water. The synthetic detergent solution (100 mL) is prepared by dissolving 4.8 g of sodium pyrophosphate in 100 mL of water and adding 1 mL of Triton X100.[5]

Waste Disposal

Wear goggles and protective gloves and clothing. Slowly add 20 mL of synthetic detergent to lomustine (100 mg) contained in a 125-mL Erlenmeyer flask equipped with a magnetic stirrer. Heat the mixture at 55°C for 90 minutes. The cooled solution can be safely flushed into the drain with water.[5]

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2,6-LUTIDINE C_7H_9N

FLAMMABLE, TOXIC

Synonyms

2,6-Dimethylpyridine.

Physical Properties

Oily liquid; mp, –5.8°C; bp, 144°C.[1]

Fire Hazard

Flammable.[2]

Chemical Properties

Soluble in water, alcohol, and ether. Miscible with dimethylformamide and tetrahydrofuran.[1]

Physiological Properties and Health Hazards

Toxic.[2]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl[10] rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the lutidine has been absorbed, scoop the mixture into a plastic pail and add enough water to dissolve the sodium carbonate. Allow the solids to settle and decant the liquid to another container. Discard the solids with normal refuse. To the liquid, slowly add (frothing will occur) 6 M sulfuric acid to pH 2. Stir into the acidified solution sufficient solid potassium permanganate so that the liquid is purple (a drop of the liquid on a filter paper will show a purple ring). Allow the mixture to stand at room temperature for 48 hours, and then neutralize with solid sodium carbonate (frothing will occur), or with a 10% aqueous solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[3]

Waste Disposal

Package Lots. Distill for reuse or place in a separate labeled container for disposal by burning in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear eye protection, laboratory coat, and rubber gloves. Dissolve the 2,6-lutidine (1 mL) in 50 mL of 3 M sulfuric acid (prepared by slowly adding 8 mL of concentrated sulfuric acid to 21 mL of water). Weigh 10 g of potassium permanganate and stir small portions of the solid into the 2,6-lutidine solution over a period of about 1 hour. Stir the mixture at room temperature for 48 hours, and then neutralize the solution by adding solid sodium carbonate or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until solution is colorless. Decant the clear liquid into the drain and discard any brown solid with regular refuse.[3]

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MAGNESIUM Mg

EASILY IGNITED

Physical Properties

Silvery-white metal; slowly oxidizes in moist air. Available as bars, turnings, wire, and powder; mp, 651°C; bp, 1100°C.[1]

Fire Hazard

Powder is easily ignited. Use class D extinguisher or smother with dry sand. Do not use water or carbon dioxide.[2]

Chemical Properties

Powerful reducing agent that undergoes violent or explosive reaction with a variety of oxidants, especially when powdered.[2,3]

Hazardous Reactions

Air. Ignites in air[1]; powder forms explosive mixtures with air that may be ignited with a spark.[3] CO_2 supports the combustion of Mg.[4]

Halocarbons. A vigorous and sometimes explosive reaction occurs on contact of Mg powder with chloromethane, chloroform, or carbon tetrachloride; with carbon tetrachloride or trichloroethylene, heavy impact causes ignition; with 1,1,1-trichloroethane, violent decomposition occurs.[5]

Halogens. If moist, ignites in fluorine and chlorine and, if finely divided, in iodine vapor.[6]

Hydrogen Iodide. Contact causes momentary ignition.[7]

Metal Oxides. Violently reduces metal oxides on heating.[8,9]

Metal Oxosalts. Interacts vigorously with fused ammonium nitrate, metal nitrates, phosphates, or sulfates, and lithium and sodium carbonates.[10]

Methanol. Reaction may become vigorous; sufficient methanol must be present to dissipate heat of reaction; mixtures of powdered Mg with methanol can be detonated.[11]

Potassium Carbonate. Heating a mixture can be hazardous.[12]

Physiological Properties and Health Hazards

Avoid direct viewing of magnesium fires as eye injury may result.[3] Inhalation of the dust is irritating.[1]

Spillage Disposal

Magnesium Powder. Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a pail and, in the fume hood, slowly add to a large volume of butanol. When the reaction is complete, allow the solids to settle. Wash the solution into the drain. The solid residue may be discarded with normal refuse.[13]

Magnesium Turnings. Wear nitrile rubber gloves, laboratory coat, and eye protection. Sweep into a container, and, in the fume hood, add to a pail of water. Slowly add dilute hydrochloric acid dropwise until reaction begins. When reaction is complete, wash into the drain.[13]

Waste Disposal

Package Lots. Reuse or recycle.[3]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Slowly add 10 g (0.42 mol) of magnesium to water (0.5 L). Carefully add 5% hydrochloric acid until reaction begins. Let stand until reaction ceases. If further addition of acid has no effect, then wash the solution into the drain with at least 50 times its volume of water.[13]

Reactions for Spillage and Waste Disposal

$$Mg + 2H_2O + H^+ \rightarrow \underset{\substack{\text{magnesium}\\\text{hydroxide}\\\text{(insoluble)}}}{Mg(OH)_2} + H_2 + H^+$$

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MAGNESIUM PERCHLORATE $Mg(ClO_4)_2$

IRRITANT, AVOID CONTACT WITH ACIDS

Synonyms

Anhydrone, dehydrite.

Physical Properties

White hygroscopic powder. Dissolves in water with evolution of considerable heat.[1]

Fire Hazard

Powerful oxidizing material; forms explosive mixtures with combustible organic or other easily oxidizable materials. Such mixtures are ignited readily by friction or heat. To fight fire, flood with water.[2]

Chemical Properties

Used as a drying agent for gases. Unsuitable for drying organic solvents.[1]

Hazardous Reactions

Contact with acidic materials must be avoided.[3]

Arylhydrazine. Explosion followed the use of the anhydrous salt to dry an ethereal solution of arylhydrazine.[3]

Cellulose, Dinitrogen Tetroxide, and Oxygen. An explosion occurred in a drying tube containing the desiccant between cotton wool pads that had been used for drying an O_2/N_2O_4 mixture.[3]

Dimethyl Sulfoxide. Explosion occurred during distillation of dimethyl sulfoxide from the drying agent.[4]

Hydrogen Fluoride. Violent explosion resulted from the drying of fluorobutane due to the presence of HF.[5]

Organic Materials. Explosions have followed the use of the anhydrous salt for drying organic solvents and solutions of organic compounds in these solvents.[3] Use of perchlorate in a drybox where there is a danger of contamination by organic compounds can be dangerous.[6]

Physiological Properties and Health Hazards

Dust can cause irritation of the skin and mucous membranes.[1]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into container and transfer to fume hood. Carefully add to a pail of water and dilute to a concentration of less than 0.1%. When solids have settled, wash liquid into drain. Discard solid residue with normal refuse.[7–9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Slowly add perchlorate to large excess of water. Dilute to a concentration of less than 0.1%, and then wash down drain with at least 50 times its volume of water.[9,10]

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MALATHION $C_{10}H_{19}O_6PS_2$

INSECTICIDE

Synonyms

[(Dimethoxyphosphinothioyl)thio]butanedioic acid diethyl ester, mercaptosuccinic acid diethyl ester, S-ester with O,O-dimethyl phosphorothioate, ENT 17034, Prioderm®.

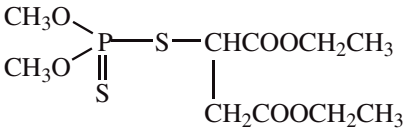
Physical Properties

Deep brown to yellow liquid, mp, 2.9°C; bp, 156–157°C (0.7 mm). Characteristic odor.[1]

Chemical Properties

Solubility in water, 145 ppm. Limited solubility in petroleum oils but miscible with most organic solvents. Light petroleum (30–60 is soluble in malathion to the extent of 35%.[2] Rapidly hydrolyzed at pH above 7.0 but stable in aqueous solution buffered at pH 5.26.[2] Reacts with heavy metals, especially iron.[1]

Structure



Malathion

Physiological Properties and Health Hazards

LD_{50} (oral rat) 1375 mg/kg. LD_{50} (dermal rabbit) 4100 mg/kg.[2] LC_{50} (rainbow trout, 96 hours) 170 µg/L.[3] Highly toxic. Acute effects include anorexia, nausea, vomiting, diarrhea, excessive salivation, pupillary constriction, broncho constriction, muscle twitching, convulsions, coma, and respiratory failure.[2] TLV-TWA 10 mg/m^3.[4]

Formulations

Emulsifiable concentrate, liquid, and dust.

Waste Disposal

Residues of malathion can be degraded to nonhazardous products using aqueous acidic potassium permanganate solution. Thus, to each 1 mL of commercial malathion solution, add 50 mL of 3 M sulfuric acid (8.5 mL of concentrated sulfuric acid added to 41.5 mL of water) and 3 g of potassium permanganate. Stir the mixture at room temperature for 5 hours. Neutralize the solution by careful addition of soda ash, and then decolorize by adding, while stirring, a saturated solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless solution is formed. Wash the clear solution into the drain.[5]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder

contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer by running the machine through a complete wash cycle with detergent (but without clothing) before washing other clothes.

Emulsifiable concentrate formulation of malathion, diluted to field strength (0.5% a.i.) was used to contaminate 100% cotton. A single warm water wash (50°C) results in 6% malathion residue remaining on the fabric. A double wash results in approximately 3% residue remaining. Using a bleach/soak pretreatment, a prewash soil and stain remover, or an additional wash does not significantly improve residue removal.

Do not wash clothing that has been contaminated with concentrated malathion, because approximately 30% remains after one wash. It should be placed in a plastic bag and taken to an approved landfill site.[6]

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MALEIC ANHYDRIDE $C_4H_2O_3$

EYE IRRITANT

Physical Properties

White needles from chloroform, or lumps; mp, 53°C.[1]

Fire Hazard

Gives off flammable vapors when molten. Dust cloud may be exploded by flame or a spark. Use water, dry chemical, or carbon dioxide to extinguish.[2]

Chemical Properties

Dissolves in water, forming maleic acid. Very soluble in acetone, ethyl acetate, chloroform, benzene, toluene, and xylene; soluble in dioxane; slightly soluble in carbon tetrachloride and ligroin.[1]

Hazardous Reactions

Cations or Bases. Decomposes exothermally in the presence of alkali- or alkaline earth-metal or ammonium ions, dimethylamine, triethylamine, or pyridine or quinoline at temperatures above 150°C.[3]

Physiological Properties and Health Hazards

The dust and vapor irritate the eyes, skin, and respiratory system. Swallowing causes internal irritation and damage. Prolonged exposure to skin may cause burns. Avoid breathing dust. Avoid contact with skin and eyes.[4] TLV-TWA 0.1 ppm (1 mg/m^3).[5]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, goggles, and a face shield. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Wash the solution into the drain. Treat the solid residue as normal refuse.[6]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into a furnace equipped with afterburner and scrubber.[7]

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7. Ald 2183D.
8. ITI 387.

MECHLORETHAMINE HYDROCHLORIDE $C_5H_{12}Cl_3N$

ANTINEOPLASTIC

Synonyms

Caryolysine, Cloramin, Dichloren, Embichen, Erasol, Mustargen hydrochloride, Mustine hydrochloride, Nitrogranulogen.

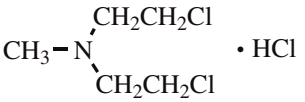
Physical Properties

Hygroscopic leaflets from acetone or chloroform; mp, 109–111°C.[1]

Chemical Properties

Very soluble in water; soluble in alcohol. The dry crystals are stable at temperatures up to 40°C.[1]

Structure



Mode of Action

This nitrogen mustard is a biological alkylating agent that inhibits replication and transcription of DNA by forming cross-links between two molecules of nucleoprotein. DNA miscoding and breakage may also occur.[2]

Physiological Properties and Health Hazards

Mechlorethamine is very irritating to mucous membranes and is especially harmful to the eyes. In patients, it causes nausea and vomiting, thromobophlebitis, granulocytopenia, thrombocytopenia, stomatitis, maculopapular skin eruption, herpes zoster, amenorrhea, azoospermia, thrombosis, bone marrow depression, tinnitis, vertigo, and hearing loss.[2] LD_{50} (i.v., rat) 1.1 mg/kg.[1]

Waste Disposal

Deactivate any unused solution by mixing with an excess of a solution containing equal volumes of 5% sodium thiosulphate and 5% sodium bicarbonate. Allow the solution to stand for 45 minutes and wash into the drain with water. Treat glassware, tubing, and other items similarly.[3]

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3. Bacovsky, R., *Can. J. Hosp. Pharm.*, 34, 13, 1980.

6-MERCAPTOPURINE $C_5H_4N_4S$

ANTINEOPLASTIC

Synonyms

6-Purinethiol, 6 MP, Leukerin, Mercaleukin, Purinethiol.

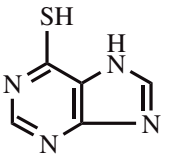
Physical Properties

Monohydrate; yellow prisms form from water. Becomes anhydrous at 140°C; dec, 313–314°C.[1]

Chemical Properties

Insoluble in water, acetone, and ether. Soluble in hot ethanol and in alkaline solutions with slow decomposition.[1]

Structure



Mode of Action

This compound is a purine antagonist that inhibits RNA and DNA synthesis.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include anorexia, nausea, vomiting, gastrointestinal ulceration, skin eruptions, bone marrow depression, reversible cholestatic jaundice, hepatic necrosis, dermatitis, and drug fever.[2] LD_{50} (i.p., mouse) 157 mg/kg; (i.p., hamster) 364 mg/kg.[1]

Waste Disposal

Wear rubber gloves, goggles, and protective clothing. Work in the fume hood. Dissolve 6-mercaptopurine (40 mg) in 80 mL of 3 M sulfuric acid (13 mL of concentrated sulfuric acid slowly added to 67 mL of cold water). While stirring, add 0.5 g of potassium permanganate in small portions over a period of about 10 minutes. Stir the mixture at room temperature for 12 hours and neutralize by the careful addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, saturated aqueous sodium bisulfite solution until a colorless liquid is formed. Wash into the drain with water.[3,4]

To 1 mL of an aqueous solution containing 2–3 mg of mercaptopurine, add 25 mL of household bleach. Allow the mixture to stir at room temperature for 90 minutes. When reaction is complete, wash the solution into the drain with water.[4]

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1. Merck 5896.

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4. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 104.

MERCURY Hg

GIVES OFF VERY POISONOUS VAPOR, DANGER OF CUMULATIVE EFFECTS

Synonyms

Liquid silver, quicksilver.

Physical Properties

Heavy silvery, mobile liquid metal; mp, –39°C; bp, 357°C.[1]

Chemical Properties

Insoluble in water.[1]

Fire Hazard

Ammonia. Prolonged contact may yield explosive solid; use of mercury manometers with NH_3 should be avoided.[2]

Bromine. Reacts violently with dry bromine.[3]

Metals. Ease with which Hg forms alloys with many laboratory and electrical contact metals can cause severe corrosion problems.[4]

Peroxyformic Acid. Readily causes explosions when added to 90% acid.[5]

Physiological Properties and Health Hazards

High concentrations of vapor may cause metallic taste, nausea, abdominal pain, vomiting, diarrhea, and headache. Prolonged exposure to small concentrations of vapor may result in severe nervous disturbance, including tremor of the hands, insomnia, loss of memory, irritability, and depression; other possible effects are loosening of teeth and excessive salivation. Continued skin contact with mercury may cause dermatitis and the above effects may be caused by absorption through the skin or following ingestion. Kidney damage may ensue. Avoid breathing vapor. Avoid contact with eyes and skin.[6] TLV-TWA 0.025 mg/m^3.[7]

Spillage Disposal

Because of the high toxicity of mercury vapor, it is important to clean up mercury as thoroughly as possible, especially in confined areas. Wear nitrile rubber gloves, laboratory coat, and eye protection. A self-contained breathing apparatus will be necessary for large spills. A small aspirator with a capillary tube and connected to a pump can be used for collecting droplets. For fine drops of mercury in inaccessible sites, treat with Merconvap[8] or use a kit designed for the purpose.[9] Combine collected mercury spills in a tightly stoppered, thick-walled, high-density polyethylene bottle and send to central department for reclamation or return to manufacturer.[9]

Waste Disposal

CAUTION: The toxicity of mercury is such that the element and its compounds should not be allowed to contaminate air or water. Wear nitrile rubber gloves, self-contained breathing apparatus, and laboratory coat. Collect all droplets and pools at once by means of suction pump and aspirator bottle with a long capillary tube. Cover fine droplets in nonaccessible cracks with Merconvap[8] or use a kit designed for the purpose.[9] Combine all contaminated mercury in a tightly stoppered bottle. Return to manufacturer for recycling.[9]

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1. Merck 5925.
2. Sampey, J., *Chem. Eng. News,* 25, 2138, 1947; *J. Chem. Educ.,* 44, A324, 1967.
3. B 101.
4. B 1218.
5. D'Ans, J. et al., *Chemische Berichte*, 48, 1136, 1915.
6. Lux 465.
7. ACGIH 39.
8. Merconvap, EPS Chemicals Inc., P.O. Box 570, Point Roberts, WA 98281-9602; Ross Chempharma Inc., No. 6, 1520 Cliveden Avenue, Delta, BC, V3M 6J8, Canada.
9. PPL 89.

MERCURY COMPOUNDS Hg^{+}, Hg^{2+}

POISONOUS DUST; SERIOUS RISK OF POISONING BY INHALATION, SWALLOWING, OR SKIN CONTACT; DANGER OF CUMULATIVE EFFECTS; CAUSES IRRITATION OF SKIN AND EYES

Mercury compounds vary widely in appearance, solubility in water, and toxicity. Mercuric compounds are generally more toxic than mercurous compounds. Some organic mercurial compounds are liquids with an extremely poisonous vapor, and others are solids, the toxicity of which is not known with certainty. Thus, phenylmercuric acetate appears to be relatively nontoxic but the alkyl mercurials are highly poisonous. Their toxicity by skin absorption is uncertain, but some, such as ethyl mercury phosphate and mercury fulminate, can cause dermatitis. The effects vary greatly according to the nature of the organic mercurial. Some compounds cause kidney damage while others can cause irreversible damage to the central nervous system.[1]

Hazardous Reactions

Mercuric Azide $Hg(N_3)_2$

Explosively unstable.[2,3]

Mercuric Chlorite $Hg(ClO_2)_2$

Explosive salt.[4]

Mercuric Nitrate $Hg(NO_3)_2$

Acetylene. Contact with acetylene gives explosive mercury(II) acetylide.[5]

Ethanol. In ethanol, mercury(II) fulminate may be formed.[5]

Petroleum Hydrocarbons. Risk of violent reaction with petroleum hydrocarbons.[6]

Phosphine. Aqueous solution reacts with phosphine to give explosive complex.[5]

Mercuric Oxide HgO

Hydrazine. Explosive reaction with hydrazine hydrate.[7]

Mercuric Sulfate $HgSO_4$

Hydrogen Chloride. Absorption of gaseous hydrochloric acid becomes violent at 125°C.[8]

Mercurous Azide $Hg_2(N_3)_2$

Explodes in air on heating at 270°C.[2,9]

Mercurous Oxide Hg_2O

Hydrogen Peroxide. Reacts explosively with hydrogen peroxide.[10]

Mercury Halides HgX_2, Hg_2X_2

Potassium. Mixtures of mercuric bromide, mercuric chloride, mercuric fluoride, mercuric iodide, or mercurous chloride with potassium explode on impact.[11]

Physiological Properties and Health Hazards

Inhalation of dust may cause nausea, abdominal pain, vomiting, diarrhea. and headache. Skin absorption may give rise to similar effects and the eyes may be damaged by direct contact with some salts. Abdominal pain, nausea, vomiting, diarrhea, and shock follow ingestion of the soluble mercuric salts. Kidney damage may ensue. The intake of small amounts of mercury compounds by inhalation, skin absorption, or ingestion over a long period may cause nervous disturbance, including hand tremors, insomnia, memory loss, irritability, and depression; other possible effects are loosening of the teeth and excessive salivation. Avoid breathing dusts. Avoid contact with skin and eyes.[1] TLV-TWA 0.025 mg/m^3.[12]

Spillage Disposal

See waste disposal.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal in a special hazardous waste site.

Small Quantities.

> CAUTION: The toxicity of mercury is such that the element and its compounds should not be allowed to contaminate air or water. Wear eye protection, nitrile rubber gloves, and laboratory coat. Work in the fume hood. Dissolve the waste mercury salts (10 g) as far as possible in water (100 mL). Adjust the solution to pH 10 with 10% sodium hydroxide solution. Add 20% sodium sulfide solution while stirring until no further precipitation occurs. Withdraw a small sample of supernatant liquid and add sodium sulfide solution to check that precipitation is complete. Allow the precipitate to settle, and then decant or filter the supernatant liquid into the drain with at least 50 times its volume of water. Package the dry mercuric sulfide for disposal in a secure landfill.[13–15]

Reactions for Spillage and Waste Disposal

$$Hg^{2+} + Na_2S \rightarrow \underset{\substack{\text{mercurous}\\\text{sulfide}\\\text{(insoluble)}}}{HgS} + 2Na^+$$

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4. NFPA 491M-127; Levi, G.R., *Gaz. Chim. Ital*., 45, 161, 1915.
5. Mellor, Vol. 4, 933, 1940.
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7. Mellor, Vol. 4, 778, 1940; Vol. 8, 318, 1940.
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14. PPL 167.
15. Ald 2217–2220.

METHANOL CH_3OH

HIGHLY FLAMMABLE, SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING

Synonyms

Methyl alcohol, methyl hydrate, wood alcohol.

Physical Properties

Colorless, volatile liquid; bp, 65°C.[1]

Fire Hazard

Flash point, 11°C; explosive limits, 6–36%; ignition temperature, 464°C. Extinguish fire with water spray, dry powder, carbon dioxide, or vaporizing liquid.[2]

Chemical Properties

Miscible with water, ethanol, ether, benzene, ketones, and most other organic solvents.[1]

Hazardous Reactions

Acetyl Bromide. Interaction is violent with HBr being evolved.[3]

Air. Methanol–air mixtures at 1.81 bar and 120°C may explode with or without the addition of oxygen and water.[4]

Alkylaluminum Solutions. Methanol reacts violently with dilute alkylaluminum solutions.[4]

Beryllium Hydride. Reaction with the ether-containing hydride is violent, even at −196°C.[5]

Cyanuric Chloride. Cyanuric chloride dissolved in methanol reacted violently and uncontrollably with the solvent.[6]

Metals. Reaction with Mg is vigorous and often subject to a lengthy induction period. Mixtures with powdered Mg or Al are capable of powerful detonation.[7] Reaction with K may lead to an explosion. Methanol should not be substituted for *tert*-butanol in the disposal of waste K.[8]

Methylene Chloride. Methylene chloride may become flammable in the presence of small amounts of methanol.[9]

Oxidants. Methanol may react violently with oxidizing agents.[4]

Phosphorus(III) Oxide. Reacts very violently with the liquid oxide (above 24°C).[10]

Potassium *tert*-Butoxide. Reaction of liquid with the solid butoxide causes ignition after 2 minutes.[11,12]

Water. A 40:60 mixture of methanol and water at 30°C and 30:70 mixture of methanol and water can be ignited by a static discharge.[12]

Physiological Properties and Health Hazards

Vapor irritates mucous membranes and may cause headache, nausea, and vomiting. Breathing high concentrations of vapor may cause giddiness, stupor, and stomach upset. The vapor and the liquid can cause severe eye damage, sometimes several hours after exposure. Swallowing the liquid or prolonged exposure to low vapor concentrations may cause temporary or permanent blindness and damages the central nervous system, the kidneys, liver, and heart, eventually resulting in death. Avoid breathing vapor. Avoid contact with skin and eyes.[9] TLV-TWA 200 ppm (262 mg/m^3); TLV-STEL 250 ppm (328 mg/m^3).[13]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to maintain a safe distance. Wear breathing apparatus if necessary, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (bentonite), and sand. Scoop into a container. If local regulations permit, add the solid to a pail of water, allow to stand until the solids settle, decant the liquid to the drain and treat the solid as normal refuse. Otherwise, package the solid and label for disposal by burning.[9,14]

Waste Disposal

Place waste methanol in nonhalogenated waste solvent container labeled for disposal by burning.[15]

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15. ITI 398.

METHOTREXATE $C_{20}H_{22}N_8O_5$

ANTINEOPLASTIC

Synonyms

Amethopterin, emtrexate, methylaminopterin, MTX, Cl-14377.

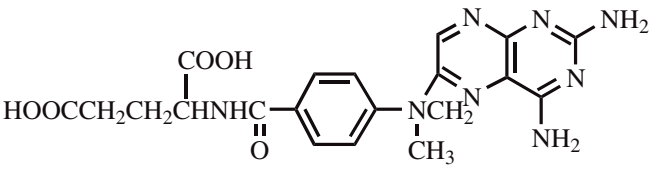
Physical Properties

Monohydrate, yellow crystals from dilute HCl; dec, 185–204°C.[1]

Chemical Properties

Soluble in alkaline solutions with decomposition.[1] Practically insoluble in water and alcohol.[2]

Structure



Mode of Action

Methotrexate is a folic acid antagonist, binding reversibly to dihydrofolate reductase. This prevents purine/pyrimidine biosynthesis and DNA synthesis.[3]

Physiological Properties and Health Hazards

Adverse effects in patients include ulcerative stomatitis, leukopenia, nausea, and abdominal distress.[2] LD_{50} (i.v., rat) 14 mg/kg.[1]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of methotrexate in the spilled liquid. Add a solution of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate per 100 mL to the absorbing mixture, allowing 20 mL of solution for each 50 mg of methotrexate. Allow to stand overnight, and then neutralize by careful addition of soda ash or 10% sodium hypochlorite solution. Decant the liquid into the drain with water; discard the residue as normal refuse.[2,4,5]

Waste Disposal

Wear goggles, rubber gloves, and protective clothing. Work in the fume hood. Add the methotrexate (80 mg) to 100 mL of household bleach (5% sodium hypochlorite) in a 200-mL beaker. Allow to stand at room temperature for 30 minutes. Wash the liquid into the drain with water. Immerse vials and glassware that contain residues of methotrexate in bleach for at least 30 minutes, and then rinse thoroughly with water and discard.[4,5]

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3-METHYLCHOLANTHRENE $C_{21}H_{16}$

SUSPECTED CANCER AGENT

Synonyms

3-MC, 3-MECA.

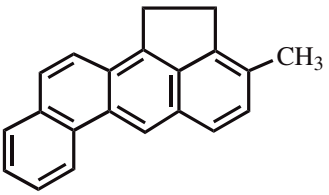
Physical Properties

Pale yellow, slender prisms from benzene and ether; mp, 179–180°C; bp_{80}, 280°C.[1]

Chemical Properties

Insoluble in water; soluble in benzene, xylene, and toluene; slightly soluble in amyl alcohol.[1]

Structure



Hazardous Reactions

Can react with oxidizing materials.[2]

Physiological Properties and Health Hazards

A powerful irritant.[2] Carcinogenic in rodents. Mutagenic in the Ames test and in mice.[3]

Spillage Disposal

Wear protective gloves, laboratory coat, and goggles. Turn off ventilation and isolate spill area. Add enough dimethylformamide to completely wet the spilled 3-methylcholanthrene, and then pour over the area an excess of a freshly prepared solution containing 4.7 g of potassium permanganate per 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow to react for at least 1 hour. Absorb the residual solution on a 1:1:1 mixture of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container, and in the fume hood, slowly add it to a beaker of cold water. If necessary, neutralize the solution with soda ash. Decant or filter the liquid into the drain with a large volume of water. Discard the solid as normal refuse.[4,5]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix the material into a combustible solvent and burn in a furnace equipped with afterburner and scrubber.[6]

Small Quantities. Wear protective gloves, laboratory coat, and goggles. Work in fume hood. Dissolve the 3-methylcholanthrene (5 mg) in acetone (2 mL). Add a freshly prepared solution of potassium permanganate (0.5 g) in 10 mL of 3 M sulfuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water). Swirl, and allow to stand at room temperature for at

least 1 hour. If necessary, add more potassium permanganate solution to maintain a purple color. Cautiously neutralize with soda ash or 10% aqueous sodium hydroxide, and then add, while stirring, a saturated aqueous solution of sodium bisulfite until a clear liquid is formed. Wash into the drain with a large volume of water.[4,5]

REFERENCES

1. Merck 6072.
2. Sax 2289.
3. Becker, F.F., Ed., *Etiology: Chemical and Physical Carcinogens, Cancer 1,* 2nd ed., Plenum Press, New York, 1982, p. 310.
4. Castagnero, M., et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Polycyclic Aromatic Hydrocarbons,* No. 49, IARC Scientific Publications, Lyon, 1983, p. 19.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.
6. Ald 2319D.

METHYLDICHLOROSILANE CH_3SiHCl_2

FLAMMABLE, CORROSIVE

Synonyms

Dichloromethylsilane.

Physical Properties

Colorless liquid; bp, 41°C.[1]

Fire Hazard

Flammable.[2] Dangerous when exposed to heat or flame.[1] Flash point, –26°C; auto-ignition temperature, 316°C; flammable limits, 6–55%.[3]

Chemical Properties

Soluble in benzene, ether, and heptane.[1]

Hazardous Reactions

Pure material does not ignite on impact.

Oxidizing Agents. Ignites in presence of potassium permanganate or trilead tetroxide,[4] or by copper or silver oxides, even under an inert gas.[5]

Physiological Properties and Health Hazards

Corrosive.[2] Vapor highly irritating to the skin, eyes, and mucous membranes.[1]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Slowly add the solid to a pail of cold water. Neutralize the solution, if necessary, with sodium carbonate. Wash the liquid into the drain. Treat the solid as normal refuse.[6,7]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Add 500 mL of water to a 1-L, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Cool the flask in an ice-water bath. Place 10 mL of methyldichlorosilane in the funnel and add dropwise to the stirred water. Do not allow the temperature to rise above 20°C. When addition is complete, neutralize the solution with sodium carbonate and wash into the drain.[6,7]

Reactions for Spillage and Waste Disposal

$$CH_3SiHCl_2 + 2H_2O \rightarrow \underset{\text{methylsilanol}}{CH_3SiH(OH)_2} + 2HCl$$

REFERENCES

1. Sax 1152.
2. Aldrich 554.

3. NFPA 49.
4. Mueller, R. et al., *J. Prakt. Chem.,* 31, 1, 1966.
5. B 170.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.
7. Havill, M.E., Joffe, I., and Post, H.W., *J. Org. Chem.*, 13, 280, 1948.

4,4'-METHYLENE-*BIS*-(2-CHLOROANILINE) $(H_2NC_6H_3Cl)_2CH_2$

ANIMAL CARCINOGEN

Synonyms

3,3'-Dichloro-4,4'-diaminophenylmethane, MBOCA.

Physical Properties

Flakes from alcohol; mp, 110°C.[1]

Chemical Properties

Slightly soluble in water. Soluble in dilute acids, ether, and alcohol.[1]

Physiological Properties and Health Hazards

Animal carcinogen.[1,2] Reasonably anticipated to be a human carcinogen.[3] TLV-TWA 0.01 ppm (0.01 mg/m^3).[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[5]

Small Quantities. To 9 mg of 4,4'-methylene-*bis*-(2-chloroaniline), add 10 mL of 0.1 M hydrochloric acid (prepared by slowly adding 1 mL of concentrated acid to 119 mL of cold water). Mix to dissolve. Add 5 mL of 0.2 M potassium permanganate solution (0.3 g of solid potassium permanganate dissolved in 10 mL of water) and 5 mL of 2.0 M sulfuric acid (prepared by cautiously adding 1 mL of concentrated acid to 8 mL of cold water). Mix and let stand overnight (at least 10 hours). Decolorize if necessary with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (20 g of NaOH dissolved in 100 mL of cold water). Wash the remaining solution into the drain.[6]

REFERENCES

1. Merck 6084.
2. Sax 2308.
3. NIEHS III–148.
4. ACGIH 41.
5. PPL 162.
6. Cas 64, 24.

METHYL FLUOROSULFONATE FSO_2OCH_3

EXTREMELY TOXIC VAPOR, CORROSIVE MUTAGEN

Synonyms

Magic Methyl®.

Physical Properties

Volatile liquid; bp, 92–94°C.[1]

Chemical Properties

Decomposes to fumes of F and SO_x when heated. Reacts with water, steam, and acids to give toxic and corrosive fumes.[2] Powerful methylating agent.[3,4]

Physiological Properties and Health Hazards

Corrosive.[4] Vapor causes severe irritation of the respiratory system with possible severe lung injury and death after a latent period.[5] Vapor and liquid irritate or burn the eyes resulting in temporary or permanent eye damage.[6] The vapor or liquid may blister the skin and skin absorption may result in severe poisoning after latent period. Prevent inhalation of vapor. Prevent contact with skin and eyes. Mutagen in Ames test.[7] Possible carcinogen.[9,10] LD_{50} (mice) 112 mg/kg; LD_{50} (rats) 0.025–0.029 mg/kg. Mean irritation index (rabbits) 8.0.[7]

Spillage Disposal

All personnel should leave the area immediately. Entrance into the spillage area should be made only by personnel wearing a self-contained, full-face breathing apparatus; long-sleeved rubber gloves; goggles; and protective clothing (nonporous). The spill should be treated with excess ammonia solution and then mopped up and run down the drain with at least 50 times its volume of water. Ventilate area. Labware should be completely immersed in an ammonia solution and allowed to stand overnight before removal from hood working area. Contaminated clothing should be removed promptly and placed in a well-ventilated area, and the affected areas of the skin should be washed immediately with copious quantities of water. Contaminated clothing should then be treated with a basic solution and discarded.[11]

Waste Disposal

Work in a highly efficient fume hood. Wear long-sleeved rubber gloves, nonporous protective clothing, eye protection, and breathing apparatus if necessary. Pour 60 mL of a 2.5 M sodium hydroxide solution (prepared by dissolving 6 g of NaOH in 60 mL of cold water; 0.15 mol, 50% excess) into a 100-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Add the methyl fluorosulfonate (0.05 mol, 5.7 g, 4 mL) dropwise while stirring. If reaction is sluggish, the mixture is heated to about 90°C. When the initially added material has dissolved, the remainder is added dropwise. When a clear solution has been obtained, the mixture is cooled to room temperature, neutralized to about pH 7 with dilute hydrochloric acid, and washed into the drain.[12]

First Aid

1. Prior to using Magic Methyl®, a consultant physician, experienced in treating pulmonary edema, should be informed of its proposed use and be prepared to accept a victim of an accident as a priority case.

2. Dexamethasone isonicotinate spray has been suggested as immediate treatment but its use may be as hazardous as the condition being treated. Beclomethasone dipropionate would be less dangerous but its effectiveness in treating this type of situation has not been tested. If it did not work, the victim might die before effective treatment was instituted.
3. Victim should report to consultant physician immediately.[13]

Reactions for Spillage and Waste Disposal

$$FSO_2OCH_3 + NaOH \rightarrow CH_3OH + FSO_3Na$$

REFERENCES

1. Merck 6099.
2. Sax 2323.
3. Alder, R.W., *Chem. Ind.,* 20, 983–985, 1973; *J. Org. Chem.*, 43, 678–679, 1978.
4. Aldrich Chemical Company, *Technical Bulletin on Magic Methyl*, Aldrich Chemical Company, Milwaukee, WI, April 1971.
5. van den Ham, D.M.W. et al., *Chem. Eng. News,* 54, 5, 1976.
6. Alder, R.W. et al., *Chem. Eng. News*, Sept. 11, 56, 1978.
7. Ashby, J. et al., *Mutation Research,* 51, 285–287, 1978.
8. Hite, M. et al., *Am. Ind. Hyg. Assoc. J.*, 40, 600–603, 1979.
9. Chem. Br., 14, 595, 1978.
10. Chem. Br., 15, 329, 1979.
11. Aldrich Chemical Company, Hazard Files, Technical Services, Milwaukee, 1971.
12. PPL 161.
13. Chem. Br., 14, 71, 1978.

METHYL HYDRAZINE CH_3NHNH_2

SUSPECTED ANIMAL CARCINOGEN

Physical Properties

Clear liquid with amine odor; bp, 87.5°C.[1]

Fire Hazard

Flammable; flash point, –8°C; ignition temperature, 194°C; flammability limits, 2.5–92%.[2] Extinguish fire with water.[2]

Chemical Properties

Miscible with water, hydrazine, and low-molecular-weight monohydric alcohols; soluble in hydrocarbons. Mild base. Strong reducing agent.[1]

Hazardous Reactions

Oxidants. Ignites when exposed to air as a thin film or on contact with hydrogen peroxide or nitrogen dioxide, among other oxidants. Powerful reducing agent.[3]

Physiological Properties and Health Hazards

Toxicity high by inhalation, dermal, and oral routes. Suspect teratogen, neoplastic, and carcinogen.[4] TLV-TWA 0.01 ppm (skin).[5]

Spillage Disposal

Wear butyl rubber gloves, self-contained breathing apparatus, eye protection, and impervious clothing. Body shield should be available. Eliminate all sources of ignition and flammables.

On skin or clothing. Wash skin immediately. Remove contaminated clothing at once.

Spills. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container, transport to the fume hood, and slowly add to water allowing 20 mL water for each 1 g of methylhydrazine. Filter off the clay and sand. For each 1 g of methylhydrazine, place 41 mL (about 25% excess) of commercial laundry bleach (containing about 5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous methylhydrazine dropwise to the stirred hypochlorite solution, monitoring the rate of addition by rise in temperature. The temperature is maintained at 45–50°C and addition takes about 1 hour. Stirring is continued for 2 hours until the temperature gradually falls to room temperature. The cooled reaction mixture can be flushed down the drain.[6,7]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Prepare a dilute (5%) aqueous solution of methylhydrazine by adding slowly to the appropriate volume of water. For each 1 g of methylhydrazine, place 41 mL (about 25% excess) of household laundry bleach (5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous methylhydrazine

dropwise to the stirred hypochlorite solution, monitoring the rate of addition by rise in temperature. The temperature is maintained at 45–50°C and addition takes about 1 hour. Continue stirring for 2 hours until the temperature gradually falls to room temperature. The cooled reaction mixture can be washed into the drain.[7]

Reactions for Spillage and Waste Disposal

$$CH_3NHNH_2 + NaOCl \rightarrow CH_4 + N_2 + NaCl + H_2O$$

REFERENCES

1. Merck 6109.
2. NFPA 49, 325M.
3. B 179.
4. Sax 2327.
5. ACGIH 42.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.
7. PP 94; PPL 45, 329.
8. Ald 2364C.

METHYL METHANESULFONATE $CH_3SO_2OCH_3$

ANIMAL CARCINOGEN

Synonyms

Methyl mesylate, methanesulfonic acid methyl ester, MMS.

Physical Properties

Colorless liquid[1]; bp, 202–203°C.[2]

Chemical Properties

Soluble in water and dimethylformamide; slightly soluble in nonpolar solvents.[3]

Hazardous Reactions

Hydrolyzed in water to the strongly corrosive methanesulfonic acid.[1]

Physiological Properties and Health Hazards

Carcinogen in rats. Adverse effects in patients include nausea, vomiting, hepatic toxicity, reduced visual acuity, and blood abnormalities. Mutagenic in the Ames test and teratogenic in mice and rats. Reasonably anticipated to be a human carcinogen.[1,4]

Spillage Disposal

Wear protective gloves, laboratory coat, goggles, and breathing apparatus if necessary. Cover spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container and either label and send for disposal by burning; or, in the fume hood, add the mixture to a solution of potassium hydroxide in ethanol allowing 0.12 mol (7.9 g) of 85% potassium hydroxide pellets and 32 mL of ethanol for each 0.1 mol (11.0 g) of methyl methanesulfonate to be destroyed. Heat the mixture under reflux for 8 hours. Decant the cooled solution into the drain with water. Treat the solid residue as normal refuse.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for burning. Dissolve or mix the material into a combustible solvent and package and label for disposal by incineration.[2]

Small Quantities. Wear protective gloves, laboratory coat, and goggles. Work in the fume hood. Place 7.9 g (0.1 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, condenser, and dropping funnel. Add 32 mL of 95% ethanol rapidly. Heat the solution to gentle reflux and add methyl methanesulfonate (11.0 g, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring for 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain with water.[6]

REFERENCES

1. IARC 7, 253.
2. Aldrich 1130; Ald 2378D.
3. Merck 6119.

4. NIEHS III–151.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.
6. PP 61.

1-METHYL-3-NITRO-1-NITROSOGUANIDINE $C_2H_5N_5O_3$

ANIMAL CARCINOGEN

Synonyms

N-nitroso-N'-nitro-N-methyl-guanidine, MNNG.

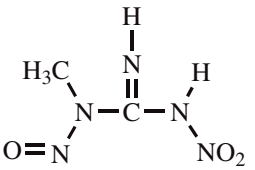
Physical Properties

Yellow crystals from methanol; mp, 118°C.[1]

Chemical Properties

Only slightly soluble in water (below 0.5%); soluble in polar organic solvents, although this is often accompanied by decomposition.[2]

Structure



Hazardous Reactions

A diazomethane precursor, this compound detonates on high impact; sample explodes when heated in a sealed capillary tube.[3]

Physiological Properties and Health Hazards

Very potent mutagen.[4] In experimental animals, causes cancer of the stomach, intestine, skin, and lung, and at the site of injection.[5] Reasonably anticipated to be a human carcinogen.[6,7] LD_{50} (oral, rat) 400 mg/kg.[8]

Spillage Disposal

Solutions. Wear protective gloves, laboratory coat, and goggles. Absorb the spill on paper towels. Place the paper towels in a beaker in the fume hood. Immediately pour over the area of the spill enough of a solution of 4.7 g of potassium permanganate in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) to thoroughly wet the contaminated area and let stand overnight. Fill the beaker containing the paper towels with acid permanganate solution. Allow to stand at room temperature overnight. Dilute the mixture in the beaker and neutralize the solution by cautiously adding 10% aqueous sodium hydroxide. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse.[9] Add a 5% solution of ascorbic acid to the area and allow to react for 15 minutes. Add solid soda ash to the decontaminated surface. Remove the decontamination mixture by absorbing on paper towels. Discard the paper towels.

Solids. Cover the spill with a solution of 4.7 g of potassium permanganate in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow to stand overnight. Absorb the liquid residue on a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the solid into a container and, in the fume hood, add to a beaker of cold water. If necessary, neutralize the solution by the cautious addition of soda ash. Decant the liquid into the drain with water. Treat the solid as normal refuse.[9,10]

Waste Disposal

Wear protective gloves, laboratory coat, and goggles. Work in the fume hood. Add sufficient 3 M sulfuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water) to the waste to produce a solution not more than 5% in 1-methyl-3-nitro-1-nitrosoguanidine. For each 10 mL of solution, add 0.5 g of potassium permanganate to give a 0.3 M solution. Stir the mixture at room temperature for 12 hours. Neutralize by cautiously adding soda ash or 10% aqueous sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless liquid is formed. Wash into the drain with water.[9,10]

REFERENCES

1. Merck 6125.
2. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides,* No. 55, IARC Scientific Publications, Lyon, 1983, p. 51.
3. Lux 483.
4. Aldrich 1137; Ald 2393A.
5. Magee, P.N., Montesano, R., and Preussman, R., N-nitroso compounds and related carcinogens, in *Chemical Carcinogens,* Searle, C.E., Ed., ACS Monograph Series No. 173, American Chemical Society, Washington, D.C., 1976.
6. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides,* No. 55, IARC Scientific Publications, Lyon, 1983, p.3.
7. NIEHS III–151.
8. Sax 2356.
9. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides,* No. 55, IARC Scientific Publications, Lyon, 1983, p. 31.
10. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.

METHYLTRICHLOROSILANE CH_3SiCl_3

FLAMMABLE

Synonyms

Trichloromethylsilane.

Physical Properties

Colorless to pale yellow volatile liquid with pungent odor; fumes in moist air; bp, 65.5°C.[1]

Fire Hazard

Flammable liquid; flash point, −15°C (closed cup); explosive limits, 5.1–20%; auto-ignition temperature, 404°C.[1]

Hazardous Reactions

Fumes in moist air. Reacts violently with water to form hydrochloric acid.[1]

Physiological Properties and Health Hazards

Vapor highly irritating to the skin, eyes, and mucous membranes. Ingestion causes severe internal damage.[1]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a suitable container and transport to the fume hood. Slowly add the solid to a pail of cold water; neutralize if necessary with sodium carbonate and decant the liquid to the drain. Treat the solid as normal refuse.[2,3]

Waste Disposal

Package Lots. Package and label for disposal by burning in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Equip a three-necked, round-bottom flask with a stirrer, dropping funnel, and thermometer. Place 500 mL of water in the flask. Cool the flask in an ice-water bath. Add 10 mL of methyltrichlorosilane to the funnel and add dropwise to the stirred water. Do not allow the temperature of the solution to rise above 20°C. When addition is complete, neutralize the solution with sodium carbonate and wash into the drain.[3]

Reactions for Spillage and Waste Disposal

$$CH_3SiCl_3 + H_2O \rightarrow \underset{\text{methylsilanol}}{CH_3Si(OH)_3} + 3HCl$$

REFERENCES

1. Lux 490.
2. Aldrich 1170; Ald 2454C.

3. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.5.

MITOMYCIN C

$C_{15}H_{18}N_4O_5$

ANTINEOPLASTIC

Synonyms

Mutamycin, Mitocin-C, MMC, Ametycine.

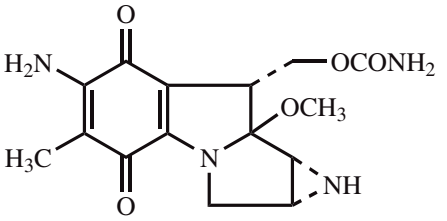
Physical Properties

Blue-violet crystals; mp, above 360°C.[1]

Chemical Properties

Soluble in water, methanol, acetone, butanol, and cyclohexanone. Slightly soluble in benzene, carbon tetrachloride, and ether. Practically insoluble in petroleum ether.[1] Light sensitive.[2]

Structure



Mode of Action

Mitomycin C is activated by intracellular reductases, and acts as an alkylating agent, forming cross-links with DNA. At high concentrations of mitomycin C, RNA and protein synthesis are suppressed.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include bone marrow toxicity, skin toxicity, renal toxicity, and pulmonary toxicity, as well as vomiting, nausea, fever, anemia, anorexia, stomatitis, hypoglycemia, mucositis, and diarrhea.[2] LD_{50} (i.v., mice) 5–9 mg/kg.[1]

Spillage Disposal

Solutions. Wear goggles and protective gloves and clothing. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Estimate the weight of mitomycin C in the spilled liquid and add 10% sodium hydroxide solution to the mixture, allowing 50 mL for each 0.5 mg of mitomycin C. Allow the mixture to stand at room temperature overnight. Decant the liquid into the drain with water and discard the solid as normal refuse.[3–5]

Waste Disposal

Wear protective gloves, clothing, and goggles. To each 1 mL of solution containing 0.5 mg of mitomycin C, add 50 mL of a 10% solution of sodium hydroxide. Allow the mixture to stand at room temperature for at least 5 hours. Wash the solution into the drain with a large volume of water. Immerse glassware that has been in contact with mitomycin C solutions in 10% sodium

hydroxide solution and allow to stand overnight. Rinse the glassware with water and wash the sodium hydroxide solution into the drain with water.[3,4]

REFERENCES

1. Merck 6236.
2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data,* 2nd ed., G.K. Hall Medical Publishers, Boston, 1982, pp. 140–141.
3. Wilson, S.J., *Journal of Clinical and Hospital Pharmacy,* 8, 295–299, 1983.
4. Armour, M.A. and McKenzie, P., *Laboratory Disposal Method for Mitocycin C*, University of Alberta, Edmonton, 1998.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.,* 62, A93, 1985; Armour, M.A., *J. Chem. Educ.,* 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.

MORPHOLINE C_4H_9NO

FLAMMABLE, IRRITANT

Synonyms

Tetrahydro-1,4-oxazine.

Physical Properties

Colorless, mobile, hygroscopic liquid with amine-like odor; bp, 127°C.[1]

Fire Hazard

Flash point, 38°C (open cup); auto-ignition temperature, 290–310°C. Extinguish fire with dry powder, carbon dioxide, or vaporizing liquid.[2]

Chemical Properties

Miscible with water with evolution of some heat; also miscible with acetone, benzene, ether, castor oil, methanol, ethanol, ethylene glycol, 2-hexanone, turpentine, and linseed and pine oils.[1]

Hazardous Reactions

Cellulose Nitrate. High-surface-area cellulose nitrate ignites spontaneously on contact with morpholine.[3]

Nitromethane. Addition of morpholine makes nitromethane susceptible to initiation by a detonator.[4]

Physiological Properties and Health Hazards

Vapor irritates the eyes and respiratory system. Liquid irritates the eyes and skin. Swallowing causes internal irritation and kidney and liver damage. Avoid breathing vapor. Avoid contact with skin, eyes, and clothing.[2] TLV-TWA 20 ppm (71 mg/m^3).[5]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl[7] rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the morpholine has been absorbed, scoop the mixture into a plastic pail and add enough water to dissolve the sodium carbonate. Allow the solids to settle and decant the liquid to another container. Discard the solids with normal refuse. To the liquid, slowly add (frothing will occur) 6 M sulfuric acid to pH 2. Stir into the acidified solution sufficient solid potassium permanganate so that the liquid is purple (a drop of the liquid on a filter paper will show a purple ring). Allow the mixture to stand at room temperature for 48 hours, and then neutralize with solid sodium carbonate (frothing will occur), or with a 10% aqueous solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[6–8]

Waste Disposal

Package Lots. Distill for reuse or package and label for disposal by burning.

Small Quantities. Wear eye protection, laboratory coat, and rubber gloves. Dissolve the morpholine (1 mL) in 50 mL of 3 M sulfuric acid (prepared by slowly adding 8 mL of concentrated sulfuric acid to 21 mL of water). Weigh 10 g of potassium permanganate and stir small portions of the solid into the morpholine solution over a period of about 1 hour. Stir the mixture at room temperature for 48 hours, and then neutralize the solution by adding solid sodium carbonate or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until solution is colorless. Decant the clear liquid into the drain and discard any brown solid with regular refuse.[8,9]

REFERENCES

1. Merck 6303 6194.
2. Lux 492.
3. B 1535.
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NAPHTHOL $C_{10}H_7OH$

IRRITANT, LIGHT SENSITIVE

Synonyms

1-Naphthol: 1-napthalenol, α-naphthol, α-hydroxynaphthalene[1]; 2-Naphthol: 2-napthalenol, β-naphthol, β-hydroxynaphthalene.[1]

Physical Properties

1-Naphthol. Prisms, mp, 96°C; phenolic odor; disagreeable, burning taste; bp, 288°C. Sublimable; volatile with steam. Darkens in light; reduces ammoniacal silver nitrate.[1]

2-Naphthol. Crystals, mp, 121–123°C; bp, 285–286°C. Flash point, 161°C. Slight phenolic odor. Darkens with age on exposure to light. Sublimes when heated, distillable *in vacuo*; volatile with vapors of alcohol or water; reduces ammoniacal silver nitrate.[1]

Fire Hazard

Extinguish fires with water spray, carbon dioxide, dry chemical powder, or alcohol or polymer foam.[2]

Chemical Properties

1-Naphthol. Slightly soluble in water, freely in alcohol, benzene, chloroform, ether, and alkali hydroxide solution. Protect from light.[1]

2-Naphthol. One gram dissolves in 1000 mL of water, 80 mL of boiling water, 0.8 mL of alcohol, 17 mL of chloroform, or 1.3 mL of ether; soluble in glycerol, olive oil, and solutions of alkali hydroxides. Protect from light.[1]

Hazardous Reactions

1-Naphthol. Incompatible with strong oxidizing agents, strong bases, acid chlorides, and acid anhydrides, halogens.[3]

2-Naphthol. Incompatible with strong oxidizing agents, strong bases, acid chlorides, and acid anhydrides.[3]

Physiological Properties and Health Hazards

Harmful if swallowed, inhaled, or absorbed through skin. Causes severe irritation. High concentrations are extremely destructive to all body tissues. Experimental teratogen.[2,3]

Spillage Disposal

Wear nitrile rubber gloves, goggles, and a laboratory coat. Sweep the spill into a beaker and place in the fume hood. Estimate the weight of the naphthol spilled and for each 1 g, add 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid added to 83 mL of water) and 6 g of potassium permanganate. Stir the mixture for 48 hours. Add solid sodium bisulfite until the solution is colorless. Neutralize with 5% aqueous sodium hydroxide and pour into the drain. Discard small quantities of brown precipitate (manganese dioxide) with normal refuse.[3–5]

Waste Disposal

Package Lots. Label for recycling or disposal by burning.[3,4]

Small Quantities. Follow spillage disposal procedure.

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1-NAPHTHYLAMINE and SALTS $C_{10}H_7NH_2$

SUSPECT HUMAN CARCINOGEN, POISONOUS DUST, TOXIC IN CONTACT WITH SKIN, DANGER OF CUMULATIVE EFFECTS

Synonyms

1-Naphthalenamine, 1-aminonaphthalene, α-naphthylamine, naphthalidine.

Physical Properties

Colorless crystals when pure, darkening on exposure to light and air; unpleasant odor; mp, 50°C (base).[1]

Chemical Properties

1-Naphthylamine is soluble in 590 parts water; the hydrochloride is soluble in 27 parts water.[1]

Physiological Properties and Health Hazards

Salts or solutions irritate eyes. Prolonged exposure to dust or skin absorption may cause bladder tumors. There is some question whether human carcinogenicity is due to contamination with 2-naphthylamine.[2] Prevent inhalation of dust. Prevent contact with skin, eyes, and clothing.[3]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus.

On skin and clothing. Wash skin with strong soap solution immediately. Rinse thoroughly. Contaminated clothing should be removed at once.

Spills. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer this solid mixture to an appropriate container labeled for disposal by burning in a furnace equipped with afterburner and scrubber.[4,5]

Waste Disposal

Package Lot. Place in a separate labeled container for disposal by burning. Dissolve the naphthylamine in a flammable solvent and spray into furnace with afterburner and scrubber.[5]

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To a 100-mL, three-necked, round-bottom flask equipped with stirrer, thermometer, and dropping funnel, add 2.5 mL of water, 7.5 mL of concentrated hydrochloric acid, and 2.86 g (0.02 mol) of naphthylamine. Maintain the temperature at −5 to 0°C by a cooling bath, while adding dropwise 1.5 g (0.0211 mol) of 97% sodium nitrite dissolved in 3.5 mL of water while stirring. After addition is complete, continue stirring an additional 30 minutes. Maintain the temperature at −5 to 0°C and add 41.6 mL (0.4 mol) of 50% hypophosphorous acid (precooled to 0°C) over 10–15 minutes. Continue stirring for 2 hours. Allow the reaction mixture to stand at room temperature for 24 hours, and then extract with 2 × 10 mL portions of toluene. The toluene extract of naphthalene is packaged and sent for disposal by burning. The aqueous portion is washed into the drain.[6]

For each 9 mg of 1-naphthylamine, add 10 mL of 0.1 M hydrochloric acid (prepared by cautiously adding 1 mL of concentrated acid to 119 mL of cold water). Mix to dissolve. For each 10 mL of the solution, add 5 mL of 0.2 M potassium permanganate solution (0.3 g of solid potassium permanganate dissolved in 10 mL of water) and 5 mL of 2.0 M sulfuric acid

(prepared by cautiously adding 1 mL of concentrated acid to 8 mL of cold water). Mix and let stand overnight (at least 10 hours). Decolorize if necessary with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (20 g of NaOH dissolved in 100 mL of cold water). Discard the remaining solution into the drain.[7]

Reactions for Spillage and Waste Disposal

$$C_{10}H_7NH_2 \cdot HCl + 2H^+ \xrightarrow[2)\ H_3PO_2]{1)\ HNO_2} C_{10}H_8 + NH_4Cl$$

naphthalene

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6. PP 70; PPL 162.
7. Cas 64, 24.

2-NAPHTHYLAMINE AND SALTS $C_{10}H_7NH_2$

HUMAN CARCINOGEN

Synonyms

2-Naphthalenamine, 2-aminonaphthalene, β-naphthalene.

Physical Properties

White to reddish crystals; mp, 111–113°C.[1]

Chemical Properties

Soluble in hot water, alcohol, and ether.[1]

Physiological Properties and Health Hazards

A human carcinogen via oral and subcutaneous routes.[2] Inhalation or absorption of the dust through the skin has been recognized as a cause of bladder tumors.[3,4] Listed as known human carcinogen.[4] Exposure by all routes should be carefully controlled to the lowest possible levels.[5]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, eye protection, and self-contained breathing apparatus.

On skin and clothing. Wash skin with strong soap solution immediately. Rinse thoroughly. Contaminated clothing should be removed at once.

Spills: Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer this solid mixture to an appropriate container, labeled for disposal by burning in a furnace equipped with afterburner and scrubber.[6,7]

Waste Disposal

Package Lot. Place in a separate labeled container for disposal by burning. Dissolve the naphthylamine in a flammable solvent and spray into furnace with afterburner and scrubber.[6]

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To a 100-mL, three-necked, round-bottom flask equipped with stirrer, thermometer, and dropping funnel, add 2.5 mL of water, 7.5 mL of concentrated hydrochloric acid, and 2.86 g (0.02 mol) of naphthylamine. The temperature is maintained at −5 to 0°C by a cooling bath while 1.5 g (0.0211 mol) of 97% sodium nitrite dissolved in 3.5 mL of water is added dropwise while stirring. After addition is complete, continue stirring an additional 30 minutes. Maintain the temperature at −5 to 0°C and add 41.6 mL (0.4 mol) of 50% hypophosphorous acid (precooled to 0°C) over 10–15 minutes. Continue stirring for 2 hours. Allow the reaction mixture to stand at room temperature for 24 hours, and then extract with 2 × 10 mL portions of toluene. The toluene extract of naphthalene is packaged and sent for disposal by burning. The aqueous portion is washed into the drain.[7]

For each 9 mg of 2-naphthylamine, add 10 mL of 0.1 M hydrochloric acid (prepared by cautiously adding 1 mL of concentrated acid to 119 mL of cold water). Mix to dissolve. For each 10 mL of the solution, add 5 mL of 0.2 M potassium permanganate solution (0.3 g of solid potassium permanganate dissolved in 10 mL of water) and 5 mL of 2.0 M sulfuric acid (prepared by cautiously adding 1 mL of concentrated acid to 8 mL of cold water). Mix and let stand overnight (at least 10 hours). Decolorize if necessary with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (20 g of NaOH dissolved in 100 mL of cold water). Discard the remaining solution into the drain.[7,9]

Reactions for Spillage and Waste Disposal

$$C_{10}H_7NH_2 \cdot HCl + 2H^+ \xrightarrow[2)\ H_3PO_2]{1)\ HNO_2} C_{10}H_8 + NH_4Cl$$

naphthalene

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9. Cas 64, 24.

NICKEL Ni

ANIMAL CARCINOGEN

Physical Properties

Lustrous white, hard, ferromagnetic metal; mp, 1555°C.[1]

Fire Hazard

Catalytic forms (e.g., Raney nickel) may ignite spontaneously in air. Extinguish fire with dry sand or class D extinguisher. Do not use carbon dioxide.[2] See notes under hazardous reactions.

Chemical Properties

Stable in air at ordinary temperature; burns in oxygen, forming nickel oxide; not affected by water; decomposes steam at a red heat. Slowly attacked by dilute hydrochloric or sulfuric acid; readily attacked by nitric acid. Not attacked by fused alkali hydroxides.[1]

Hazardous Reactions

Hydrogenation Catalysts

CAUTION: Raney nickel is pyrophoric when dry.

Many hydrogenation catalysts are sufficiently active to effect rapid interaction of hydrogen and/or solvent vapor with air, causing ignition or explosion. This is particularly so where hydrogen is adsorbed on the catalyst either before a hydrogenation (Raney cobalt, nickel, etc.) or after a hydrogenation during separation of catalyst from the reaction mixture. Exposure to air of such a catalyst should be avoided until complete purging with an inert gas, such as nitrogen, has been effected. With catalysts of high activity and readily reducible substrates, control of the exotherm may be required to prevent runaway reactions, particularly at high pressures.[3]

Spent catalysts should be purged from hydrogen and washed free from organics with water before storage in the water-wet condition. Under no circumstances should any attempt be made to dry a spent catalyst.[4]

Information on reactive nickel compounds is available from the Royal Society of Chemistry.[5]

For handling, see Adkins et al.[6]

Very fine powder may be pyrophoric; should not be degassed by heating under vacuum as explosion may result.[7]

Ammonium Nitrate. Powdered Ni with fused ammonium nitrate explodes below 200°C.[8]

p-Dioxane. Reacts almost explosively with Raney Ni above 210°C.[9]

Hydrogen. Active (W6) Raney Ni should not be used as a hydrogenation catalyst above 100°C.[6]

Methanol. Methanol will ignite when poured onto Raney Ni if traces of air are present.[10]

Nonmetals. Mixtures of powdered Ni with sulfur or selenium incandesce on heating.[11]

Organic Solvents. Raney Ni recovered from methanol, ethanol, pentanol, acetone, benzene, cyclohexane, or *p*-dioxane explodes when heated above 200°C.[10]

Peroxyformic Acid. Violent explosion with 90% acid.[12]

Sulfur Compounds. Raney Ni containing the sulfide is pyrophoric.[13]

Physiological Properties and Health Hazards

Dust irritates the skin, eyes, and mucous membranes.[14] Animal carcinogen; reasonably anticipated to be a human carcinogen.[15] May cause dermatitis in sensitive individuals.[1] TLV-TWA 1.5 mg/m^3.[16]

Spillage Disposal

See Waste Disposal.

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

Raney Nickel Catalyst. Finely divided nickel (25 g) can ignite in air if allowed to dry. Wash with 100 mL of water, and then suspend in 200 mL of water in a three-necked, round-bottom flask equipped with a stirrer. Gradually add 800 mL of 1 M hydrochloric acid (prepared by cautiously adding 67 mL of concentrated acid to 733 mL of cold water) while stirring. Continue stirring until all the nickel is dissolved. The solution is slowly basified with 5% sodium hydroxide to precipitate nickel hydroxide. The precipitate is collected and packaged for disposal in a secure landfill. The aqueous solution is washed down the drain with at least 50 times its volume of water.[17]

Metal. Sort, classify, and package for recycling.[18]

Reactions for Spillage and Waste Disposal

$$Ni + 2HCl \rightarrow NiCl_2 + H_2$$

$$NiCl_2 + 2NaOH \rightarrow Ni(OH)_2 + 2NaCl$$

nickel hydroxide (insoluble)

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16. ACGIH 44.
17. PP 90.
18. ITI 446.

NICKEL CARBONYL $Ni(CO)_4$

ANIMAL CARCINOGEN, SEVERE POISON, HIGHLY FLAMMABLE

Synonyms

Tetracarbonyl nickel.

Physical Properties

Colorless volatile liquid; bp, 43°C.[1]

Fire Hazard

Explosive limits, above 2%; flash point, below –20°C; lower flammable limit, 2%.[2] Explodes at about 60°C.[1]

Chemical Properties

Slightly soluble in air-free water; soluble in alcohol, benzene, chloroform, acetone, and carbon tetrachloride. Oxidizes in air.[1]

Hazardous Reactions

Air. In air, forms solid that may ignite.[3]

Bromine. Explosive interaction in liquid state.[4]

Dinitrogen Tetroxide. Interaction of the two liquids is rather violent.[5]

Mercury and Oxygen. A mixture of the dry carbonyl and oxygen will explode on vigorous shaking with mercury.[6]

Oxygen. Mixtures may explode.[3]

Physiological Properties and Health Hazards

Exceedingly poisonous. Vapor may cause irritation, congestion, and edema of lungs; prolonged exposure may cause cancer of lungs or nasal sinuses.[1,7] Immediate symptoms due to inhalation include headache and dizziness, which eventually lead to vomiting, insomnia, and difficult breathing; death may result several days after exposure or due to prolonged exposure.[7,8] Animal carcinogen; reasonably anticipated to be a human carcinogen.[9] TLV-TWA 0.05 ppm (0.12 mg/m^3).[10]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, goggles, and breathing apparatus. Absorb with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop solid into an appropriately labeled container for disposal by burning.[8,11]

Waste Disposal

Package Lots. Package securely, label, and dispose of by burning. Mix with a flammable solvent prior to spraying into furnace.[8]

Small Quantities. *Use extreme caution. Handle with care*. Wear butyl rubber gloves, laboratory coat, eye protection, and breathing apparatus if required. Work in the fume hood. A 5% solution of nickel carbonyl in tetrahydrofuran is added gradually while stirring to a 25% molar excess of household bleach (11 mL/g of nickel carbonyl) in a three-necked, round-bottom flask equipped with a dropping funnel or gas inlet, a nitrogen inlet, and stirrer. The decomposition procedure should be carried out under a nitrogen atmosphere.[12] The solution should be stirred overnight

after addition of the carbonyl is complete. Filter the solution. The filtrate may be poured to the drain with at least 50 times its volume of water while the wet precipitate (finely divided, pyrophoric nickel) is added to an excess of ammonium hydroxide solution. This precipitate may be filtered, packaged, and labeled before being recycled or sent to a secure landfill.[13]

Reactions for Spillage and Waste Disposal

$$Ni(CO)_4 + NaOCl + H_2O \rightarrow \underset{\substack{\text{nickel}\\\text{hydroxide}\\\text{(insoluble)}}}{Ni(OH)_2} + 4CO + NaCl$$

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NITRIC ACID HNO_3

CONTACT WITH COMBUSTIBLE MATERIALS MAY CAUSE FIRES, CAUSES SEVERE BURNS

Physical Properties

Colorless or pale yellow fuming liquid; characteristic choking odor; bp (68%), 120.5°C.[1]

Chemical Properties

Miscible with water.[1]

Hazardous Reactions

The oxidizing power (and hazard potential) of nitric acid increases progressively with increase in strength from the concentrated acid (70% weight HNO_3) through fuming acids (above 85% weight) to the anhydrous 100% acid. The presence of dissolved oxides of nitrogen in the red fuming grades of acid further enhances the potency of the oxidant.

Screw-cap containers of fuming nitric acid may develop pressure on storage, owing to attack on the plastic screw cap if the inert protective cap liner fails. Regular pressure release is recommended by one laboratory supplier. For a discussion of the hazards of mixtures of many organic compounds with HNO_3, see Andrussow.[2]

Acetic Anhydride. Mixtures containing more than 50% fuming HNO_3 can be detonated; mixtures containing less than 50% HNO_3 react uncontrollably with small quantities of water.[3–5]

Acetone. Ignites on contact with fuming HNO_3.[6]

Acetone and Acetic Acid. Mixture containing equal parts of the components will explode if kept in a closed container.[7,8]

Acetone and Sulfuric Acid. Mixture may explode if confined in narrow-mouth vessel.[9]

Acetonitrile. Mixture forms high explosive.[2]

Acetylene and Mercury Salts. Contact of acetylene with concentrated HNO_3 in the presence of Hg salts forms trinitromethane, explosive above its mp at 15°C.[10]

Alcohols. Mixtures of nitric acid and alcohols are quite unstable.[11–13]

Alcohols and Potassium Permanganate. Addition of alcohols to fuming HNO_3 containing potassium permanganate results in immediate ignition.[14]

Aliphatic Amines. Mixtures of amines with fuming HNO_3 may ignite.[15]

Alkanethiols. Oxidation of alkanethiols to alkanesulfonic acids with concentrated HNO_3 may cause ignition of the thiol.[15]

2-Aminothiazole and Sulfuric Acid. Nitration of 2-aminothiazole with H_2SO_4/HNO_3 may result in a violent explosion if cooling is omitted.[15]

Ammonia. A jet of ammonia will ignite in HNO_3 vapor.[16]

Aromatic Amines. Many aromatic amines ignite on contact with fuming HNO_3.[17]

Bromine Pentafluoride. May ignite on contact with concentrated HNO_3.[18]

Cellulose. May be converted to the highly flammable nitrate ester on contact with HNO_3 vapor as well as with liquid.[17]

4-Chloro-2-nitroaniline. Primary aromatic amines should be protected by acetylation before nitration to avoid the possibility of accidental diazotization.[19]

Crotonaldehyde. Ignites in concentrated HNO_3.[2]

1,2-Dichloroethane. Mixtures are easily detonated by heat, impact, or friction.[20]

Dichloromethane. Solution can be detonated.[2]

Dichromates. Dichromates, particularly ammonium dichromate, promote ignition of mixtures of fuming HNO_3 with cyclohexanol, *m*- or *o*-cresol and furfural.[20]

Diethyl Ether and Dimethyl Ether. Addition of ether to 50% aqueous HNO_3 can cause an explosion.[20] A mixture of the ether, HNO_3, and H_2SO_4 explodes violently.[2,21]

1,1-Dimethylhydrazine. Reacts vigorously with HNO_3.[22]

Dimethyl Sulfide and Dioxane. Mixture explodes after a delay, even at liquid nitrogen temperature.[23]

Fat and Sulfuric Acid. Mixtures of adipose tissue and fuming nitric and sulfuric acids sealed in bombs, exploded.[24,25]

Fluorine. Interaction of fluorine with concentrated or very dilute HNO_3 results in explosions.[26,27]

Hydrazine and Derivatives. Mixtures with concentrated HNO_3 ignite on contact.[2]

Hydrocarbons. Dienes and acetylene derivatives ignite in concentrated HNO_3.[2] Cyclopentadiene reacts explosively with fuming HNO_3.[28] Benzene solutions containing 84% acid are very sensitive to detonation.[29] Nitration of toluene must be carefully controlled or an explosion may occur.[29] During the nitration of mesitylene in acetic acid-anhydride solution, the temperature must be kept below 20°C.[30,31]

Hydrogen Iodide, Hydrogen Selenide, and Hydrogen Sulfide. Ignition occurs on contact with fuming HNO_3.[32]

Hydrogen Peroxide and Ketones. Explosive ketone peroxides may be produced.[33]

Ion Exchange Resins. Interaction with anion exchange resins and HNO_3 may cause explosions.[5]

Iron(II) Oxide. Finely divided oxide incandesces in HNO_3.[34]

Metals. Antimony may react violently with fuming HNO_3. Bismuth glows red hot in contact with fuming HNO_3. Germanium reacts violently; lithium ignites. Manganese powder incandesces and sodium ignites. Titanium alloys form an explosive deposit; uranium powder reacts vigorously. Molten zinc incandesces; magnesium burns brilliantly.[35]

4-Methylcyclohexanone. Mixture with concentrated HNO_3 may explode at about 75°C.[36]

Nitroaromatics. Mixtures of HNO_3 with mono- and di-nitrobenzenes and di- and tri-nitrotoluenes possess high explosive properties.[37]

Nitromethane. Mixtures with HNO_3 are extremely explosive.[38]

Nonmetal Hydrides. Arsine, phosphine, and tetraborane are oxidized explosively by fuming HNO_3 and stibine by concentrated HNO_3. Phosphine, hydrogen sulfide, and hydrogen selenide ignite in contact with fuming acid. Hydrogen telluride ignites with cold concentrated acid.[39]

Nonmetals. Boron reacts violently with concentrated HNO_3. Phosphorus vapor may ignite. Silicon, arsenic, and finely divided carbon react violently.[40]

Organic Matter. Organic matter may explode with concentrated HNO_3.[41]

Phosphorus Halides. Phosphorus trichloride explodes with HNO_3.[42]

Phthalic Anhydride and Sulfuric Acid. Excess nitrating mixture (HNO_3 + H_2SO_4) caused delayed vigorous reaction with phthalic anhydride.[43]

Polyalkenes. Fuming HNO_3 will attack polyethylene or polypropylene but not polytetrafluoroethylene.[44,45]

Sulfur Dioxide. Presence of dinitrogen tetroxide appears to be essential to catalyze smooth formation of nitrosylsulfuric acid. In its absence, reaction may be delayed and then proceed explosively.[46]

Thioaldehydes or Thioketones. Violent reaction with HNO_3.[47]

Thiophene. Violent reaction with fuming HNO_3.[48,49]

Wood. Concentrated HNO_3 may ignite sawdust, and fuming HNO_3 may ignite wood.[50]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system, eyes, and skin. Liquid burns the eyes and skin. Swallowing causes internal irritation and damage. Avoid breathing vapor. Prevent contact with eyes and skin.[51] TLV-TWA 2 mg/m^3; TLV-STEL 4 mg/m^3.[52]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and a face shield. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the nitric acid has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[53]

Waste Disposal

Wear nitrile rubber gloves, eye protection, and a laboratory coat. Have a body shield available. Work in the fume hood. Add the acid slowly to a pail of cold water. Neutralize the solution with sodium carbonate and wash into the drain.[53]

Reactions for Spillage and Waste Disposal

$$2HNO_3 + Na_2CO_3 \rightarrow 2NaNO_3 + H_2O + CO_2$$

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53. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.3; Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 25, 1999.

NITROBENZENE $C_6H_5NO_2$

TOXIC BY INHALATION, SWALLOWING, OR SKIN CONTACT; DANGER OF CUMULATIVE EFFECTS

Physical Properties

Colorless to pale yellow oily liquid with odor of bitter almonds; bp, 210–211°C.[1]

Fire Hazard

Combustible. Flash point, 88°C; ignition temperature, 496°C. Extinguish with water, dry chemical, foam, or carbon dioxide.[2]

Chemical Properties

Soluble in about 500 parts of water. Soluble in alcohol, benzene, ether, and oils. Volatile with steam.[1]

Hazardous Reactions

Alkali. Heating nitrobenzene with sodium hydroxide, potassium hydroxide, and small quantities of water may result in an explosion.[3,4,5]

Aluminum Chloride. Mixtures of aluminum chloride and nitrobenzene may decompose explosively.[6] The presence of 5% phenol in the mixture may cause a violent explosion.[7]

Aniline, Glycerol, and Sulfuric Acid. The preparation of quinoline by the Skraup reaction may be violent unless adequate stirring and temperature control are maintained.[8]

Oxidants. Mixtures of nitrobenzene with sodium chlorate,[9] nitric acid,[10] and nitric acid and water[10] are powerful explosives. Nitration of nitrobenzene with nitric and sulfuric acids may be explosive without adequate temperature control.[10] A mixture of nitrobenzene and nitrogen dioxide forms a highly explosive liquid.[11]

Phosphorus Pentachloride. A solution in nitrobenzene decomposes violently above 120°C.[12]

Potassium. Mixtures of traces of potassium or sodium–potassium alloy and nitrobenzene are extremely shock sensitive.[13]

Physiological Properties and Health Hazards

Liquid causes eye damage. Breathing vapor may result in chest pain, breathing difficulty, and finally unconsciousness. Swallowing or skin absorption may result in vomiting and unconsciousness.[14] Onset may be delayed 24 hours or longer. Chronic effects include liver damage.[15] Avoid breathing vapor. Avoid contact with skin and eyes.[14] TLV-TWA 1 ppm (5 mg/m^3) (skin).[16]

Spillage Disposal

Instruct others to maintain a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves.[17] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to an appropriate container labeled for disposal by burning in a furnace equipped with afterburner and scrubber. Spill site should be washed thoroughly with water and soap or detergent.[14,15,18]

Waste Disposal

Place in nonhalogenated solvent disposal container for recycling or disposal by burning in a furnace equipped with afterburner and scrubber.[15]

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6. B 37.
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16. ACGIH 44.
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4-NITROBIPHENYL C_6H_5-$C_6H_4NO_2$

ANIMAL CARCINOGEN

Synonyms

p-Nitrobiphenyl, *p*-nitrodiphenyl.

Physical Properties

Yellow needles; mp, 113.7°C.[1]

Chemical Properties

Insoluble in water; slightly soluble in cold alcohol but more readily soluble in hot alcohol; soluble in chloroform and ether.[1]

Physiological Properties and Health Hazards

Highly toxic.[2] An experimental carcinogen via oral route.[3] Induces bladder cancer in dogs.[4] TLV, no exposure permitted.[5]

Spillage Disposal

Wear butyl rubber gloves, protective laboratory coat, and eye protection.

On skin. Wash with strong soap solution immediately. Rinse well.

Spills. Scoop up solid into appropriately labeled container. Dispose of by burning; see waste disposal procedure.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[2]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood.

For every 10 mg of 4-nitrobiphenyl, add 10 mL of glacial acetic acid. Mix to dissolve. Add 10 mL of 2 M sulfuric acid (prepared by cautiously adding 2 mL of concentrated acid to 16 mL of cold water) and, while stirring, 165 mg of zinc powder. Continue stirring and let stand overnight. With continuous stirring, add 10 mL of 0.2 M potassium permanganate (prepared by dissolving 0.3 g of solid potassium permanganate in 10 mL of water). Let stand, while stirring, overnight (at least 10 hours). Decolorize, if necessary, with sodium metabisulfite or ascorbic acid. Neutralize by careful addition of 5 M sodium hydroxide solution (prepared by slowly adding 20 g of NaOH to 100 mL of cold water). Wash the resulting solution into the drain.[6]

REFERENCES

1. Merck 6626.
2. Ald 2568C
3. Sax 2524.
4. Cas 64, 7.
5. ACGIH 44.
6. Cas 64, 51.

2-NITROPROPANE $CH_3CHNO_2CH_3$

ANIMAL CARCINOGEN

Physical Properties

Colorless liquid; mp, –93°C; bp, 120.3°C.[1,2]

Fire Hazard

Flash point, 28°C; ignition temperature, 428°C. Extinguish with water, dry chemical, or carbon dioxide.[3]

Chemical Properties

Slightly soluble in water[1]; miscible with organic solvents.[2]

Hazardous Reactions

Amines and Heavy Metal Oxides. Contact with amines and mercury or silver oxide may lead to unstable salts analogous to nitromethane.

Carbon and Hopcalite. Carbon-containing respirators should not be used in high-vapor concentrations because heat of absorption of 2-nitropropane is very high. Ignition may occur if hopcalite catalyst is present in respirator cartridge.[4]

Physiological Properties and Health Hazards

Animal carcinogen; reasonably anticipated to be a human carcinogen.[5] TLV-TWA 10 ppm (36 mg/m^3).[6]

Spillage Disposal

Wear butyl rubber gloves,[7] eye protection, and laboratory coat. Provide good ventilation. Eliminate all sources of ignition.

On skin. Wash immediately with soap solution. Rinse thoroughly.

On clothing. Remove clothing immediately and place in a fume hood. Wash clothing before wearing again. Shoes are difficult to decontaminate and may have to be discarded and burned.[2]

Spills. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container, package, and label for disposal by burning. Wash site with soap solution.[8]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning.[9]

REFERENCES

1. Merck 6661.
2. ITI 465.
3. NFPA 49.
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5. NIEHS III–164.
6. ACGIH 45.
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9. Ald 1377B.

NITROSAMINES R_2NNO

ANIMAL CARCINOGENS

See individual entries for N-Nitrosodimethylamine, N-Nitroso-N-ethylurea, and N-Nitroso-N-methylurea.

Physiological Properties and Health Hazards

These compounds are suspected of causing cancers of the lung, nasal sinuses, brain, esophagus, stomach, liver, bladder, and kidney.[1]

Spillage Disposal

Wear nitrile rubber gloves, protective laboratory coat, self-contained breathing apparatus, eye protection, and protective shoes.

On skin. Wash with strong soap solution immediately. Rinse well.

Contaminated gloves, clothing, and shoes. Remove and clean at once or destroy by burning.

Spills. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into an appropriately labeled container for disposal by burning (see waste disposal procedure). Wash the site of spillage thoroughly with strong soap solution.[2,3]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[3]

Milligram Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Milligram quantities may be destroyed by treatment with a 3% solution of hydrobromic acid in acetic acid at room temperature. Dissolve 1 mg of nitrosamine in 1–2 mL of a suitable solvent (i.e., dichloromethane; preferably not water or dimethylsulfoxide). Carefully add 10 mL of a solution of 3% hydrogen bromide in glacial acetic acid (add 1 mL of reagent 30% hydrobromic acid in glacial acetic acid to 9 mL of glacial acetic acid). Allow to react for at least 2 hours. Neutralize with 5 M sodium hydroxide solution (dissolve 20 g of NaOH in 100 mL of water). Wash the resulting reaction mixture into the drain.[4,5]

Gram Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Prepare a solution of 0.05 mol of the dialkylnitrosamine in 500 mL of water in a 2-L, three-necked, round-bottom flask equipped with a stirrer and an outlet tube leading to the back of the fume hood. Cool in an ice bath. Add 500 mL of 1 M sodium hydroxide solution (20 g of NaOH dissolved in 500 mL of water). Gradually add in portions, 50 g of 50/50 aluminum–nickel alloy over a period of about 1 hour. The flask neck through which it is added is stoppered between additions. The reaction is highly exothermic and accompanied by frothing that can cause the reaction mixture to foam out of the flask if the alloy is added too rapidly. Continue stirring for 3 hours in the ice bath and then for 20 hours at room temperature. Allow the finely divided black nickel to settle. The aqueous phase is decanted, neutralized, and washed into the drain. Do not allow the nickel powder to dry, as it is pyrophoric.

The residual nickel is suspended in 200 mL of water in a 2-L, three-necked, round-bottom flask, and 800 mL of 1 N hydrochloric acid (prepared by slowly adding 66 mL of concentrated acid to 734 mL of cold water) is added gradually while stirring. Continue stirring until the nickel has dissolved. The solution is slowly basified with sodium hydroxide to precipitate nickel hydroxide. Collect the precipitate and package it for recycling or disposal in a secure landfill. Wash the aqueous solution into the drain.[4]

Reactions for Spillage and Waste Disposal

$$R_2NNO + HBr \rightarrow R_2NH + NOBr$$

$$R_2NNO \xrightarrow[NaOH]{Al-Ni} R_2NH$$

REFERENCES

1. Sax 2556; NIEHS III–167.
2. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.
3. Ald 2599B.
4. PP 71, PPL 298.
5. Cas 43, 17.

N-NITROSODIMETHYLAMINE $(CH_3)_2NNO$

ANIMAL CARCINOGEN, HIGHLY TOXIC

Physical Properties

Yellow liquid; bp, 151–153°C.[1]

Chemical Properties

Very soluble in water, alcohol, and ether.[1]

Physiological Properties and Health Hazards

Severe poison.[2] Has caused fatal liver disease in humans.[3] Animal carcinogen; reasonably anticipated to be a human carcinogen.[4] Exposure by all routes should be carefully controlled to the lowest possible levels.[2]

Spillage Disposal

Wear nitrile rubber gloves, eye protection or all-purpose canister respirator, and laboratory coat. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into an appropriately labeled container for disposal by burning (see Waste Disposal).[2,5]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[2]

Milligram Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Milligram quantities may be destroyed by treatment with a 3% solution of hydrobromic acid in acetic acid at room temperature. Dissolve 1 mg of N-nitrosodimethylamine in 1–2 mL of a suitable solvent (i.e., dichloromethane; preferably not water or dimethylsulfoxide). Carefully add 10 mL of a solution of 3% hydrogen bromide in glacial acetic acid (add 1 mL of reagent 30% hydrobromic acid in glacial acetic acid to 9 mL of glacial acetic acid). Allow to react for at least 2 hours. Neutralize with 5 M sodium hydroxide solution (dissolve 20 g of NaOH in 100 mL of water). Wash the remaining reaction mixture into the drain.[6,7]

Gram Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Prepare a solution of 0.05 mol (3.7 g, 3.7 mL) of the N-nitrosodimethylamine in 500 mL of water in a 2-L, three-necked, round-bottom flask equipped with a stirrer and an outlet tube leading to the back of the fume hood. Cool in an ice bath. Add 500 mL of 1 M sodium hydroxide (20 g of NaOH dissolved in 500 mL of water). Gradually add portions of 50 g of 50/50 aluminum–nickel alloy over a period of about 1 hour, stoppering the flask neck through which it is added between additions. The reaction is highly exothermic and accompanied by frothing that can cause the reaction mixture to foam out of the flask if the alloy is added too rapidly. Continue stirring for 3 hours in the ice bath and then for 20 hours at room temperature. The finely divided black nickel is allowed to settle, and the aqueous phase is decanted, neutralized, and flushed down the drain with at least 50 times its volume of water. Do not allow the nickel powder to dry, as it is pyrophoric.

The residual nickel is suspended in 200 mL of water in a 2-L, three-necked, round-bottom flask, and 800 mL of 1 N hydrochloric acid (slowly add 66 mL of concentrated acid to 734 mL of cold water) is gradually added while stirring. Stirring is continued until the nickel has dissolved. The solution is slowly basified with sodium hydroxide to precipitate nickel hydroxide. The precipitate is collected and packaged for recycling or disposal in a secure landfill. The aqueous solution is washed into the drain.[6]

Reactions for Spillage and Waste Disposal

$$(CH_3)_2NNO + HBr \rightarrow (CH_3)_2NH + NOBr$$

$$(CH_3)_2NNO \xrightarrow[NaOH]{Al/Ni} (CH_3)_2NH$$

dimethylamine

REFERENCES

1. Merck 6671.
2. Ald 2599B, ACGIH 45.
3. Sax 2564.
4. NIEHS III–168.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.,* 29.7.
6. PP 71; PPL 298.
7. Cas 43, 17.

N-NITROSO-N-ETHYLUREA $C_3H_7N_3O_2$

ANIMAL CARCINOGEN

Synonyms

N-Ethyl-N-nitrosourea.

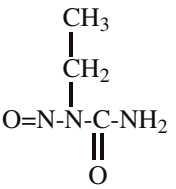
Physical Properties

Yellow-pink crystals; mp, 103–104°C (dec).[1]

Chemical Properties

Sparingly soluble in water; soluble in polar organic solvents; sensitive to humidity and light.[1]

Structure



Hazardous Reactions

At alkaline pH, it decomposes to diazoethane, an irritant and toxic gas.[1]

Physiological Properties and Health Hazards

Animal carcinogen. A single dose can often be carcinogenic. This compound is also both mutagenic and teratogenic. Reasonably anticipated to be a human carcinogen.[1–3]

Spillage Disposal

Solutions. Wear protective gloves, laboratory coat, and goggles. Absorb the spill on paper towels. Place the towel in a beaker in the fume hood. Immediately pour over the spill area a *freshly* prepared solution of 4.7 g of potassium permanganate in 100 mL of 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) to thoroughly wet the contaminated area, and let it stand overnight. Fill the beaker with acid permanganate solution and allow it to stand at room temperature overnight. Dilute the mixture with water and neutralize by cautiously adding 10% aqueous sodium hydroxide solution. Decant or filter the liquid into the drain with water. Discard the solid as normal refuse. Add a 5% solution of ascorbic acid to the spill area and allow it to react for 15 minutes. Add solid soda ash to the decontaminated surface. Remove the decontamination mixture by absorbing on paper towels.[4]

Solid. Cover the area of the spill with a 0.3 M solution of potassium permanganate in 3 M sulfuric acid (prepared as for solution spills). Allow to stand overnight. Absorb the liquid residue on a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the solid into a container and, in the fume hood, add to a beaker of cold water. If necessary, neutralize the solution by the cautious addition of soda ash. Decant the liquid into the drain with water. Treat the solid as normal refuse.[4,5]

Waste Disposal

Wear protective gloves, laboratory coat, and goggles. Work in the fume hood. Add sufficient 3 M sulfuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water) to the waste to produce a solution not more than 5% in N-nitroso-N-ethylurea. Add potassium permanganate to give a 0.3 M solution (0.5 g/10 mL). Stir the mixture at room temperature for 12 hours. Neutralize by cautiously adding 10% aqueous sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water) until a colorless liquid is formed. Wash into the drain with water.[4,5]

REFERENCES

1. IARC 1, 135; see also IARC 5.7 (1987).
2. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides*, No. 55, IARC Scientific Publications, Lyon, 1983, p. 3.
3. NIEHS III–171.
4. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides*, No. 55, IARC Scientific Publications, Lyon, 1983, p. 31.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

N-NITROSO-N-METHYLUREA $C_2H_5O_2N_3$

ANIMAL CARCINOGEN

Synonyms

N-Methyl-N-nitrosourea, MNU.

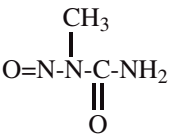
Physical Properties

Pale yellow crystals; mp, 124°C (dec).[1]

Chemical Properties

Sparingly soluble in water; soluble in polar organic solvents. Highly reactive.[1]

Structure



Hazardous Reactions

At alkaline pH, it decomposes to diazomethane, a highly irritant and toxic gas.[1]

Physiological Properties and Health Hazards

Animal carcinogen. A single dose can often be carcinogenic. This compound is also both mutagenic and teratogenic. Reasonably anticipated to be a human carcinogen.[1–3] It has been known to cause contact dermatitis. LD_{50} (oral, rat) 110 mg/kg.[4]

Spillage Disposal

Solutions. Wear protective gloves, laboratory coat, and goggles. Absorb the spill on paper towels. Place the paper towels in a beaker in the fume hood and cover with a solution of 4.7 g of potassium permanganate in 3 M sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water). Allow it to stand at room temperature overnight. Neutralize the solution by cautiously adding 10% aqueous sodium hydroxide. Decant the liquid into the drain with a large volume of water. Discard the solid as normal refuse.[5]

Solid. Cover the spill area with a 0.3 M solution of potassium permanganate in 3 M sulfuric acid (prepared as for solution spills). Allow it to stand overnight. Absorb the liquid residue on a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the solid into a container and, in the fume hood, add to a beaker of cold water. If necessary, neutralize the solution by the cautious addition of soda ash. Decant the liquid into the drain with water. Treat the solid as normal refuse.[5,6]

Waste Disposal

Wear protective gloves, laboratory coat, and goggles. Work in a fume hood. Add sufficient 3 M sulfuric acid (1.7 mL of concentrated sulfuric acid slowly added to 8.3 mL of cold water) to the waste to produce a solution not more than 5% in N-nitroso-N-methylurea. Add potassium perman-

ganate to give a 0.3 M solution (0.5 g/10 mL). Stir the mixture at room temperature for 12 hours. Neutralize by cautiously adding 10% aqueous sodium hydroxide solution, then, while stirring, add a saturated aqueous solution of sodium bisulfite (approximately 10 g of sodium bisulfite per 35 mL of water), until a colorless liquid is formed. Wash into the drain with water.[5,6]

REFERENCES

1. IARC 1, 125; see also IARC S 7 (l987).
2. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides*, No. 55, IARC Scientific Publications, Lyon, 1983, p. 3.
3. NIEHS III–171.
4. Sax 2365.
5. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some N-Nitrosamides*, No. 55, IARC Scientific Publications, Lyon, 1983, p. 31.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

NITROSOPHENOLS HOC_6H_4NO

SKIN IRRITANTS

Physical Properties

p-Nitrosophenol exists as pale yellow needles; browns at 126°C; dec, 144°C.[1]

Fire Hazard

p-Nitrosophenol burns explosively.[2]

Chemical Properties

p-Nitrosophenol is moderately soluble in water; soluble in alcohol, ether, and acetone.[1]

Hazardous Reactions

Technical grade of *p*-nitrosophenol may explode on storage.[1] *o*-Nitrosophenol explodes on heating,[3] while *p*-nitrosophenol may ignite spontaneously if nitrates were present in the sodium nitrite used for its preparation.[4]

Acids. Contact with concentrated acids causes *o*-nitrosophenol to explode.[3] The addition of sulfuric acid to *p*-nitrosophenol containing nitrate impurities results in effervescence or ignition and can be used as a test for the presence of these impurities.[4]

Physiological Properties and Health Hazards

p-Nitrosophenol can cause skin irritation and sensitization.[1]

Spillage Disposal

Wear nitrile rubber gloves, protective laboratory coat, and eye protection. Spills on skin should be immediately washed with strong soap and then rinsed well. Contaminated clothing such as gloves and shoes should be removed and cleaned at once or destroyed by burning.

Spills. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriately labeled container for disposal by burning in a furnace equipped with afterburner and scrubber.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.

Small Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Milligram quantities may be destroyed by treatment with a 3% solution of hydrobromic acid in acetic acid at room temperature. Dissolve 1 mg of nitrosophenol in 1–2 mL of a suitable solvent (i.e., dichloromethane; preferably not water or dimethylsulfoxide). Carefully add 10 mL of a solution of 3% hydrogen bromide in glacial acetic acid (add 1 mL of reagent 30% hydrobromic acid in glacial acetic acid to 9 mL of glacial acetic acid). Allow to react for at least 2 hours. Neutralize with 5 M sodium hydroxide solution (dissolve 20 g of NaOH in 100 mL of water). Wash the remaining reaction mixture into the drain.[7,8]

Reactions for Spillage and Waste Disposal

$$HOC_6H_4NO + HBr \rightarrow HOC_6H_5 + NOBr$$

REFERENCES

1. Merck 6676.
2. Sax 2580.
3. Baeyer, A. et al., *Chemische Berichte*, 35, 3037, 1902.
4. B 606.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
6. Ald 2603B.
7. PP 71; PPL 45.
8. Cas 43, 17.

OSMIUM TETROXIDE OsO_4

POISONOUS, HARMFUL VAPOR, CAUSES BURNS

Synonyms

Osmic acid, osmic acid anhydride, perosmic oxide.

Physical Properties

Pale yellow crystals with pungent odor; mp, 40°C[1]; bp, 180°C.[2]

Chemical Properties

Soluble in water. Vapor is poisonous! Safeguards necessary when opening container.[1]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system. Vapor, solid, and solution irritate the eyes and burn the eyes and skin. Swallowing causes severe internal irritation and damage. Prolonged exposure to vapor causes disturbances of vision, while continued skin contact results in dermatitis and ulceration. Prevent inhalation of vapor. Prevent contact with eyes and skin.[3] TLV-TWA 0.0002 ppm (0.0016 mg/m^3); TLV-STEL 0.0006 ppm (0.0047 mg/m^3).[4]

Spillage Disposal

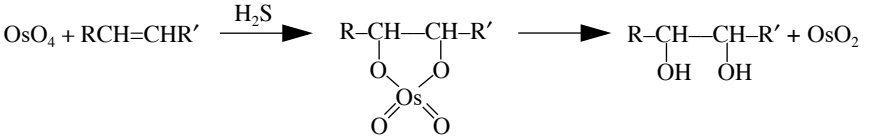
Wear face shield and goggles, laboratory coat, and nitrile rubber gloves. Follow waste disposal procedure.

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Reaction Mixtures. Osmium tetroxide can be reduced to the dioxide by reacting it with an olefin to form the osmate ester and then bubbling hydrogen sulfide through the solution. The black precipitate of osmium dioxide is removed by filtration. Package OsO_2 for disposal or recycle.[5]

Reactions for Spillage and Waste Disposal



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2. ITI 480.
3. Lux 513.
4. ACGIH 45.
5. F & F 1, 761.
6. Ald 2658C.

OXALIC ACID HOOCCOOH

POISON, CORROSIVE

Physical Properties

Colorless crystals. Dihydrate mp, 101–102°C; anhydrous mp, 189.5°C (dec); sublimes at 157°C.[1]

Chemical Properties

Soluble in water, alcohol, and glycerol; slightly soluble in ether; insoluble in benzene, chloroform, and petroleum ether.[1]

Hazardous Reactions

Silver. Silver oxalate explodes at 140°C.[2]

Sodium Chlorite and Water. Addition of water to a mixture of sodium chlorite and oxalic acid results in the formation of highly explosive chlorine dioxide.[3]

Physiological Properties and Health Hazards

Caustic and corrosive to skin and mucous membranes.[1] Dust irritates the respiratory system and eyes. Splashing solution into the eyes also causes irritation.[4] Swallowing can cause severe internal pain, renal damage, convulsions, coma, and death from cardiovascular collapse.[1] Avoid contact with eyes and skin.[4] TLV-TWA 1 mg/m^3; TLV-STEL 2 mg/m^3.[5]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and a face shield. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Wash the solution into the drain. Treat the solid residue as normal refuse.[4,6]

Waste Disposal

Package Lots. Oxalic acid can be placed in a separate labeled container for recycling or disposal by burning.[7,8]

Small Quantities. Oxalic acid is decomposed to carbon dioxide, carbon monoxide, and water by heating in concentrated sulfuric acid. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, oxalic acid (5 g) is added to 25 mL of concentrated sulfuric acid in a 100-mL round-bottom flask. Using a heating mantle, the mixture is maintained at 80–100°C for 30 minutes. The sulfuric acid can be re-used for the same procedure because the only nonvolatile product of the decomposition is a small quantity of water. Otherwise, allow the reaction mixture to cool to room temperature, and then slowly and carefully pour the sulfuric acid into a pail of cold water (or cold water and ice), neutralize with sodium carbonate, and wash into the drain.[6]

Oxalic acid salts can be decomposed in a manner analogous to oxalic acid.[6]

Reactions for Spillage and Waste Disposal

$$HOOCCOOH + H_2SO_4 \rightarrow H_2O + CO_2 + CO + H_2SO_4$$

REFERENCES

1. Merck 6980.
2. B 3.
3. B 987.
4. Lux 515.
5. ACGIH 46.
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7. ITI 481.
8. PP 66.

OXALYL CHLORIDE ClCOCOCl

TOXIC BY INHALATION, CAUSES BURNS

Synonyms

Oxalyl dichloride.

Physical Properties

Colorless fuming liquid with penetrating odor; bp, 63–64°C.[1]

Chemical Properties

Reacts vigorously with water forming hydrochloric and oxalic acids.[2]

Hazardous Reactions

Potassium and Potassium-Sodium. Mixtures are shock sensitive and explode very violently.[2,3]

Physiological Properties and Health Hazards

Vapor severely irritates the respiratory system and eyes. Liquid irritates eyes and skin and may cause burns. Assumed to be very irritating and poisonous if swallowed. Avoid breathing vapor. Prevent contact with skin and eyes.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the oxalyl chloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain flushing with 50 times its volume of water. Treat the solid residue as normal refuse.[2,4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning in a furnace equipped with afterburner and scrubber.[5]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Convert the chloride to oxalic acid by slowly adding it to cold water (3 mL water for each 1 mL chloride). Allow to stand for 1 hour. Slowly, carefully, and while stirring, add concentrated sulfuric acid (10 mL acid for each 1 mL of starting chloride) to the aqueous solution. Heat the mixture to 80–90°C for 30 minutes using a heating mantle. The resulting sulfuric acid is diluted but otherwise pure. Either re-use it, or allow it to cool to room temperature, and then add it slowly and carefully to a pail of cold water. Neutralize with sodium carbonate and wash into the drain.[4]

Reactions for Spillage and Waste Disposal

$$ClCOCOCl + 2H_2O \rightarrow HOOCCOOH + 2HCl$$

$$HOOCCOOH + H_2SO_4 \rightarrow H_2O + CO_2 + CO + H_2SO_4$$

REFERENCES

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2. Lux 515.
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4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ*., 62, A93, 1985; Armour, M.A., *J. Chem. Educ*., 65, A64, 1988; *Haz. Mat. Spills Tec*., 29.5.
5. Ald 2662D.

OZONE O_3

TOXIC BY INHALATION, EXPLOSIVE

Physical Properties

Colorless gas or dark blue liquid with characteristic odor; bp, −111.9°C.[1]

Fire Hazard

Solid or liquid is highly explosive.[2] May ignite or explode in aqueous solution or in contact with organic substances.[3]

Chemical Properties

Powerful oxidizing agent. Unstable solutions of ozone explode on warming.[1]

Hazardous Reactions

Pure solid or liquid is highly explosive; evaporation of a solution in liquid O_2 results in explosion[4]; liquid explodes on contact with organic liquids and oxidizable materials[2]; safe handling techniques have been described,[5–7] and interaction with organic compounds has been reviewed.[8]

Alkenes. Products can be explosive.[4,9]

Aromatic Compounds. Products can be explosive.[10]

Bromine. Interaction can be explosive above 20°C and minimum critical pressure.[11]

Combustible Gases. Contact with CO, NO, N_2O_4, NH_3, PH_3, dicyanogen, or ethylene causes immediate explosion at 0°C or −78°C.[4,10,12]

Diethyl Ether. Contact produces explosive diethyl peroxide.[10]

Hydrogen Bromide. Explosive reaction even at −104°C.[13]

Isopropylidene Compounds. Ozonolysis of any compound containing an isopropylidene group may yield the highly explosive trimeric acetone peroxide.[14]

Silica Gel. Ozone absorbed by silica gel is a potential explosion hazard below −100°C if organic material is present.[15]

Physiological Properties and Health Hazards

Gas strongly irritates the upper respiratory system and may cause headache. High concentrations have caused death in animals by lung congestion. Avoid inhalation.[2] TLV-TWA 0.1 ppm (0.2 mg/m^3) (light work).[16]

Waste Disposal

In the fume hood, slowly release wastes to air.[3]

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3. ITI 486.
4. B 1418.
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7. Streng, A.G., *Explosivstoffe*, 8, 225, 1960.
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PALLADIUM Pd

CATALYST FORMS ARE PYROPHORIC WHEN DRY

See also Hydrogenation Catalysts.

Physical Properties

Silver-white metal. Occurs also as black powder and as spongy masses that can be compressed to a compact mass; mp, 1555°C; bp, 3167°C.[1]

Hazardous Reactions

Air and Hydrogen. Pd catalysts prepared on high surface-area supports cause catalytic ignition of H_2/air or solvent/air mixtures[2,3]; Pd on charcoal, when thoroughly dry, is extremely pyrophoric.[4]

Formic Acid. Addition of Pd/C to 98% acid is extremely hazardous.[5]

Sodium Tetrahydroborate. Addition of Pd/C to tetrahydroborate solution may cause ignition.[6]

Sulfur. Sulfur and Pd react with incandescence.[7]

Spillage Disposal

See waste disposal procedure.

Waste Disposal

Spent catalysts should be purged from hydrogen and washed free from organics with water before storage in water-wet conditions. Under no circumstances should any attempt be made to dry a spent catalyst.

Spent catalysts should be kept under water and recycled.[3,8,9]

REFERENCES

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3. Mozingo, R., *Org. Synth.*, Coll. Vol. 3, 687, 1955.
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5. B 151.
6. B 1443.
7. Mellor, Vol. 15, 149, 527, 627, 696, 1942.
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PARATHION $C_{10}H_{14}NO_5PS$

INSECTICIDE, ACARICIDE

Synonyms

Ethyl parathion, DNTP, O,O-diethyl-O-*p*-nitrophenylphosphorothioate, SNP, E-605, AC 3422, ENT 15108, Alkron, Alleron, Aphamite, Etilon, Folisol, Fosferno, Niran, Paraphos, Rhodiatox, Thiophor.

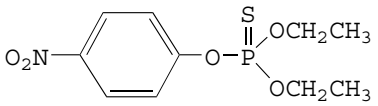
Physical Properties

Pale yellow liquid; mp, 6°C; bp, 375°C.[1]

Chemical Properties

Freely soluble in alcohols, esters, ethers, ketones, and aromatic hydrocarbons. Practically insoluble in water (20 ppm), petroleum ether, kerosene, and the usual spray oils. Incompatible with substances having a pH higher than 7.5.[1] Darkens on exposure to sunlight.[2]

Structure



Physiological Properties and Health Hazards

Highly toxic. Causes skin, eye, and respiratory system mutation. Acute effects include anorexia, nausea, vomiting, diarrhea, excessive salivation, pupillary constriction, broncho constriction, muscle twitching, convulsions, coma, respiratory failure. Special precautions are necessary to prevent inhalation and skin contamination.[1] LD_{50} (oral rat) 13 mg/kg. LD_{50} (dermal rat) 21 mg/kg. LC_{50} (rainbow trout, 96 hours) 1.5 mg/L.[3] TLV-TWA 0.1 mg/m^3.[4]

Waste Disposal

Wear eye protection and gloves. To each 1 mL of parathion, add 50 mL of 3 M sulfuric acid (8.5 mL of concentrated acid added to 41.5 mL of cold water) and 2.5 g of potassium permanganate. The mixture is stirred at room temperature for 4 hours. Saturated aqueous sodium bisulfite is added until the solution is colorless. The solution is neutralized by the addition of soda ash (foaming may occur) or 5% aqueous sodium hydroxide. The clear solution is washed into the drain with water.[5]

REFERENCES

1. Merck 7105
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PENTACHLOROPHENOL C_6Cl_5OH
and SODIUM PENTACHLOROPHENATE C_6Cl_5ONa

SERIOUS RISK OF POISONING BY INHALATION, SWALLOWING, OR SKIN CONTACT; CAUSES IRRITATION OF SKIN AND EYES

Physical Properties

Pentachlorophenol is a colorless to yellow crystalline solid with a phenolic (carbolic) odor; mp, 190–191°C.[1] The sodium salt is a buff powder or flaked solid.[1]

Chemical Properties

Pentachlorophenol is insoluble in water. Sodium salt is soluble in water.[12]

Physiological Properties and Health Hazards

Dust of both compounds irritates the nose and eyes. Swallowing or absorption through the skin may cause feverishness and weakness, and finally convulsions and unconsciousness. Avoid breathing dust. Prevent contact with skin and eyes.[1] TLV-TWA 0.5 mg/m^3 (skin).[3]

Spillage Disposal

Wear goggles, laboratory coat, and Viton® gloves.[4] Sweep up spills into metal container. Follow waste disposal. Wash site with soap solution.[1,5]

Waste Disposal

Place in a separate labeled container for disposal by burning.

Dissolve the compound in a flammable solvent and burn in furnace equipped with afterburner and scrubber.[5]

REFERENCES

1. Lux 519.
2. Merck 7180.
3. ACGIH 47.
4. LSS.
5. ITI 493.

PERACETIC ACID CH_3CO_3H

EXPLOSIVE, STRONG IRRITANT

Physical Properties

Liquid with acrid odor. Explodes violently on heating to 110°C.[1]

Fire Hazard

Explodes violently on heating and is shock sensitive. May ignite or explode if mixed with readily oxidizable, organic, or flammable materials. Flash point, 41°C; ignition temperature, 200°C. Use water to extinguish fire.[2]

Chemical Properties

Freely soluble in water, alcohol, ether, and sulfuric acid; stable in dilute aqueous solution. Strong oxidizing agent.[1]

Hazardous Reactions

Explodes violently at 100°C[3]; safe procedures for the preparation of anhydrous peracetic acid in chloroform or ester solvents have been recorded.[4]

Safe Handling Procedures

When peracetic acid is used in organic synthesis, the following precautions are recommended. All laboratory reactions and subsequent operations should be run behind a safety shield. The peracetic acid should be added to the organic material (never the reverse), and the rate of addition should be slow enough so that the peracetic acid reacts as rapidly as it is added and no unreacted excess is allowed to build up. The reaction mixture should be stirred efficiently while the peracetic acid is being added, and cooling facilities should be provided because most of the reactions of peracetic acid are exothermic. New or unfamiliar reactions, particularly those run at high temperatures, should be tried first on a very small scale. Reaction products should never be recovered from the final reaction mixture by distillation until all residual active oxygen compounds have been destroyed. This includes the hydrogen peroxide present in commercial 40% peracetic acid as well as the unreacted peracid. Decomposition may be readily accomplished by catalytic quantities of activated carbon or ferric sulfate added at 25–50°C. The course of the decomposition may be followed by titration.[5]

Physiological Properties and Health Hazards

Strongly irritating to skin and eyes.[1] Highly toxic. LD_{50} (oral, rat) 1540 mg/kg.[6]

Spillage Disposal

Wear a face shield and goggles, butyl rubber gloves, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic scoop, shovel the mixture into a plastic container and transport to the fume hood. Slowly add to a pail of cold water. Avoid contact with metal. To the aqueous solution add an ice-cold concentrated aqueous solution of sodium bisulfite until a drop of the mixture on starch-iodide paper does not produce a blue color. Test the pH of the solution and neutralize with 5% aqueous hydrochloric acid or sodium carbonate as appropriate. Decant the solution to the drain. Treat the solid residue as normal refuse.[6,7]

Waste Disposal

Wear a face shield, goggles, butyl rubber gloves, and a laboratory coat. In the fume hood behind a shield, slowly add the peracetic acid to a large volume of ice-cold, saturated sodium bisulfite solution. Test a drop of the solution with starch-iodide paper. If a blue color is produced, add more saturated sodium bisulfite solution. Neutralize the solution with 5% aqueous hydrochloric acid or with sodium carbonate as appropriate. Wash into the drain.[7]

Reactions for Spillage and Waste Disposal

$$CH_3CO_3H + NaHSO_3 \rightarrow CH_3CO_2H + NaHSO_4$$

REFERENCES

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6. ITI 495.
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PERBENZOIC ACID $C_6H_5CO_3H$

EXPLOSIVE

Synonyms

Peroxybenzoic acid, benzoyl hydroperoxide.

Physical Properties

Leaflets from benzene. Acrid odor; mp, 41–43°C. Sublimes in desiccator; volatile with steam.[1]

Fire Hazard

Explodes on heating.[2]

Chemical Properties

Insoluble in cold water; sparingly soluble in petroleum ether; freely soluble in other organic solvents.[1]

Hazardous Reactions

Explodes weakly on heating[2]; evaporation of solvent from solutions should be avoided if possible.[3]

Physiological Properties and Health Hazards

Moderately irritating to the skin, eyes, and mucous membranes via oral and inhalation routes.[4]

Spillage Disposal

Wear a face shield, goggles, nitrile rubber gloves, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic scoop, shovel the mixture into a plastic container, and transport to the fume hood. Avoid contact with metal. Slowly add the mixture to a large volume of ice-cold, concentrated sodium bisulfite solution. Test a drop of the solution on starch-iodide paper. If a blue color results, add more bisulfite solution. Neutralize with 5% hydrochloric acid or sodium carbonate as appropriate. Decant the solution to the drain. Treat the solid residue as normal refuse.[5]

Waste Disposal

Wear nitrile rubber gloves, a face shield, goggles, and a laboratory coat. In the fume hood, behind a shield, add the perbenzoic acid to a large volume of ice-cold saturated sodium bisulfite solution. Test a drop of the solution with starch-iodide paper. If a blue color is produced, add more saturated bisulfite solution. Neutralize the solution with 5% hydrochloric acid or sodium carbonate as appropriate and wash into the drain.[5]

Reactions for Spillage and Waste Disposal

$$C_6H_5CO_3H + NaHSO_3 \rightarrow C_6H_5CO_2H + NaHSO_4$$

REFERENCES

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PERCHLORATES

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIAL

See individual entries for Magnesium Perchlorate, Pyridinium Perchlorate, and Silver Perchlorate.

Physical Properties

The perchlorates of ammonium, magnesium, sodium, and potassium are colorless crystalline solids; all, except that of potassium, are readily soluble in water. Ammonium perchlorate is explosive when dry.[1]

Fire Hazard

Mixtures of inorganic perchlorates with combustible materials are readily ignited; mixtures with finely divided combustible materials frequently react explosively. Organic perchlorates are self-contained explosives.[1]

Chemical Properties

See individual entries.

Hazardous Reactions

See fire hazard information.

Methyl, ethyl, and propyl perchlorates are highly explosive oils sensitive to shock, heat, and friction; all organic perchlorates are self-contained explosives; mixtures of inorganic perchlorates with combustible materials are readily ignited; mixtures with finely divided combustibles frequently react explosively.[2–6]

SAFE HANDLING OF PERCHLORATES

According to Schumacher,[3]

> Perchlorates appear to fall into two broad categories: those more[4] or less[5] sensitive to heat and shock. Included in the group of those qualitatively less sensitive are pure ammonium perchlorate, the alkali metal perchlorates, the alkaline earth perchlorates, and perchloryl fluoride. Among the more sensitive compounds are the pure inorganic nitrogenous perchlorates, the heavy metal perchlorates, fluorine perchlorate, the organic perchlorate salts, the perchlorate esters and mixtures of any perchlorates with organic substances, finely divided metals or sulfur. Any attempt to establish a more precise order of the degree of hazard to be expected from any given perchlorate seems unwarranted on the basis of data available. Each perchlorate system must be separately (and cautiously) evaluated.
>
> There do not appear to be any uniform recommendations for the safe handling of perchlorates which are generally applicable. A number of heavy metal and organic perchlorates, as well as hydrazine perchlorate (hydrazinium diperchlorate) and fluorine perchlorate, are extremely sensitive and must be handled with great caution as initiating explosives. Mixtures of any perchlorates with oxidizable substances are also highly explosive and must be treated accordingly. For all of these, it is essential to avoid friction, heating, sparks or shock from any source (as well as heavy metal contamination), and to provide suitable isolation, barricades and protective clothing for personnel.

"However," adds Schumacher, "the more common ammonium, alkali metal, and alkaline earth perchlorates are considerably less hazardous."

Synthesis of new inorganic or organic perchlorates should only be undertaken by an experienced, cautious, investigator who is familiar with the literature.

A simple test to evaluate impact sensitivity can be conducted by placing a crystal or two of the perchlorate on a steel block and striking with a hammer. The degree of noise and relative impact to produce the explosion can be roughly correlated with the impact sensitivity.

A simple thermal stability test can be conducted by placing a crystal or two on a hot plate and observing the time it takes to create a violent decomposition reaction. A gram of the material can be heated slowly in a loosely closed vial for more exact determination of thermal stability.

Physiological Properties and Health Hazards

The dust and strong solutions irritate the skin, eyes, and respiratory system. Swallowing causes irritation and internal damage. Avoid contact with combustible materials. Avoid contact with skin, eyes, and clothing.[1]

Waste Disposal

See individual entries.

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PERCHLORIC ACID $HClO_4$

CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE, CAUSES BURNS

Physical Properties

Colorless, volatile, very hygroscopic liquid; miscible with water; sold commercially only as 60–70%.[1]

Fire Hazard

Anhydrous acid (which may be formed with strong dehydrating agents) decomposes at ordinary temperatures and explodes on contact with most organic materials. Extinguish fire with water spray.[2]

Chemical Properties

Very hygroscopic. Combines vigorously with water with evolution of heat. Undergoes spontaneous and explosive decomposition. Very caustic.[1]

Hazardous Reactions

Exceptionally powerful oxidizing agent that readily forms explosive salts. Safe handling procedures have been described.[3,4]

Acetic Anhydride, Acetic Acid, and Organic Materials. Anhydrous $HClO_4$ is formed in the presence of acetic anhydride; the mixture is sensitive to shock and heat and may explode violently in the presence of organic material.[5,10]

Acetic Anhydride, Carbon Tetrachloride, and 2-Methylcyclohexanone. During acetylation of the enolized ketone, the 70% perchloric acid must be added last.[6]

Alcohols. Explosions occurred during reactions with traces of methanol-chloroform[7] or ethanol[9]; partial esterification of polyfunctional alcohols with the anhydrous acid gives liquids that may explode on pouring.[8]

Aniline and Formaldehyde. Explosive resin formed from mixture.[8]

Antimony or Bismuth. Treatment of antimony(III)[10] compounds or bismuth[11] can be very hazardous.

Carbon. Violent explosion occurs between the anhydrous acid and wood charcoal or carbon black.[12]

Cellulose and Derivatives. Hot concentrated acid or cold anhydrous acid ignites on paper, wood fiber, or sawdust; long-term contact of dilute acid with wood may cause fire; explodes on contact with cellulose acetate or benzyl cellulose.[13]

Dehydrating Agents. Dehydration of 72% acid by heating with H_2SO_4, P_2O_5, or H_3PO_3, or by distillation under reduced pressure yields anhydrous perchloric acid, which explodes after storage at room temperature for 30 days. Mixtures with excess cold acetic anhydride are particularly dangerous because they are sensitive to mechanical shock, heat, or the introduction of organic contaminants. Chloroform solution of the monohydrate exploded on contact with P_2O_5. Dichloromethane is a safer solvent for preparing anhydrous solutions.[5,10,13,14]

Deoxyribonucleic Acid. Hydrolysis using $HClO_4$ is hazardous.[15]

Diethyl Ether. Anhydrous acid and ether may explode on contact.[9]

Glycols and Glycol Ethers. Perchloric acid (70%) violently decomposes glycols and their ethers.[17]

Iodides. Anhydrous $HClO_4$ ignites on contact with NaI or HI.[9]

Ketones. Ketones may undergo violent decomposition in contact with 70% acid.[18]

Methanol and Triglycerides. Transesterification of triglycerides in methanol using $HClO_4$ is hazardous.[18]

Nitric Acid and Organic Matter. The use of the mixed acids to digest organic matter may result in explosions. Explosions with vegetable oil,[19] milk,[20] calcium oxalate precipitates from plants,[21] rat carcasses,[22] finely ground plant material,[21] and animal tissues[21] have been documented.

Nitric Acid, Pyridine, and Sulfuric Acid. Formation of pyridinium perchlorate presents an explosion hazard during wet-ashing of rare earth fluorides.[21]

Nitrogenous Epoxides. Traces of $HClO_4$ used for ring opening of nitrogenous epoxides caused explosion.[23]

Oleic Acid. Preparation of 1,4-octadecanolactone from oleic acid by heating with 70% $HClO_4$ at 115°C is potentially dangerous.[24]

Phosphine. Salt formed with 68% $HClO_4$ at −20°C is dangerously explosive.[25]

Sodium Phosphinate. No interaction in the cold, but violent explosion occurs on heating.[26]

Sulfoxides. Lower members of the series of salts formed with organic sulfoxides are explosive when dry.[16,27,28]

Thallium Triacetate. Pasty residue from evaporation of mixture of thallium triacetate, acetic acid, perchloric acid, and ethyl benzene exploded.[16]

Trichloroethylene. Violent reaction occurs with anhydrous $HClO_4$.[8]

Acid Handling

85% or Less Perchloric Acid

1. Use goggles for eye protection whenever the acid is handled.
2. Always transfer acid over a sink in order to catch any spills and afford a ready means of disposal.
3. In wet combustions with perchloric acid, treat the sample first with nitric acid to destroy easily oxidizable matter.
4. Any procedure involving heating of the perchloric acid must be conducted in a ventilated hood.
5. No organic materials should be stored in the perchloric acid hood.
6. Do not allow perchloric acid to come in contact with strong dehydrating agents (concentrated sulfuric acid, anhydrous phosphorus pentoxide, etc.).
7. Perchloric acid should be used only in standard analytical procedures from well-recognized analytical texts. (This does not apply to analytical research workers.)

Anhydrous Perchloric Acid (Greater than 85%)

1. Only experienced research workers should handle anhydrous perchloric acid. These workers must be thoroughly familiar with the literature on the acid.
2. A safety shield must be used to protect against a possible explosion, and the acid must be used in an appropriate hood with a minimum of equipment present. No extraneous chemicals should be present in the hood.
3. A second person should be informed of the intended use of anhydrous acid and be in the same room with the research worker using this extremely strong oxidizer.
4. Safety goggles, face shield, thick gauntlets, and rubber apron must be worn.
5. Only freshly prepared acid should be used.
6. Dispose of the unused anhydrous acid at the end of each day. (See waste disposal procedure for small quantities.)
7. Do not make any more anhydrous perchloric acid than is required for a single day's work.
8. Contact of the anhydrous acid with organic materials will usually result in an explosion.
9. Anhydrous acid showing discoloration should be disposed of immediately.

Physiological Properties and Health Hazards

Burns the eyes and skin severely. Assumed to cause severe internal irritation and damage if taken by mouth. Prevent contact with eyes and skin.[29]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection.

Perchloric acid spilled on the floor or bench top represents a hazard. It should not be mopped up, nor dry combustibles used to soak up the acid. The spilled acid should first be neutralized and then soaked up with rags or paper towels. The contaminated rags and paper towels must be kept wet to prevent combustion upon drying. They should be placed in a plastic bag and sealed and then placed in a flammable waste disposal can. If the spill can be rinsed down a chemical drain, neutralization of the wetted area is recommended, followed by additional rinsing.[16,30,31]

Waste Disposal

Package Lots. Original packages can be sent for disposal. Concentrated perchloric acid (especially greater than 60%) must be kept away from reducing agents and organic substances because it can react violently with them. Dilute perchloric acid is not reduced by common reducing agents such as bisulfite, iron, or zinc.

Small Quantities. Perchloric acid is best disposed of by stirring it gradually into enough cold water to make its concentration less than 0.1%, neutralizing it with aqueous sodium hydroxide, and washing the solution into the drain.[31,32]

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28. Eigenmann, K., et al., *Angew. Chem.* (*Int. Ed.*), 14, 647, 1975.
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30. ITI 496.
31. Schiltz, A.A., *Perchloric Acid and Perchlorates*, G. Frederick Smith Chemical Co., Columbus, OH, 1979, p. 161.
32. PPL 171.

PERFORMIC ACID HCO_3H

IRRITANT

Synonyms

Peroxyformic acid, formyl hydroperoxide.

Physical Properties

90% Solution is a colorless liquid. On distillation, prone to explode on contact with metals and reducing agents.[1]

Fire Hazard

Explosive.[1]

Chemical Properties

Solutions are unstable, with gassing noticed after a few hours, and the effective concentration showing a definite decline in 2 hours.[1]

Hazardous Reactions

Solutions unstable even at room temperature; distillation is extremely dangerous.[2,3]

Metals or Metal Oxides. Explosions result on contact with Ni powder, Hg, colloidal Ag, thallium powder, Zn powder, PbO, Pb_3O_4, and Na_2O_2; violent decomposition occurs with barium peroxide, CuO, impure CrO_3, iridium dioxide, PbO_2, MnO_2, and V_2O_5 and with Fe powder contaminated with a trace of MnO_2.[3]

Nonmetals. Violent oxidation occurs with C, red phosphorus, and Si contaminated with a trace of MnO_2.[3]

Organic Materials. Violent reaction with formaldehyde, benzaldehyde, and aniline[3]; vigorous reaction with alkenes.[4]

Sodium Nitrate. Decomposes explosively in the presence of $NaNO_3$.[3]

Physiological Properties and Health Hazards

Irritating to the skin, eyes, and mucous membranes.[15]

Spillage Disposal

Wear a face shield, goggles, nitrile rubber gloves, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic scoop, shovel the mixture into a plastic container and transport to the fume hood. Slowly add to a pail of cold water. Avoid contact with metal. To the aqueous solution, add an ice-cold concentrated aqueous solution of sodium bisulfite until a drop of the mixture on starch-iodide paper does not produce a blue color. Test the pH of the solution and neutralize with 5% aqueous hydrochloric acid or sodium carbonate as appropriate. Decant the solution to the drain. Treat the solid residue as normal refuse.[6,7]

Waste Disposal

Wear eye protection, nitrile rubber gloves, and a laboratory coat. In the fume hood behind a shield, add to sufficient cold water to give a 5% solution of performic acid. Slowly add 20% aqueous

sodium bisulfite solution until a drop of the mixture on starch-iodide paper does not produce a blue color. Neutralize the solution with sodium carbonate and wash into the drain.[7]

Reactions for Spillage and Waste Disposal

$$HCO_3H + NaHSO_3 \rightarrow HCO_2H + NaHSO_4$$

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7. See "peracetic acid," ITI 495.

PERMETHRIN $C_{21}H_{20}Cl_2O_3$

INSECTICIDE

Synonyms

3-(2,2-Dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (3-phenoxyphenyl) methyl ester, 3-(phenoxyphenyl) methyl (±)-cis, *trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate, Ambush, Pounce.

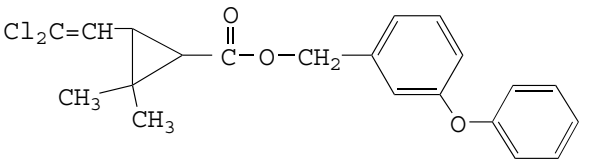
Physical Properties

Colorless crystals to a pale yellow viscous liquid, mp, 35°C, bp, 220°C (0.05 mm)[1]

Chemical Properties

Solubility in water below 1 ppm. Soluble or miscible with organic solvents, except ethylene glycol.[1] Easily hydrolyzed. Noncorrosive. Degraded by UV light.[2]

Structure



Physiological Properties and Health Hazards

Mild irritant to the skin and eyes.[1] LD_{50} (oral rat) 3800 mg/kg. LD_{50} (dermal rabbit) above 2000 mg/kg.[2] LC_{50} (rainbow trout, 48 hours) 5.4 µg/L.[3]

Waste Disposal

To each 500 mg of permethrin is added 100 mL of 10% sodium hydroxide solution and 4.7 g of potassium permanganate. The mixture is stirred at room temperature for 18 hours. Excess permanganate is reduced with sodium bisulfite and the solution is neutralized with dilute hydrochloric acid (1 volume of concentrated acid added to 5 volumes of water). The solution is poured into the drain and any residual brown manganese dioxide is discarded with regular garbage.[4]

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2. Agro. Handbook, A0316.
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4. Armour, M.A., *Laboratory Disposal Method for Permethrin*, University of Alberta, Edmonton, 2001.

PEROXIDES

EXPLOSIVE

Organic Peroxides

General guides to safe handling and use of peroxides[1,2] and peroxidizable compounds[3] have been published; data on fire and explosion hazards are available[4]; the hazards involved in the synthesis of organic peroxides have been reviewed.[5,6] See also Bretherick[7] and Steere.[8]

Alkyl Hydroperoxides

See entry for *tert*-Butyl Hydroperoxide.
Tendency to explode greater for shorter chain compounds.[9]

Diacyl Peroxides

See entry for Benzoyl Peroxide.
Lower members will detonate on the slightest disturbance.[10]

Dialkyl Peroxides

See entry for Di-*tert*-butyl Peroxide.
Lower members are highly explosive.[11,12]

Peroxyacids

See entries for *m*-Chloroperbenzoic Acid, Peracetic Acid, Perbenzoic Acid, and Performic Acid.
Very powerful oxidants, which should be used *in situ*; lower members are explosive while aromatic peroxyacids are more stable.[4,13]

Waste Disposal

See individual entries.

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PHENANTHRENE $C_{14}H_{10}$

MAY SENSITIZE SKIN TO LIGHT

Physical Properties

Monoclinic plates; mp, 100°C.[1]

Chemical Properties

Practically insoluble in water; soluble in organic solvents, especially in aromatic hydrocarbons.[1]

Physiological Properties and Health Hazards

Can cause photosensitization of skin.[1] Poison by intravenous route. Moderately toxic by ingestion.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Sweep onto paper and place in a separate labeled container for recycling or disposal by burning.[3]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[3]

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Destroy quantities of up to 1 g only. A 0.27 M solution of sodium dichromate is prepared by dissolving 20 g of $Na_2Cr_2O_7 \cdot 2H_2O$ in 10 mL of water and gradually adding enough concentrated sulfuric acid to bring the volume to 250 mL. Phenanthrene (1g) is gradually added at room temperature, while stirring, until it is dissolved. The solution is allowed to stand for 2 days, and then carefully poured into 1 L of water while stirring, treated with up to 84 g of sodium bisulfite to reduce Cr(VI) to Cr(III), and brought to above pH 7 with sodium hydroxide solution to precipitate the chromium as $Cr(OH)_3$, which is filtered and packaged for disposal. The aqueous filtrate is washed into the drain.[4]

REFERENCES

1. Merck 7289.
2. Sax 2154.
3. ITI 499.
4. PP 60.

PHENOL C_6H_5OH

TOXIC BY SKIN ABSORPTION, CAUSES BURNS

Synonyms

Carbolic acid, hydroxybenzene.

Physical Properties

Colorless crystalline substance with distinctive odor; reddens on exposure to air and light; mp, 43°C.[1]

Chemical Properties

One gram dissolves in about 15 mL water and 12 mL of benzene at 25°C; very soluble in alcohol, chloroform, ether, glycerol, carbon disulfide, petrolatum, volatile and fixed oils, and aqueous alkali hydroxides; almost insoluble in petroleum ether.[1]

Hazardous Reactions

Aluminum Trichloride and Nitrobenzene. Mixtures of aluminum trichloride, nitrobenzene, and phenol react violently at 120°C.[2]

Aluminum Trichloride, Nitromethane, and Carbon Monoxide. Formulation of phenol in the presence of nitromethane and aluminum chloride in a bomb at 100 bar and 100°C resulted in a high-energy explosion.[3]

Formaldehyde. Preparation of phenol-formaldehyde resins can result in uncontrollable reactions.[4]

Sodium Nitrate and Trifluoroacetic Acid. A rapid exothermic reaction occurred when phenol was added to a sodium nitrate–trifluoroacetic acid mixture.[5]

Sodium Nitrite. A heated mixture with phenol exploded violently.[6]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. Solid burns the skin. It is readily absorbed through the skin and causes headache, dizziness, weakness, and collapse. Swallowing causes severe internal damage, pain, nausea, and vomiting. Prolonged inhalation of the vapor results in disruption of the digestive and nervous systems, skin eruptions, and damage to the liver and kidneys. Avoid breathing vapor. Avoid contact with skin and eyes.[7] TLV-TWA 5 ppm (19 mg/m^3) (skin).[8]

Spillage Disposal

Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Eliminate all sources of ignition. Sweep onto paper. Follow waste disposal procedure.[9]

Spill site should be washed thoroughly with water and soap or detergent.[7]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning.[9]

Small Quantities. Wear butyl rubber[10] gloves, laboratory coat, and eye protection. Work in the fume hood. A solution of 4.7 g (0.05 mol) of phenol in 75 mL of water is prepared in a 200-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Ferrous sulfate heptahydrate (2.35 g, 0.0085 mol) is dissolved in the mixture and the pH is adjusted to

5–6 with dilute sulfuric acid. Thirty-percent hydrogen peroxide (41 mL, 0.4 mol) is added dropwise while stirring over 1 hour.

> ***CAUTION:*** The order of addition of the reagents is important. If hydrogen peroxide and ferrous sulfate are premixed, a violent reaction may occur.

Heat is evolved, and the reaction temperature is controlled at 50–60°C by adjusting the rate of addition of hydrogen peroxide or with an ice bath. Stirring is continued for 2 hours. The solution is allowed to stand overnight and then washed into the drain.[11]

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3. Webb, H.F., *Chem. Eng. News*, 55, 4, 1977.
4. B 149.
5. Spitzer, U.A. et al., *J. Org. Chem.*, 39, 3936, 1974.
6. B 1336.
7. Lux 529.
8. ACGIH 47.
9. Ald 2744.
10. LSS.
11. PP 63.

PHENYL AZIDE $C_6H_5N_3$

EXPLOSIVE

Synonyms

Triazobenzene, azidobenzene.

Physical Properties

Liquid; bp, 41–43°C/5mm. Phenyl azide explodes when heated at ordinary pressure and occasionally at a lower pressure. The distillation of phenyl azide must be carried out behind an explosion-proof screen.[1]

Fire Hazard

Explodes on heating.[2]

Chemical Properties

Insoluble in water.[3]

Hazardous Reactions

Explodes when heated at atmospheric pressure.[2]

Lewis Acids. Vigorous or violent decomposition with H_2SO_4 and Lewis acids.[4]

Spillage Disposal

Wear leather gloves, a heavy face shield, goggles, and a laboratory coat. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. With a plastic scoop, shovel the mixture into a plastic container and transport to the fume hood. Very slowly, add the mixture to a large beaker containing concentrated hydrochloric acid (100 mL/g azide) and granular tin (6 g/g azide). Stir for 30 minutes, and then cautiously add the solution to a pail of cold water. The solid may be treated as normal refuse. Neutralize the aqueous solution with sodium carbonate and wash into the drain.[5]

Waste Disposal

Wear leather gloves, heavy face shield, goggles, and laboratory coat. Work in the fume hood behind a body shield. Slowly add the azide (1 g) to a stirred mixture of granular tin (6 g) in concentrated hydrochloric acid (100 mL). Continue stirring for 30 minutes. Cautiously add the solution to a pail of cold water. Any residual tin can be washed with water and re-used. The aqueous solution is neutralized with sodium carbonate and washed into the drain.[5]

Reactions for Spillage and Waste Disposal

$$C_6H_5N_3 + Sn + 2HCl \rightarrow \underset{\text{aniline}}{C_6H_5NH_2} + N_2 + SnCl_2$$

REFERENCES

1. F & F 1, 829.
2. Lindsay, R.O. et al., *Org. Synth.*, Coll. Vol. 3, 710, 1955.

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PHENYLHYDRAZINE $C_6H_5NHNH_2$

PHENYLHYDRAZINE HYDROCHLORIDE $C_6H_5NHNH_2 \cdot HCl$

TOXIC, HARMFUL IF TAKEN INTERNALLY OR IF IN CONTACT WITH SKIN

Physical Properties

Monoclinic prisms or oil; turns yellow to dark red on exposure to air and light; faint aromatic odor; mp, 19.5°C (dihydrate 24°C). Hydrochloride consists of colorless to brown leaflets; mp, 243–246°C (slight browning).[1,2]

Chemical Properties

Phenylhydrazine is sparingly soluble in water. Phenylhydrazine hydrochloride is soluble in water. Keep in tightly closed containers protected from light.[1]

Hazardous Reactions

Lead(IV) Oxide. Ignites on contact with the oxide.[3]

Physiological Properties and Health Hazards

Absorption resulting from inhalation, skin contact, or ingestion may result in blood and liver damage, giving rise to nausea, vomiting, and jaundice. Causes hemolysis of erythrocytes. Dermatitis may follow skin contact. Must be considered injurious to the eyes. Avoid inhalation of vapor or dust. Prevent contact with skin and eyes.[2] TLV-TWA 0.1 ppm (0.44 mg/m^3) (skin).[4]

Spillage Disposal

Wear eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container, transport to the fume hood, and slowly add to water allowing 20 mL water for each 1 g of phenylhydrazine or phenylhydrazine hydrochloride (to the aqueous phenylhydrazine · HCl, add 3 mL of 10% NaOH). Filter off the clay and sand. For each 1 g of phenylhydrazine (or phenylhydrazine · HCl), place 18 mL (about 25% excess) of commercial laundry bleach (containing about 5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous phenylhydrazine dropwise to the stirred hypochlorite solution, monitoring the rate of addition by rise in temperature. The temperature is maintained at 45–50°C, and addition takes about 1 hour. Stirring is continued for 2 hours until the temperature gradually falls to room temperature. The cooled reaction mixture can be washed into the drain.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[7]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. Prepare a dilute aqueous (5%) solution of phenylhydrazine (phenylhydrazine · HCl; add 3 mL of 10% NaOH for each 1 g of the hydrochloride) by adding slowly to the appropriate volume of water. For each 1 g of phenylhydrazine or phenylhydrazine · HCl, place 18 mL (about 25% excess) of commercial laundry bleach (containing about 5.25% sodium hypochlorite) into a three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel. Add the aqueous phenylhydrazine to the stirred hypochlorite solution, monitoring the rate of

addition by rise in temperature. The temperature is maintained at 45–50°C, and addition takes about 1 hour. Stirring is continued for 2 hours until the temperature gradually falls to room temperature. The cooled reaction mixture can be flushed down the drain with at least 50 times its volume of water.[6]

Reactions for Spillage and Waste Disposal

$$C_6H_5NHNH_2 + NaOCl \rightarrow C_6H_6 + N_2 + NaCl + H_2O$$

REFERENCES

1. Merck 7377.
2. Lux 532.
3. Mellor, Vol. 7, 637, 1941; Vol. 8, 291, 1940.
4. ACGIH 48.
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6. PP 94.
7. Ald 2780C.

N-PHENYL-β-NAPHTHYLAMINE $C_{10}H_7NHC_6H_5$

ANIMAL CARCINOGEN

Physical Properties

Rhombic crystals from methanol; mp, 108°C.[1]

Chemical Properties

Insoluble in water; soluble in hot benzene; very soluble in ether and hot alcohol.[1]

Hazardous Reactions

Emits highly toxic fumes when heated to decomposition; can react with oxidizing materials.[1]

Physiological Properties and Health Hazards

An experimental carcinogen via oral and subcutaneous routes.[1]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection.

On skin and clothing. Wash skin with strong soap solution immediately. Rinse thoroughly. Contaminated clothing should be removed, dried, and washed with strong soap solution, or destroyed. It may be necessary to destroy shoes by burning.

Spills. Scoop into appropriately labeled container for recycling or disposal by burning (see waste disposal procedure). Wash site with strong soap solution.[2,3]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning.[2,3]

REFERENCES

1. Sax 2767.
2. Ald 2786C.
3. PP 70.

PHOSGENE AND SOLUTIONS $COCl_2$

TOXIC BY INHALATION

Synonyms

Carbonyl chloride.

Physical Properties

Colorless gas with odor reminiscent of moldy hay; bp, 8.2°C.[1]

Chemical Properties

Slightly soluble in water and slowly hydrolyzed by it; freely soluble in benzene, toluene, glacial acetic acid, and most liquid hydrocarbons.[1]

Hazardous Reactions

Potassium. Mixture with K is shock sensitive.[2]

Safe Handling Procedure[1]

> ***WARNING:*** Paper soaked in alcoholic or carbon tetrachloride solution containing 10% of a mixture of equal parts of p-dimethylaminobenzaldehyde and colorless diphenylamine, and then dried, will turn from yellow to deep orange in the presence of approximately the maximum allowable concentration of phosgene, and should always be used where the generation of this gas is possible or suspected. See also Braker and Mossman.[3]

Physiological Properties and Health Hazards

Insidious poison, as it is not irritating immediately, even when fatal concentrations are inhaled. May cause severe pulmonary edema (may be quickly fatal) or pneumonia. Inhalation of high concentrations causes choking, constricted feeling in chest, coughing, painful breathing, and bloody sputum. Vapors are strongly irritating to the eyes.

Do not breathe gas. Avoid contact of solutions with skin and eyes.[1] TLV-TWA 0.1 ppm (0.40 mg/m^3).[4]

Waste Disposal

Seal cylinder and return to supplier.

Phosgene can be vented into an ammonia scrubber[5] or decomposed by bubbling into 20% NaOH.[6]

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1. Merck 7421.
2. Mellor, Vol. 10, 642, 646, 908, 912, 1947.
3. Braker, W. and Mossman, A.L., *Matheson Gas Data Book*, 5th ed., Matheson Gas Products, East Rutherford, NJ, 1971.
4. ACGIH 48.
5. Wagner, E.C. et al., *Org. Synth.*, Coll. Vol. 3, 488, 1955.
6. Shriner, R.L. et al., *Org. Synth.*, Coll. Vol. 2, 454, 1943.

PHOSPHINE PH_3

EXTREMELY FLAMMABLE, TOXIC BY INHALATION

Synonyms

Hydrogen phosphide.

Physical Properties

Colorless gas with smell somewhat like that of rotting fish; bp, –87.7°C.[1]

Fire Hazard

Impure phosphine must be regarded as spontaneously flammable. Because the gas is supplied in a cylinder, turning off the valve will reduce any fire involving it; if possible, cylinders should be removed quickly from an area in which a fire has developed.[2]

Chemical Properties

Slightly soluble in water. With a trace of P_2H_4 present, phosphine is spontaneously flammable in air, burning with a luminous flame.[1,2]

Hazardous Reactions

Liquified phosphine is detonatable.[3]

Air. Dry phosphine ignites in air at room temperature. Small traces of impurities such as diphosphane, dinitrogen trioxide, nitrous acid, or other oxidant cause phosphine to ignite at temperatures below –15°C.[3,4]

Dichlorine Oxide. Explosion occurs on contact with phosphine.[5]

Halogens. Phosphine ignites on contact with chlorine, bromine, or their aqueous solutions.[4]

Metal Nitrates. Ignition of the gas or explosion occurs when phosphine is bubbled into concentrated silver nitrate solution. With mercury(II) nitrate, a complex phosphide, which is explosive when dry, is formed .[6]

Nitric Acid. Phosphine is oxidized explosively by fuming nitric acid and ignites when fuming acid is dripped into the gas.[7]

Oxygen. Phosphine–oxygen mixtures are explosive.[8]

Physiological Properties and Health Hazards

Breathing gas results in chest pain, weakness, damage to the lungs, coma, and finally death. Prevent inhalation of gas.[2] TLV-TWA 0.3 ppm (0.42 mg/m^3); TLV-STEL 1 ppm (1.4 mg/m^3).[9]

Waste Disposal

Phosphine can be oxidized to phosphoric acid by slowly venting gas into excess 1 M copper sulfate solution (prepared by dissolving 127.7 g of $CuSO_4$ or 199.7 g of $CuSO_4 \cdot 5H_2O$ in 800 mL of water). Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. To avoid violent reaction with air, the oxidation must be carried out under nitrogen, most conveniently in a three-necked, round-bottom flask equipped with a stirrer, nitrogen inlet, and gas inlet. Allow the reaction mixture to stand for several days. The precipitate is separated and while it is still wet, transferred to a beaker containing excess laundry bleach (about 5% sodium hypochlorite) and stirred for an hour to ensure oxidation of any copper phosphide to phosphate. The copper salt solution can be flushed into the drain.[10]

Reactions for Spillage and Waste Disposal

$$PH_3 + 4CuSO_4 + 4H_2O \rightarrow H_3PO_4 + 4Cu + 4H_2SO_4$$

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10. PP 92.

PHOSPHORIC ACID H_3PO_4

CORROSIVE

Synonyms

Orthophosphoric acid.

Physical Properties

Colorless viscous liquid (88–93%) or moist white crystals (100%); mp, 42.4°C.[1,2]

Chemical Properties

Soluble in water and alcohol.[2]

Hazardous Reactions

Hot concentrated acid attacks porcelain and granite ware.[2]

Chlorides. Contact of 75% phosphoric acid containing traces of chlorides within a closed storage tank may liberate hydrogen, which will explode on ignition.[3,4]

Nitromethane. Mixtures of nitromethane and phosphoric acid can be detonated.[5]

Sodium Borohydride. Generation of diborane by addition of sodium borohydride to anhydrous phosphoric acid may be dangerously violent.[6]

Physiological Properties and Health Hazards

Vapor irritates and liquid severely burns the eyes, skin, and mucous membranes. Ingestion causes internal damage, nausea, vomiting, intoxication, and shock.[1,2,7] TLV-TWA 1 mg/m^3; TLV-STEL 3 mg/m^3.[8]

Spillage Disposal

Wear a face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Slowly add to a pail of ice-cold water. Test the pH and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solids as normal refuse. Wash the site of the spill thoroughly with water.[1,9,10]

Waste Disposal

Wear a face shield, goggles, laboratory coat, and nitrile rubber gloves. In the fume hood, slowly add the acid to a pail of cold water. Cautiously neutralize the solution by adding solid sodium carbonate while stirring. Wash the solution into the drain.[9,10]

Reactions for Spillage and Waste Disposal

$$2H_3PO_4 + 3Na_2CO_3 \rightarrow 2Na_3PO_4 + 3H_2O + 3CO_2$$

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PHOSPHORUS, Red and White (Yellow) P

RED: HIGHLY FLAMMABLE, EXPLOSIVE WHEN MIXED WITH OXIDIZING SUBSTANCES

WHITE (YELLOW): SPONTANEOUSLY FLAMMABLE IN AIR, SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING

Physical Properties

Red (amorphous) phosphorus sublimes at 416°C; white (yellow) phosphorus is a pale yellow, waxy, translucent solid; mp, 44°C; usually stored under water. Red yields the white form when distilled at 290°C.[1]

Fire Hazard

White phosphorus ignites at about 30°C in moist air.[1] Water is the best medium for fighting a phosphorus fire caused by its spontaneous ignition; then smother with wet sand.[2]

Chemical Properties

Red phosphorus is insoluble in water. White phosphorus ignites rapidly in air, giving off fumes of the oxide.[3]

Hazardous Reactions

Red phosphorus may ignite on impact.[4]

Alkali Metal Nitrides. Highly inflammable mixture formed on heating with phosphorous.[5]

Alkalies. Contact of phosphorus with boiling caustic alkalies or with hot calcium hydroxide produces phosphine, which can ignite in air.[6]

Chlorosulfuric Acid. Reaction with both white and red phosphorus accelerates to explosion.[7]

Halogens and Interhalogens. Yellow and red phosphorus ignite on contact with fluorine or chlorine, and red ignites in liquid bromine. Yellow phosphorus explodes in liquid chlorine or bromine and ignites in bromine vapor or solid iodine.[8–11]

Hydriodic Acid. The use of red phosphorus and hydriodic acid as a reducing agent for organic carbonyl compounds is potentially hazardous.[12,13]

Hydrogen Peroxide. If white or red phosphorus is not completely immersed while undergoing oxidation in H_2O_2 solutions (30% or stronger), it may ignite and cause a violent reaction.[14]

Magnesium Perchlorate. Mixtures may explode violently.[15]

Metal Halogenates. Dry, finely divided mixtures of red or white phosphorus and chlorates, bromates, or iodates of barium, calcium, magnesium, potassium, sodium, or zinc explode when subjected to friction, impact, or heat. Mixtures of potassium iodate with white or red phosphorus react violently or explosively on addition of a small quantity of water.[16]

Metal Halides. Phosphorus ignites in contact with antimony pentafluoride and explodes with chromyl chloride when moist.[17]

Metal Oxides. Red phosphorus reacts vigorously on heating with copper oxide or manganese dioxide. It may ignite on grinding or warming with lead monoxide or mercury or silver oxides. It ignites in contact with lead, sodium, or potassium peroxides. White phosphorus explodes in contact with the latter peroxides.[18]

Metals. Beryllium, manganese, thorium, copper, and zirconium react with incandescence when heated with phosphorus.[19] Cerium, lanthanum, neodymium, and praseodymium react violently above 400°C. Osmium incandesces in phosphorus vapor and platinum burns vividly.[20]

Nitrates. Yellow phosphorus ignites in molten ammonium nitrate and mixtures with ammonium, mercury(I), or silver nitrates explode on impact.[21]

Nitric Acid. The vapor from phosphorus heated in HNO_3 may ignite.[22]

Nonmetal Halides. White and red phosphorus incandesce with boron triiodide. White phosphorus explodes with seleninyl chloride. Red phosphorus incandesces in seleninyl chloride and reacts vigorously on warming with sulfuryl or disulfuryl chlorides and violently with disulfur dibromide.[23]

Nonmetal Oxides. Warm or molten white phosphorus burns vigorously in nitrogen oxide, dinitrogen tetroxide or pentoxide, and ignites in sulfur trioxide vapor or liquid.[24]

Oxygen. Finely divided white phosphorus ignites in air. Contact with charcoal or amalgamated aluminum promotes ignition.[25]

Performic Acid. Red phosphorus is oxidized violently.[26]

Potassium Permanganate. Mixtures of phosphorus and potassium permanganate explode on grinding.[27]

Selenium. Mixture incandesces on warming.[28]

Sulfur. White phosphorus ignites on contact with sulfur and explodes on heating.[24] Red phosphorus may ignite on mixing and cause a violent reaction.[29]

Sulfuric Acid. White phosphorus ignites in contact with boiling H_2SO_4 or its vapor.[24]

Physiological Properties and Health Hazards

Red phosphorus is not considered to be harmful physiologically. White phosphorus burns the skin and eyes and causes severe internal damage if swallowed. The vapor from burning phosphorus irritates the respiratory system and eyes. Prolonged absorption of small quantities results in anemia, intestinal weakness, and bone and liver damage. Avoid contact with skin and eyes.[3] TLV-TWA (yellow phosphorus) 0.1 mg/m^3.[30]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves.

Red Form. Moisten with water, shovel into a bucket, and transport to a safe isolated area where the moisture can be allowed to dry off and the phosphorus burned off. This applies to small spills only.[3]

Yellow or White Form. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Moisten with water. Scoop into a container and cover with water. Transfer the container to the fume hood. Add 1 M cupric sulfate solution (127.7 g or 199.7 g of $CuSO_4$ or $CuSO_4 \cdot 5H_2O$, respectively, dissolved in 800 mL of water; about 160 mL for each 1 g of phosphorus). Allow it to stand for a week. Decant the liquid to the drain with at least 50 volumes of water. Add laundry bleach to the solid (about 100 mL for each 1 g of phosphorus). Stir for 1 hour. The liquid can be washed into the drain and the solid treated as normal refuse.[31,33]

Waste Disposal

Package Lots. Put the phosphorus under water in a large container. Repackage in the water, label, and return to suppliers.[32]

Small Quantities (White Phosphorus). Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Five g (0.16 mol) of white phosphorus are cut under water into 5-mm pellets. The pellets are added to 800 mL (0.8 mol) of 1 M cupric sulfate (127.7 g or 199.7 g of $CuSO_4$ or $CuSO_4 \cdot 5H_2O$, respectively, dissolved in 800 mL of water) solution in a 2-L beaker. The mixture is allowed to stand for about a week with occasional stirring. The phosphorus grad-

ually disappears, and a fine black precipitate of copper and copper phosphide is formed. The reaction is complete when no waxy white phosphorus is observed when one of the pellets is cut under water. The precipitate is separated, and while still wet, transferred to 500 mL of laundry bleach (5% sodium hypochlorite), and then stirred for about 1 hour to ensure complete decomposition of copper phosphide. The solids are separated and packaged for disposal. The aqueous solution is washed into the drain.[33]

Small Quantities (Red Phosphorus). Add the red phosphorus (5 g) to a solution of potassium chlorate (33 g) in 2 L of 1 N sulfuric acid (prepared by cautiously adding 56 mL of concentrated acid to 1944 mL of cold water) in a 3-L, three-necked, round-bottom flask equipped with water-cooled condenser and heating mantle. Heat the mixture under reflux until all of the phosphorus has dissolved (5–10 hours). When the solution has cooled to room temperature, reduce the excess chlorate by adding about 14 g of sodium bisulfite. Wash the solution into the drain.[33]

Reactions for Spillage and Waste Disposal

$$\underset{\text{white phosphorus}}{2P} + 5CuSO_4 + 8H_2O \rightarrow 5Cu + 2H_3PO_4 + 5H_2SO_4$$

$$\underset{\text{red phosphorus}}{6P} + 5KClO_3 + 9H_2O \rightarrow 6H_3PO_4 + 5KCl$$

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PHOSPHORUS PENTACHLORIDE PCl_5

IRRITANT VAPOR AND DUST, CAUSES BURNS

Physical Properties

White to pale yellow, fuming, deliquescent, crystalline masses with pungent, unpleasant odor; mp, 148°C under pressure. Sublimes at about 100°C without melting; bp, 160°C.[1]

Chemical Properties

Violently decomposed by water with formation of hydrochloric acid and phosphoric acid.[12] Reacts with alcohols to form corresponding chlorides; soluble in carbon disulfide and tetrachloride.[1]

Hazardous Reactions

Aluminum. Ignites on contact with Al powder.[3]

Carbamates. Unstable salts formed between carbamates and PCl_5, which may decompose explosively.[4]

Chlorine Dioxide and Chlorine. Explodes on contact with mixture of chlorine dioxide and chlorine.[5]

Diphosphorus Trioxide. Violent interaction at room temperature.[6]

Fluorine. Ignites on contact.[7]

Hydroxylamine. Ignites on contact.[8]

Nitrobenzene. Solution decomposes violently above 120°C.[9]

Urea. Dry mixture explodes after heating.[10]

Water. Reacts violently with water.[11]

Physiological Properties and Health Hazards

Vapor and dust severely irritate the mucous membranes and respiratory system. Vapor severely irritates and the solid burns the eyes. The vapor and solid burn the skin. Swallowing would cause internal irritation and damage. Prolonged exposure to low concentrations of vapor may cause damage to lungs. Avoid breathing vapor and dust. Prevent contact with eyes and skin.[2] TLV-TWA 0.1 ppm (0.85 mg/m^3).[12]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, face shield, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[13]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, place the phosphorus pentachloride in a large evaporating dish and cover with excess solid sodium carbonate or calcium carbonate. Very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.[13]

Reactions for Spillage and Waste Disposal

$$PCl_5 + 4H_2O \xrightarrow{NaOH} H_3PO_4 + 5HCl \rightarrow \underset{\text{sodium phosphate}}{Na_3PO_4} + 5NaCl$$

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PHOSPHORUS PENTOXIDE P_2O_5

CAUSES SEVERE BURNS, CORROSIVE

Synonyms

Phosphorus(V) oxide, phosphoric oxide.

Physical Properties

White crystalline deliquescent powder; mp, 340°C. Sublimation temperature, 360°C.[1]

Chemical Properties

Reacts violently with water.[1]

Hazardous Reactions

Formic Acid. Attempted dehydration of 95% acid caused rapid evolution of CO.[2]

Hydrogen Fluoride. Vigorous interaction below 20°C.[3]

Inorganic Bases. Mixtures with Na_2O or CaO react violently if warmed or moistened.[4,5]

Iodides. Interaction is violent.[5]

Metals. Reaction with warm Na or K is incandescent; reaction with Ca is explosive when heated.[6]

Water. Highly exothermic reaction.[7]

Physiological Properties and Health Hazards

Dust irritates the respiratory system and burns the eyes and skin. Swallowing would cause internal irritation and damage. Prevent contact with eyes and skin.[8]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, face shield, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, place the phosphorus pentoxide in a large evaporating dish and cover with excess solid sodium carbonate or calcium carbonate. Very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize with sodium carbonate or 5% sodium hydroxide solution if necessary. Wash the solution into the drain.[9]

Reactions for Spillage and Waste Disposal

$$P_2O_5 + 3H_2O \; 2H_3PO_4 \xrightarrow{NaOH} 2Na_3PO_4$$

sodium phosphate

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PHOSPHORUS TRIBROMIDE PBr_3

HARMFUL VAPOR, CAUSES BURNS

Physical Properties

Colorless fuming liquid; bp, 173°C.[1]

Chemical Properties

Reacts violently with water, forming hydrobromic acid and phosphorous acid.[2]

Hazardous Reactions

Calcium Hydroxide and Sodium Carbonate. Disposal by slow addition to sodium carbonate and dry slaked lime resulted in a violent reaction.[3]

Potassium. PBr_3 liquid or vapor ignites on contact with K.[4]

Sodium. Na floats on liquid PBr_3, but addition of a little water causes a violent explosion[5]; mixture of Na with PBr_3 is shock sensitive.[6]

Water. Reacts rapidly with warm water, and reacts violently with limited quantities.[7]

Physiological Properties and Health Hazards

Vapor severely irritates the respiratory system. The vapor irritates and the liquid burns the eyes and skin. Swallowing assumed to cause severe internal irritation and damage. Prolonged exposure to low concentrations of vapor may cause damage to lungs. Avoid breathing vapor. Prevent contact with eyes and skin.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the phosphorus tribromide has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, place the phosphorus tribromide in a large evaporating dish. Cover with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize with sodium carbonate or dilute sodium hydroxide solution if necessary. Wash the solution into the drain.[8]

Reactions for Spillage and Waste Disposal

$$PBr_3 + 3H_2O \rightarrow H_3PO_3 + 3HBr$$

$$H_3PO_3 + HBr + 2Na_2CO_3 \rightarrow Na_3PO_3 + NaBr + 2H_2O + 2CO_2$$

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PHOSPHORUS TRICHLORIDE PCl_3

HARMFUL VAPOR, CAUSES BURNS

Physical Properties

Colorless fuming liquid; bp, 76°C.[1]

Chemical Properties

Violently decomposed by water, forming hydrochloric acid and phosphorous acid.[2]

Hazardous Reactions

Acetic Acid. Residue in preparation of acetyl chloride from PCl_3 and acetic acid may decompose violently with evolution of flammable phosphine.[3,4]

Carboxylic Acids. Use of PCl_3 as chlorinating agent for carboxylic acids may result in explosion when product is distilled.[5–7]

Dimethyl Sulfoxide. Reacts explosively with PCl_3.[8]

Hydroxylamine. Ignites on contact with PCl_3.[9]

Metals. K ignites in PCl_3; molten Na explodes on contact.[10]

Nitric or Nitrous Acids. Explodes on contact with either acid.[11]

Water. Reacts violently on contact with water with liberation of some diphosphane that ignites.[3]

Physiological Properties and Health Hazards

Vapor severely irritates the respiratory system. The vapor and liquid burn the eyes and skin. Swallowing causes severe internal irritation and damage. Prolonged exposure to low concentrations of vapor may cause lung damage. Avoid breathing vapor. Prevent contact with eyes and skin.[2] TLV-TWA 0.2 ppm (1 mg/m^3); TLV-STEL 0.5 ppm (2.8 mg/m^3).[12]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the phosphorus trichloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[13]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the phosphorus trichloride into a large evaporating dish. Cover with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize with sodium carbonate or 5% sodium hydroxide solution if necessary. Pour the solution into the drain, flushing with at least 50 times its volume of water.[13]

Reactions for Spillage and Waste Disposal

$$PCl_3 + 3H_2O \rightarrow H_3PO_3 + 3HCl$$

$$H_3PO_3 + HCl + 2Na_2CO_3 \rightarrow Na_3PO_3 + NaCl + 2H_2O + 2CO_2$$

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PHOSPHORYL CHLORIDE $POCl_3$

HARMFUL VAPOR, CAUSES BURNS

Synonyms

Phosphorus oxychloride.

Physical Properties

Colorless fuming liquid; bp, 106°C; mp, 1°C.[1]

Chemical Properties

Violently decomposed by water, forming hydrochloric acid and phosphoric acid.[2]

Hazardous Reactions

Carbon Disulfide. Solution in benzene reacts immediately with carbon disulfide with evolution of hydrochloric acid.[3]

Dimethyl Sulfoxide. May react explosively.[4]

Water. There is considerable delay in its reaction with water, which ultimately becomes violent. Vessels containing the solution must not be closed or only slightly vented.[5]

Zinc Dust. Ignites on contact with $POCl_3$.[6]

Physiological Properties and Health Hazards

Vapor severely irritates the respiratory system; there may be sudden or delayed pulmonary edema. Vapor and liquid burn the eyes and skin. Swallowing causes severe internal irritation and damage. Avoid breathing vapor. Prevent contact with eyes and skin.[2] TLV 0.1 ppm (0.63 mg/m^3).[7]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the phosphoryl chloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution into the drain, flushing with 50 times its volume of water. Treat the solid residue as normal refuse.[8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the phosphoryl chloride into a large evaporating dish. Cover with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow it to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Pour the solution to the drain flushing with at least 50 times its volume of water.[8]

Reactions for Spillage and Waste Disposal

$$POCl_3 + 3H_2O \xrightarrow{Na_2CO_3} H_3PO_4 + 3HCl \rightarrow \underset{\text{sodium phosphate}}{Na_3PO_4} + 3NaCl + H_2O + CO_2$$

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PICLORAM $C_6H_3Cl_3N_2O_2$

HERBICIDE

Synonyms

4-Amino-3,5,6-trichloro-2-pyridinecarboxylic acid; 4-amino-3,5,6-trichloropicolinic acid, Tordon.

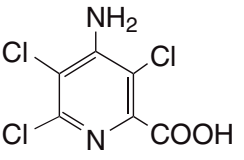
Physical Properties

Colorless powder with chlorine-like odor; decomposes before melting at 215°C.[1–3]

Chemical Properties

Solubility at 25°C is 430 mg/L water, 19.8 g/L acetone, and 5.5 g/L propan-2-ol.[1]

Structure



Physiological Properties and Health Hazards

Acute oral LD_{50} 8200 mg/kg (rats), 2000–4000 mg/kg (mice), above 1000 mg/kg (sheep), above 750 mg/kg (cattle).[2] TLV-TWA 10 mg/m^3.[4]

Spillage Disposal

Wear gloves and eye protection. The powder can be swept up and discarded as for waste disposal. For spills of solutions of Picloram, cover with a 1:1:1 mixture of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and add to a pail of water. Acidify the liquid by the careful addition of 3 M sulfuric acid (8 mL of concentrated sulfuric acid added to 42 mL of water) to pH 1. Estimate the weight of Picloram in the spill and add 5 g of potassium permanganate for each 0.3 g of Picloram. Stir thoroughly and allow it to stand at room temperature overnight. Reduce the excess permanganate by the addition of sodium bisulfite until no purple color remains. Neutralize the solution with sodium carbonate (foaming may occur) or dilute (5%) sodium hydroxide solution. Decant the liquid into the drain with water. The solid residue can be treated as normal refuse.[5]

Waste Disposal

Wear gloves and eye protection. To each 0.3 g of Picloram, add 50 mL of 3 M sulfuric acid (8 mL of concentrated sulfuric acid added to 42 mL of water). Stir solid potassium permanganate (5 g) into the solution and allow the mixture to stand at room temperature for 24 hours. The excess permanganate is reduced by the addition of sodium bisulfite and the solution neutralized with 10% aqueous sodium hydroxide. The clear solution is poured into the drain and any residual brown manganese dioxide discarded with regular refuse.[5]

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PICRIC ACID $(NO_2)_3C_6H_2OH$

RISK OF EXPLOSION BY SHOCK, FRICTION, FIRE, OR OTHER SOURCE OF IGNITION; FORMS VERY SENSITIVE EXPLOSIVE METALLIC COMPOUNDS

NOTE: Picric acid should be kept moist with not less than half its own weight of water. In the laboratory, it is commonly used in alcoholic solutions.

Synonyms

2,4,6,-Trinitrophenol.

Physical Properties

Pale yellow, odorless, intensely bitter crystals; mp, 122–123°C. Explodes above 300°C.[1]

Fire Hazard

Explosive; flash point, 150°C; ignition temperature, less than 300°C; oxidizing agent. Extinguish fire with water; do not use carbon dioxide.[2]

Chemical Properties

One gram dissolves in 78 mL of water, 15 mL of boiling water, 12 mL of alcohol, 10 mL of benzene, 35 mL of chloroform, and 65 mL of ether.[1]

Hazardous Reactions

Explosive solid, which is usually stored as water-wet paste.[3]

Metals and Salts. Forms salts with many metals, some of which (Pb, Hg, Cu, or Zn) are sensitive to heat, friction, or impact; contact of acid with concrete floors may form friction-sensitive Ca salt.[3]

Physiological Properties and Health Hazards

Skin contact may result in dermatitis. Swallowing causes poisoning. Prolonged absorption through the skin or inhalation of dust may result in skin eruptions, headache, nausea, vomiting, or diarrhea; the skin may become yellow. Avoid contact with skin and eyes.[4] TLV-TWA 0.1 mg/m^3.[5]

Spillage Disposal

Wear face shield, goggles, nitrile rubber gloves, and laboratory coat. Moisten the spilled picric acid thoroughly with water. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic scoop, shovel into a labeled container for disposal by burning in an open incinerator or in a furnace with afterburner and scrubber.[6,7] Wash the spill site with large quantities of water and detergent.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve in a combustible solvent and spray into furnace with afterburner and scrubber.[6]

Small Quantities. This method has been used to decompose batches of up to 8.5 g of picric acid at a time.[8] Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood behind a shatter-proof screen. Place the picric acid (1 g) in a three-necked, round-bottomed flask fitted with a dropping funnel and condenser. Rinse any traces of acid on glassware or equipment into the flask using about 10 mL of water. Add 4 g of tin and stir the mixture magnetically. Place

15 mL of concentrated hydrochloric acid in the dropping funnel and, while cooling the flask in an ice-water bath and while stirring, add the hydrochloric acid dropwise. The first few milliliters of hydrochloric acid should be added slowly because the initial reaction is vigorous; the rate of addition may be increased as the reaction moderates. When all of the hydrochloric acid has been added, allow the mixture to warm to room temperature, and then heat under reflux for 1 hour to complete the reduction. Filter unreacted tin and wash the precipitate with about 10 mL of 2 M hydrochloric acid (prepared by cautiously adding 1.6 mL of concentrated acid to 8.4 mL of cold water; the tin can be recycled). Neutralize the filtrate with 10% sodium hydroxide solution and refilter. The tin chloride precipitate can be treated as normal refuse. The solution that contains 2,4,6-triaminophenol and which oxidizes rapidly to a black solution can be packaged, labeled, and sent for incineration, or it can be treated as follows. Slowly and cautiously add to the solution 150 mL of 3 M sulfuric acid containing 12 g of potassium permanganate. Stir the mixture at room temperature for 24 hours and then add solid sodium bisulfite until a clear solution is obtained. Neutralize with 10% sodium hydroxide solution and wash the liquid into the drain.

CAUTION: Under no circumstances should dry picric acid be touched or moved. Do not handle. Contact disposal authorities. The only safe method of decomposing dry picric acid is by controlled detonation.

Dilute Aqueous Solutions. Acidify a dilute solution of picric acid (100 mL of 0.4%) to pH 2 by the addition of 2 mL of concentrated hydrochloric acid. Add granular tin (30 mesh, 1 g) and allow the mixture to stand at room temperature. The solution will darken gradually as the picric acid is reduced. After 14 days, no picric acid remains. This method can be used to dispose of 45-gallon drums of dilute solutions of picric acid. To determine when the picric acid is completely reacted, samples of the solution are analyzed by thin-layer chromatography on silica gel, eluting with methanol:toluene:glacial acetic acid, 8:45:4. Picric acid has an Rf value of about 0.3, and the bright yellow spot is easily visible. The detection limit can be increased by developing the plate in iodine vapor.[8]

Reactions for Spillage and Waste Disposal

$$(NO_2)_3C_6H_2OH + 9Sn + 18HCl \rightarrow \underset{\text{2,4,6-triaminophenol}}{(NH_2)_3C_6H_2OH} + 6H_2O + 9SnCl_2$$

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PIRIMICARB $C_{11}H_{18}N_4O_2$

INSECTICIDE, APHICIDE

Synonyms

Dimethylcarbamic acid-2-(dimethylamino)-5,6-dimethyl-4-pyrimidinyl ester, 2-(dimethylamino)-5,6-dimethyl-4-pyrimidinyl dimethyl carbamate, Pirimor.

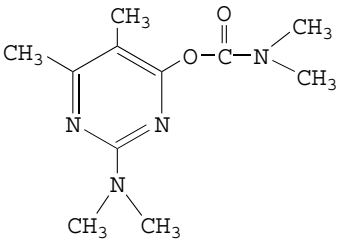
Physical Properties

Colorless, odorless, crystalline solid; mp, 90.5°C.[1]

Chemical Properties

Solubile in water at 25°C 2.7 g/L.[1] Very soluble in most organic solvents. Decomposed by UV light.[2] Decomposed by prolonged boiling in acids or bases. Forms crystalline salts with organic and inorganic acids.[2]

Structure



Physiological Properties and Health Hazards

LD_{50} (oral rat) 147 mg/kg.[1] Low toxicity to fish. Avoid breathing dust or spray mist. Can cause blurred vision and/or breathing difficulties.[2]

Formulations

Wettable powder, granules, and emulsifiable concentrate.[2]

Waste Disposal

Wear gloves and eye protection. For each 1 g of commercial Pirimicarb, add 250 mL of household chlorine bleach. Stir, and allow it to stand at room temperature for 24 hours. Wash into the drain with water.[3]

Laundering of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash. Launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer by running machine

through a complete wash cycle with detergent (but without clothing) before washing other clothes.

Wettable powder formulation of Pirimicarb was diluted to field strength (0.025% AI) and used to contaminate 100% cotton and 65/35 polyester/cotton fabric. A single warm-water wash (50°C) leaves approximately 1% Pirimicarb residues on the fabric.[4]

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PLATINUM (Metal and Hydrogenation Catalyst) Pt

CATALYST FORMS ARE EXPLOSIVE WHEN DRY

Physical Properties

Silver-gray, lustrous, malleable, and ductile metal; mp, 1774°C.[1]

Chemical Properties

Pt catalysts may explode if handled when dry.[2,3] Powder is flammable.[4]

Hazardous Reactions

Arsenic. Mixture in sealed tube at 270°C exploded.[5]

Ethanol. Addition of Pt-black, catalyst to ethanol caused ignition.[2]

Hydrazine. Violently decomposed by Pt-black and H_2 evolved may ignite.[6]

Hydrogen and Air. Explosion occurred when platinized alumina catalyst was purged with air instead of with inert gas after being treated with H_2.[2]

Hydrogen Peroxide. Explosion may occur on addition of Pt-black to concentrated H_2O_2.[7]

Lithium. Violent reaction at about 540°C.[8]

Selenium and Tellurium. Reacts incandescently when heated with selenium or tellurium.[9]

Usage Notes on Hydrogenation Catalysts

Many hydrogenation catalysts are sufficiently active to effect rapid interaction of hydrogen and/or solvent vapor with air, causing ignition or explosion. This is particularly so where hydrogen is adsorbed on the catalyst either before a hydrogenation or after a hydrogenation during separation of catalyst from the reaction mixture. Exposure to air of such a catalyst should be avoided until complete purging with an inert gas, such as nitrogen, has been effected.

With catalysts of high activity and readily reducible substrates, control of the exotherm may be required to prevent runaway reactions, particularly at high pressures.[10]

Platinum-metal catalysts are preferably introduced to the reactor or hydrogenation system in the form of a water-wet paste or slurry. The latter is charged to the empty reactor; air is removed by purging with nitrogen or by several evacuations alternating with nitrogen filling; and the reaction mixture is charged, after which hydrogen is admitted. The same procedure applies where it is mandatory to charge the catalyst in the dry state, but in this case the complete removal of air before introduction of the reaction mixture and/or hydrogen is of vital importance.

Platinum catalysts are nonpyrophoric as normally manufactured.

Physiological Properties and Health Hazards

Dust is irritating to the eyes, skin, mucous membranes, and respiratory system.[11]

Waste Disposal

Spent catalysts should be purged from hydrogen and washed free from organics with water before storage in the water-wet condition. Under no circumstances should any attempt be made to dry a spent catalyst.

Spent catalysts should be kept under water and recycled.[12–15]

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POLYCHLORINATED BIPHENYLS $C_{12}H_xCl_{10-x}$

ANIMAL CARCINOGEN

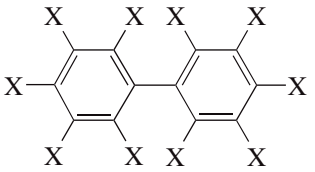
Physical Properties

Extremely stable compounds; high resistance to thermal and chemical degradation; properties vary with mixtures.[1] Mobile oil to white solid; bp, 274–475°C.[2]

Chemical Properties

Low water solubilities; low volatilities; high organic solvent solubilities.[1]

Structure



Physiological Properties and Health Hazards

Exposure may result in chloracne, pigmentation, swelling of eyelids, eye discharge, and deformation of nails.[1] Animal carcinogen and teratogen.[2–4] Reasonably anticipated to be a human carcinogen.[2] LD_{50} (oral, weanling rat) (Aroclor 1254) 1295 mg/kg. LD_{50} (oral, weanling rat) (Aroclor 1260) 1315 mg/kg.[5]

Spillage Disposal

Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a plastic container, label, and send for burning in a high-temperature incinerator equipped with afterburner and scrubber.[1,6]

Waste Disposal

Because of the stability of polychlorinated biphenyls, a high-temperature incinerator (1200–1600°C) is required to completely decompose them. The incinerator should be equipped with an afterburner and scrubber.[1]

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POTASSIUM K

VIOLENT REACTION WITH WATER, CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GASES, CAUSES BURNS

Physical Properties

Soft, silvery white metal; tarnishes on exposure to air; mp, 63°C.[1]

Fire Hazard

A fire resulting from the ignition of potassium metal is best extinguished by smothering it with dry sodium carbonate or dry chemical. Do not use carbon dioxide or water.[2]

Chemical Properties

Reacts violently with water with the formation of potassium hydroxide and hydrogen gas, which will ignite. Explosions have occurred when old, heavily crusted potassium metal has been cut with a knife.[3] Dispose of such old stock by dissolving it, uncut, in *tert*-butyl alcohol.[4]

The following procedure is recommended for the safe handling of potassium. The metal may be cut conveniently under xylene (which has been dried over sodium wire) contained in a mortar. A beaker or crystallizing dish should not be used because it is too fragile. Each scrap obtained in cutting off the outer oxide-coated surface of the metal should be immediately transferred with tweezers to a second deep mortar containing dry xylene. The accumulated residues are decomposed as described in the waste disposal procedure as soon as the cutting operation is completed. To weigh the freshly cut metal, it may be removed with tweezers, blotted rapidly with a piece of filter paper, and introduced into a tared beaker containing dry xylene. The weighed potassium is then introduced into the reaction mixture. Proper precautions, such as exclusion of air and moisture and rate of addition, depend on the nature of the reaction involved.

> ***CAUTION:*** The small scraps of metal that adhere to the knife or float on top of the xylene are most likely to start a fire.[5]

Hazardous Reactions

Detailed laboratory procedures for the safe handling of K have been published.[6]

Air. In contact with moist air, rapid oxidation occurs, which may become so fast as to result in melting or ignition.[7] On prolonged exposure to air, a coating of yellow potassium superoxide (KO_2) is formed. Percussion or dry cutting brings the metal in contact with the superoxide and a violent explosion occurs.[8–10] K should be stored under dry xylene in airtight containers.[8]

Alcohols. Reacts explosively with methanol, ethanol, *n*-propanol to *n*-octanol, benzyl alcohol, and cyclohexanol, unless air in the reaction vessel is displaced by N_2.[11]

Carbon. Vigorous exothermic reaction with soot, graphite, or activated charcoal.[12–14]

Carbon Disulfide. Mixtures explode on impact but not on heating.[15]

Dimethyl Sulfoxide. Violent interaction with potassium "sand."[16]

Halocarbons. Mixtures of K and a wide range of halocarbons are shock sensitive and may explode violently. Explosions have been recorded with mono-, di-, tri-, tetra-, and pentachloroethane; bromoform; dibromo- and diiodomethane; carbon tetrachloride; and tetrachloroethylene.[17–19]

Halogens. Ignites in F_2 and dry Cl_2; incandesces in Br_2 vapor and on heating with I_2 and explodes violently in liquid Br_2.[20]

Hydrazine. Explosive interaction.[21]

Hydrogen Iodide. Mixture is impact sensitive.[22]

Mercury. Reaction to form amalgams is vigorous or violent.[20]

Metal Halides. Reacts explosively or violently with the following: calcium bromide; iron(III) bromide or chloride; iron(II) bromide or iodide; cobalt(II) chloride; silver fluoride; all four mercury(II) halides; copper(I) chloride, bromide or iodide; copper(II) chloride and bromide; ammonium tetrachlorocuprate; zinc and cadmium chlorides, bromides, and iodides; aluminum fluoride, chloride, and bromide; thallium(I) bromide; tin(II) or (IV) chloride; tin(IV) iodide; arsenic trichloride and triiodide; antimony and bismuth trichlorides, tribromides, and triiodides; vanadium(V) chloride; chromium(IV) chloride; manganese(II) and iron(II) chlorides; and nickel chloride, bromide, and iodide.[17,22–25]

Metal Oxides. Reacts explosively with lead peroxide,[24] and on warming with Cu(II) oxide,[26] Hg(II) oxide and Hg(I) oxide,[27] tin(IV) oxide,[28] and molybdenum(III) oxide.[29]

Nitric Acid. Explosive reaction with concentrated acid.[30]

Nonmetal Halides. Reacts explosively or forms shock-sensitive mixtures with diselenium dichloride and seleninyl chloride,[31,32] PCl_3 vapor or liquid,[33] sulfur dichloride or dibromide, and PBr_3 and phosgene.[32]

Nonmetal Oxides. Forms shock-sensitive mixtures with solid CO_2 and CO.[34]

Oxalyl Dihalides. Forms shock-sensitive mixtures with oxalyl dibromide and dichloride.[17,35]

Oxidants. Reactions with oxidants are usually explosive.[24]

Sulfuric Acid. Reaction is explosive.[36]

Water. Heat evolved is enough to ignite liberated hydrogen; large pieces of the metal explode on the surface of water, scattering burning particles over a wide area.[20,37]

Physiological Properties and Health Hazards

Reaction of the metal with skin moisture yields potassium hydroxide and can cause thermal and caustic burns of the skin and eyes. Avoid contact with skin and eyes.[3]

Spillage Disposal

Potassium metal. Instruct others to maintain a safe distance. Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover with dry sodium carbonate or calcium carbonate, scoop into a dry plastic pail, and transport to fume hood.[3] Behind a shield, add a little at a time to a large excess of dry *tert*-butyl alcohol. Allow it to stand for 24 hours, and then wash into the drain, diluting greatly with at least 50 times its volume of water.[4]

Waste Disposal

Danger! Potassium residues have been known to explode even when under a protective liquid. It is therefore important that all such residues be decomposed immediately; under no circumstances should they be stored. The mortar containing the scraps is moved to the rear of the hood and *tert*-butyl alcohol is added in small portions from a medicine dropper or beaker at such a rate that the reaction does not become too vigorous. A square sheet of asbestos large enough to cover the mortar should be at hand. If the liquid should catch fire, it may be extinguished easily by covering the mortar with the asbestos sheet. There should be no other inflammable material or flames in the hood during this treatment. Sufficient *tert*-butyl alcohol must be employed to ensure complete decomposition of all the potassium.[38]

Package Lots. Keep metal under dry xylene and place in a separate labeled container for recycling or disposal. Larger quantities should be burned in an open furnace.[39]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Equip a dry, three-necked, round-bottom flask with a stirrer, dropping funnel, condenser, and heating mantle. Flush the flask with nitrogen and place the potassium in it. Add 21 mL of *tert*-butyl alcohol per gram of potassium at such a rate that rapid refluxing occurs. Stirring is started as soon as enough t-butyl alcohol has been added. Stir the mixture and heat under reflux until the potassium is dissolved. Turn off the heat and add an equal volume of water at a rate that causes mild refluxing. Cool the solution, neutralize with 5% hydrochloric acid, and wash into the drain.[40]

Reactions for Spillage and Waste Disposal

$$2K + 2(CH_3)_3OH \rightarrow \underset{\substack{\text{potassium}\\ \textit{tert}\text{-butoxide}}}{2(CH_3)_3COK} + H_2$$

$$(CH_3)_3COK + H_2O \rightarrow \underset{\substack{\textit{tert}\text{-butyl}\\ \text{alcohol}}}{(CH_3)_3COH} + KOH$$

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POTASSIUM AMIDE KNH_2

FLAMMABLE

Physical Properties

White or yellow-green hydroscopic crystals; mp, 335°C; sublimes at 400°C.[1]

Chemical Properties

Similar properties to the much more widely investigated sodium amide, but may be expected on general grounds to be more violently reactive. The frequent fires or explosions observed during work-up of reaction mixtures involving the amide were attributed to presence of unreacted (oxide-coated) particles of potassium in the amide solution in ammonia. A safe filtration technique is described.[2]

Hazardous Reactions

More violently reactive than sodium amide.[3]

Ammonia and Copper Nitrate. Explosive precipitate formed.[4]

Potassium Nitrite. Explodes when heated with potassium nitrite under vacuum.[5]

Water. Violent reaction; ignition may occur.[6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, nitrile rubber gloves, and a laboratory coat. Work in the fume hood. Cover the potassium amide with toluene or kerosene and add 95% ethanol slowly while stirring.[7] When reaction is complete, the solution should be placed in a separate labeled container for disposal by burning. *Alternatively*, small portions of potassium amide can be stirred into excess solid ammonium chloride in the fume hood. The resulting mixture is slowly added to a pail of cold water and the solution washed into the drain.[8,9]

Reactions for Spillage and Waste Disposal

$$1)\ KNH_2 + CH_3CH_2OH \rightarrow CH_3CH_2O^-K^+ + NH_3$$

$$2)\ KNH_2 + NH_4Cl \rightarrow \underset{\text{potassium chloride}}{KCl} + 2NH_3$$

REFERENCES

1. CRC.
2. Brandsma, L., *Preparative Acetylenic Chemistry*, Elsevier, Amsterdam, New York, 1971, 20, 21.
3. Lux 546.
4. B 1086.
5. Bergstrom, F.W. et al., *Chem. Rev.*, 12, 64, 1933.
6. Mellor, Vol. 8, 255, 1940.
7. Bergstrom, F.W., *Org. Synth.*, Coll. Vol. 3, 782, 1955.
8. PP 89.
9. F & F 1, 1034.

POTASSIUM CHLORATE $KClO_3$

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIAL

Physical Properties

Colorless lustrous crystals; mp, 368°C.[1]

Fire Hazard

Powerful oxidizing agent. Forms explosive mixtures with combustible materials. Extinguish with water.[2]

Chemical Properties

Soluble in hot water; insoluble in cold water.[2]

Hazardous Reactions

Ammonia or Ammonium Sulfate. Vigorous reaction with high concentrations of NH_3; on heating, a mixture with $(NH_4)_2SO_4$ decomposes with incandescence.[3]

Fabric. Fabric gloves (wrongly used in place of impervious plastic gloves) became impregnated during handling operations and subsequently ignited from cigarette ash.[4]

Hydrocarbons. Violent explosions occur on mixing powdered potassium chlorate with hydrocarbons.[4]

Hydrogen Iodide. Molten potassium chlorate ignites HI gas.[5]

Manganese Dioxide. Explosion may occur on heating mixture.[6]

Manganese Dioxide and Potassium Hydroxide. Mixture explodes on heating above 80–90°C at pressures above 19 kbar.[7]

Metals. Sensitive explosives are formed by mixtures with finely divided Al, Cu, Mg,[5,8] or Zn.[9] Mixtures with Cr,[10] Ge, Ti, and Zr incandesce or explode on heating.[11]

Metal Sulfides. Sensitive explosives are formed with many metal sulfides.[5,12,13]

Metal Thiocyanates. Mixtures are friction and heat sensitive.[4]

Nitric Acid and Organic Matter. In destroying organic matter prior to analysis, not more than 0.3 g of chlorate should be added or explosive oxidation may result.[14]

Nonmetals. Mixtures with arsenic,[5] carbon,[5] phosphorus,[5,15] sulfur,[16] or other readily oxidized material may explode violently.

Sodium Amide. Mixture explodes.[17]

Sulfuric Acid. Addition of $KClO_3$ in portions to H_2SO_4 below 60°C and above 200°C causes brisk effervescence; between these temperatures, explosions are caused by the ClO_2 produced.[18]

Physiological Properties and Health Hazards

Swallowing may irritate the intestinal tract and kidneys. Avoid contact with skin, eyes, and clothing.[19]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, a face shield, and goggles. Scoop the spilled chlorate into a container and transport to the fume hood. Add to sufficient water to give an approximately 10% solution. For each 10 mL of solution, slowly and while stirring, add 18 mL of a 10% solution of sodium bisulfite. Neutralize with sodium carbonate and wash into the drain. Wash the spill site and any contaminated clothing thoroughly with water.[20]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Prepare a 10% aqueous solution of the waste potassium chlorate. For each 10 mL of solution, slowly and while stirring, add 18 mL of a 10% solution of sodium bisulfite. Neutralize with sodium carbonate and pour into the drain with at least 50 times its volume of water.[20]

Reactions for Spillage and Waste Disposal

$$ClO_3^- + 3HSO_3^- \rightarrow Cl^- + 3SO_4^{2-} + 3H^+$$

REFERENCES

1. Merck 7703.
2. NFPA 49.
3. Mellor, Vol. 2, 702, 1941; Vol. 8, 217, 1940.
4. B 972–974.
5. Mellor, Vol. 2, 310, 1941.
6. Mellor, J.W., *Modern Inorganic Chemistry*, revised and edited by G.D. Parkes, Longman's Green, London, 1961, p. 333.
7. Molinari, E. et al., *Inorg. Chem.*, 3, 898, 1964.
8. Anonymous, *Chem. Eng. News*, 14, 451, 1936.
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10. Mellor, Vol. 11, 163, 1943.
11. Mellor, Vol. 7, 20, 116, 260, 1941.
12. Mellor, Vol. 9, 523, 1939.
13. Mellor, Vol. 3, 447, 1941.
14. Asthana, S.S. et al., *Chem. Ind.*, 953, 1976.
15. Mellor, Vol. 8, 785, 1940.
16. Tanner, H.G., *J. Chem. Educ.*, 36, 59, 1959.
17. Mellor, Vol. 8, 258, 1940.
18. Mellor, Vol. 10, 435, 1947.
19. Lux 546.
20. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988, 1988.

POTASSIUM CHROMATE K_2CrO_4

HUMAN CARCINOGEN AS DUST, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

Lemon-yellow crystals; mp, 975°C.[1]

Chemical Properties

Soluble in water.[1]

Physiological Properties and Health Hazards

Dust irritates the respiratory tract and eyes. Swallowing causes irritation and internal damage. Prolonged exposure causes ulceration, damage to liver and kidneys, and even cancer. Carcinogen as dust. Avoid inhaling dust. Avoid contact with eyes and skin.[2] TLV-TWA 0.05 mg/m^3.[3]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a container of water. Carefully acidify this solution with 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water) until a pH of 1 (to pHydrion paper) is obtained. Slowly, and while stirring, add solid sodium thiosulfate until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Let the mixture stand for a week or filter immediately through Celite. After standing, much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or is filtered off. The liquid, in both procedures, can be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, dried, packaged, labeled, and sent to a secure landfill site.[4] Spill site and contaminated clothing should be washed thoroughly with soap and water to remove all oxidant.[2]

Waste Disposal

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. For each 2-g quantity of potassium chromate, add at least 100 mL of H_2O. Acidify with approximately 55 mL of 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water). The color of the solution is orange. While stirring, add sodium thiosulfate (about 10 g). The solution becomes blue colored and cloudy. After all of the sodium thiosulfate has disappeared, neutralize the solution with sodium carbonate (about 10 g). A blue-gray flocculent precipitate forms. The mixture can be filtered immediately with Celite or allowed to stand for 1 week when much of the supernatant liquid can be decanted. The liquid can be washed into the drain as it contains less than 0.5 ppm of chromium. The solid residue should be washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to a secure landfill site.[4]

Reactions for Spillage and Waste Disposal

$$2K_2CrO_4 + 3Na_2S_2O_3 + 5H_2O \rightarrow \underset{\substack{\text{chromium}\\\text{hydroxide}\\\text{(insoluble)}}}{2Cr(OH)_3} + 3Na_2SO_4 + 3S + 4KOH$$

REFERENCES

1. Merck 7705.
2. Ald 2897D.
3. ACGIH 23.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

POTASSIUM CYANIDE KCN

VIOLENT POISON

Physical Properties

White deliquescent granular powder or fused pieces; odor of bitter almond; mp, 634°C.[1]

Fire Hazard

Contact with acids liberates highly flammable hydrogen cyanide. Extinguish fire with dry chemical; do not use carbon dioxide.[2]

Chemical Properties

Soluble in water; strongly alkaline.[1]

Hazardous Reactions

Oxidants. Oxidized violently or explosively in contact with hot oxidants.

Sodium Nitrite. Mixture explodes on heating.[3]

Physiological Properties and Health Hazards

Swallowing or absorption of solution through the skin results in severe poisoning. The early symptoms are general weakness and heaviness of the arms and legs, increased difficulty breathing, headache, dizziness, nausea, and vomiting, and these may be rapidly followed by pallor, unconsciousness, cessation of breathing, and death.[4] TLV-STEL-C (as CN) 5 mg/m^3 (skin).[5]

Spillage Disposal

Potassium Cyanide Solutions. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Instruct others to keep a safe distance. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Slowly, and while stirring, add the slurry to a pail containing household bleach (about 70 mL/g of cyanide). Test the solution for the presence of cyanide using the Prussian blue test. To 1 mL of the solution, add 2 drops of a freshly prepared 5% aqueous ferrous sulfate solution. Boil the mixture for at least 60 seconds, cool to room temperature and add 2 drops of 1% ferric chloride solution. Add 6 M hydrochloric acid (prepared by cautiously adding a volume of concentrated acid to an equal volume of cold water) until the mixture is acid to litmus. If cyanide remains in the solution, a deep blue precipitate forms. (Cyanide concentrations greater than 1 ppm can be detected.) If the test is positive, more bleach is added to the cyanide solution and the test repeated. When the test is negative, the solution is washed into the drain with at least 50 times its volume of water. Wash the area of the spill thoroughly with water. The solid residue may be disposed of with normal refuse.[4,6,7]

Solid Potassium Cyanide. Wear nitrile rubber gloves, laboratory coat, and eye protection. Scoop the solid into a container and transport to the fume hood. Add to 1% sodium hydroxide solution (about 50 mL/g of cyanide). While stirring, slowly add household bleach (about 70 mL/g of cyanide). Continue as for solution spills. Wash the area of the spill thoroughly with soap and water.[4,6,7]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, add to a 1% solution of sodium hydroxide (about 50 mL/g of cyanide). While stirring, slowly add household bleach (about 70 mL/g of cyanide). Continue as for solution spills.[4,6,7]

Reactions for Spillage and Waste Disposal

$$CN^- + ClO^- \rightarrow \underset{\text{cyanate}}{CNO^-} + Cl^-$$

REFERENCES

1. Merck 7709.
2. NFPA 49.
3. Mellor, Vol. 8, 478, 1940.
4. Lux 119.
5. ACGIH 36.
6. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 285.
7. ITI 529; Ald 2898C.

POTASSIUM DICHROMATE $K_2Cr_2O_7$

HUMAN CARCINOGEN AS DUST, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

Bright orange-red crystals; not hygroscopic or deliquescent; mp, 398°C.[1]

Chemical Properties

Soluble in water; oxidizing agent.[1]

Hazardous Reactions

Boron. Mixtures of boron and potassium dichromate with or without silicon may ignite.[2]

Hydroxylamine. Aqueous hydroxylamine ignites on contact with potassium dichromate moistened with sulfuric acid. The anhydrous base explodes violently on contact with potassium dichromate.[3]

Physiological Properties and Health Hazards

Dust is destructive to the tissues of mucous membranes, respiratory tract, and eyes. Swallowing causes irritation and internal damage and may be fatal. Prolonged skin exposure can result in ulceration, damage to liver and kidneys, and even cancer. Avoid inhaling dust. Avoid contact with eyes and skin.[4] TLV-TWA 0.05 mg/m^3.[5]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a container of water in the fume hood. Carefully acidify this solution with 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water) until a pH of 1 (to pHydrion paper) is obtained. Slowly, and while stirring, add solid sodium thiosulfate until the solution becomes cloudy and blue colored. Neutralize the solution by adding sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Let the mixture stand for a week or filter immediately through Celite. After standing, much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or the solid filtered. The liquid in both methods can be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, dried, packaged, labeled, and sent to a secure landfill site.[6] The spill site and contaminated clothing should be washed thoroughly with soap and water to remove the oxidant.[4]

Waste Disposal

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add solid potassium dichromate to a container of water (about 100 mL/5 g). Acidify with 3 M sulfuric acid (35–55 mL, pH 1 with pHydrion paper). While stirring, slowly add solid sodium thiosulfate (about 13.5 g) until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes a blue-gray flocculent precipitate is formed. Let the mixture stand for a week or filter immediately through Celite. After a week, much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or the solid filtered. The liquid may be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to a secure landfill site.[6]

Reactions for Spillage and Waste Disposal

$$Cr_2O_7^{2-} + 3S_2O_3^{2-} + 2H_3O^+ \rightarrow \underset{\substack{\text{chromium}\\ \text{hydroxide}\\ \text{(insoluble)}}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

REFERENCES

1. Merck 7710.
2. B 56.
3. Mellor, Vol. 3, 670, 1941; Vol. 8, 287, 1940.
4. Ald 2899.
5. ACGIH 23.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

POTASSIUM FLUORIDE KF

POISONOUS

Physical Properties

White deliquescent powder or solid; bp, 859.9°C.[1]

Chemical Properties

Freely soluble in boiling water; soluble in water and hydrofluoric acid; insoluble in alcohol. Aqueous solution erodes glass and porcelain.[1]

Physiological Properties and Health Hazards

Breathing dust or swallowing the powder will cause severe poisoning. Irritates the skin, eyes, mucous membranes, and tissue. Prolonged contact can cause shortness of breath, cough, elevated temperature, and cyanosis.[2] TLV-STEL-C (as F) 3.0 ppm.[3]

Spillage Disposal

Wear a face shield and goggles, laboratory coat, and butyl rubber gloves. Sweep potassium fluoride into a large beaker. In the fume hood, add water (about 13 mL/g of fluoride) and solid calcium hydroxide (about 0.6 g/g of fluoride) to the beaker, and stir for 24 hours. Filter. Wash the filtrate. The solid residue can be discarded with normal refuse.[4,5]

Waste Disposal

Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. In a beaker, add 0.6 g of calcium hydroxide and 13 mL of water for every g of fluoride. Stir the resulting mixture for 24 hours. Filter. Wash the filtrate into the drain. The solid residue can be discarded with normal refuse.[4,5]

Reactions for Spillage and Waste Disposal

$$2KF + Ca(OH)_2 \rightarrow \underset{\text{calcium fluoride}}{CaF_2} + 2KOH$$

REFERENCES

1. Merck 7714.
2. Lux 548.
3. ACGIH 36.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.
5. ITI 532.

POTASSIUM HYDRIDE KH

CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GAS

Physical Properties

White needles that decompose on heating.[1]

Chemical Properties

Decomposes on contact with water.[1]

Hazardous Reactions

Air. Ignites on exposure to air.[2]

Fluorine. Ignites on contact at room temperature.[3]

Spillage Disposal

Wear nitrile rubber gloves, fireproof clothing, goggles, and face shield. Work from behind body shield where possible. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Carefully transfer to a pail in the fume hood. Slowly and cautiously add butanol (about 10 mol solvent/mol hydride or about 22 mL per gram) to the hydride until the reaction ceases. Then add water very carefully until last of hydride is destroyed. Let stand until solids settle. Decant liquid into the drain. The solid residue may be disposed of with normal refuse.[4]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, face shield and goggles. Work from behind a body shield. In the fume hood, mix the hydride with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Slowly and cautiously add butanol (about 22 mL solvent/g of hydride) until the reaction ceases. Then add water carefully until all of the hydride is destroyed. Let stand until solids settle. Decant liquid into the drain. The solid residue may be discarded with normal refuse.[4]

Reactions for Spillage and Waste Disposal

$$2KH + 2CH_3CH_2CH_2CH_2OH \rightarrow \underset{\text{potassium butoxide}}{KOCH_2CH_2CH_2CH_3} + H_2$$

$$KOCH_2CH_2CH_2CH_3 + H_2O \rightarrow KOH + CH_3CH_2CH_2CH_2OH$$

REFERENCES

1. CRC.
2. Brown, C.A., *J. Org. Chem.,* 39, 3913, 1974.
3. Mellor, Vol. 2, 12, 483, 1940; Vol. 2, Suppl. 1, 56, 1956; Vol. 3, 73, 1941.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

POTASSIUM HYDROXIDE KOH

CORROSIVE, CAUSES SEVERE BURNS

Physical Properties

White or slightly yellow lumps, rods, or pellets; mp, 360°C (anhydrous 380°C).[1]

Chemical Properties

Very soluble in water; rapidly absorbs carbon dioxide and water from the air; deliquescent.[1]

Hazardous Reactions

Acetic Acid. Addition of acetic acid to potassium hydroxide may cause eruption of the mixture.[2]

Ammonium Hexachloroplatinate. Heating with potassium hydroxide yields product, which when dry, explodes on contact with combustible materials.[3]

Chlorine Dioxide. Explodes on contact with solid potassium hydroxide or a concentrated solution.[4]

Germanium. Incandesces on contact with the fused hydroxide.[5]

Hyponitrous Acid. Ignites on contact with solid potassium hydroxide.[5]

Maleic Anhydride. Decomposes violently in potassium hydroxide solution above 150°C.[6]

Nitroalkanes. Potassium salts of aci-nitroparaffins may be produced from nitroalkanes and potassium hydroxide. These salts are explosive when dry.[7]

Nitrobenzene. Heating with powdered potassium hydroxide results in violent conversion to *o*-nitrophenol.[8,9]

Nitrogen Trichloride. Potassium hydroxide solution initiates the violent decomposition of the trichloride.[10]

Potassium Peroxodisulfate. Ignites on addition of a very small quantity of moist potassium hydroxide.[11]

Tetrahydrofuran. Tetrahydrofuran should not be dried over potassium hydroxide unless it has been shown to be free of peroxides.[12]

2,4,6-Trinitrotoluene. Interaction with potassium hydroxide in methanol at –65°C produces explosive aci-nitro salts. Added potassium hydroxide reduces the explosion temperature of TNT to 160°C.[13]

Water. Strongly exothermic reaction with limited amounts of water.[5]

Physiological Properties and Health Hazards

Extremely corrosive to tissue; ingestion causes violent pain, damage to esophagus, and severe internal irritation.[14] TLV-STEL-C 2 mg/m^3.[15]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Shovel into bucket and add, a little at a time and while stirring, to a large volume of ice water. Neutralize with 5% hydrochloric acid and wash into the drain.[16] Spill site should be washed thoroughly with water.[14]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Add slowly while stirring to a large volume of ice water. Neutralize with 5% hydrochloric acid and wash into the drain.[16,17]

Reactions for Spillage and Waste Disposal

$$KOH + HCl \rightarrow KCl + H_2O$$

potassium chloride

REFERENCES

1. Merck 7724.
2. B 1143.
3. Mellor, Vol. 16, 336, 1942.
4. Mellor, Vol. 2, 289, 1941.
5. Mellor, Vol. 8, 407, 1940.
6. B 418.
7. Watts, C.E., *Chem. Eng. News*, 30, 2344, 1952.
8. Wohl, A., *Chemische Berichte*, 32, 3846; 1901, 34, 2444, 1899.
9. Bretherick, L., *Chem. Ind.*, 576, 1976.
10. Mellor, Vol. 8, 601, 1940; Vol. 8, Suppl. 2.2, 412, 1967.
11. B 1307.
12. Anonymous, *Org. Synth.*, 46, 105, 1966.
13. B 694.
14. Lux 549.
15. ACGIH 49.
16. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.4.
17. Ald 2905.

POTASSIUM NITRATE KNO_3

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIALS

Physical Properties

Colorless crystals; mp, 333°C.[1]

Fire Hazard

Mixtures of potassium nitrate and combustible materials are readily ignited; mixtures with finely divided combustible materials can react explosively.[2]

Chemical Properties

Soluble in water and glycerol; insoluble in absolute alcohol.[1]

Hazardous Reactions

Metals. Mixtures with powdered Ti, Sb, or Ge explode on heating.[3]

Metal Sulfides. Mixtures with antimony trisulfide, barium or calcium sulfides, or germanium monosulfide or titanium disulfide explode on heating[4–6]; mixtures with arsenic disulfide[6] or molybdenum disulfide[7] are detonatable.

Nonmetals. Finely divided mixture with boron ignites and explodes on percussion[8,9]; the oldest explosive, gunpowder, is a mixture of the nitrate with charcoal and sulfur[10]; mixture with red phosphorus reacts vigorously on heating[11]; mixtures with arsenic explode when ignited.[12]

Organic Materials. Mixtures with combustible materials (e.g., jute) are easily ignited.[13]

Reductants. Mixtures with sodium phosphinate and sodium thiosulfate are explosive.[10]

Sodium Acetate. Mixtures may be explosive.[14]

Physiological Properties and Health Hazards

Swallowing large quantities may produce gastroenteritis. Prolonged exposure to small quantities may result in anemia. LD_{50} (oral, rabbit) 1.166 g anion/kg.[1]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Shovel into bucket of water and wash the solution into the drain with water. Spill site should be washed thoroughly to remove all oxidant, which is liable to render any organic matter (particularly wood, paper, and textiles) with which it comes into contact, dangerously combustible when dry. Clothing wetted with the solution should be washed thoroughly.[2,15]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Add product to a large excess of water. Slowly add dry sodium carbonate while stirring. After 24 hours, neutralize (1:1 aqueous hydrochloric acid), and wash down the drain with at least 50 times its volume of water.[2,15]

REFERENCES

1. Merck 7733.
2. Lux 550.
3. Mellor, Vol. 7, 20, 116, 261, 1941; Vol. 9, 382, 1939.
4. Mellor, Vol. 9, 270, 524, 1939.
5. Mellor, Vol. 3, 745, 1941.
6. Mellor, Vol. 7, 91, 274, 1941.
7. Mellor, Vol. 11, 647, 1943.
8. B 1302.
9. Mellor, Vol. 5, 16, 1946.
10. Mellor, Vol. 2, 820, 825, 1941; Vol. 2, Suppl. 2.2, 1939, 1963.
11. Mellor, Vol. 8, 788, 1940.
12. Mellor, Vol. 9, 35, 1939.
13. B 1303.
14. Pieters, H.A.J. and Creyghton, J.W., *Safety in the Chemical Laboratory*, 2nd ed., Butterworth Scientific Publications, London, 1957, p. 30.
15. PPL 171.

POTASSIUM NITRITE KNO_2

CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE, HARMFUL IF TAKEN INTERNALLY

Physical Properties

White or slightly yellow, deliquescent, crystalline solid; mp, 441°C (decomposition starts at 350°C).[1]

Fire Hazard

Avoid contact with clothing and other absorbent fabrics.[2]

Chemical Properties

Soluble in water; slightly soluble in alcohol. Decomposed even by weak acids with evolution of brown fumes of nitrogen dioxide.[1]

Physiological Properties and Health Hazards

Toxic if swallowed.[2] LD_{50} (oral, rabbit) 108 mg anion/kg.[1]

Hazardous Reactions

Ammonium Sulfate. Addition to the fused nitrite causes effervescence and ignition.[3]

Boron. Addition to the fused nitrite causes violent decomposition.[4]

Potassium Amide. Explosion occurs when mixture with potassium amide is heated under vacuum.[5]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Scoop into a pail of water. In the fume hood, slowly add a freshly prepared 10% solution of sodium bisulfite or sodium metabisulfite (50 mL/g or 75 mL/g, respectively). Test for presence of nitrite using starch-iodide paper dipped in dilute acetic acid.[7] When nitrite is completely destroyed, decant the liquid to the drain.[6] Spill site should be washed thoroughly with water to remove all oxidant, which is liable to render any organic matter (e.g., wood, paper, textiles) dangerously combustible when dry. Clothing wetted with the solution should be removed and washed immediately.[2]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add nitrite to a large excess of water (30–50 mL/g). Slowly add freshly prepared 10% sodium bisulfite (50 mL/g) or sodium metabisulfite (75 mL/g) until reaction is complete (about 5 minutes). Test for presence of nitrite with starch-iodide paper dipped in dilute acetic acid. When reaction is complete, neutralize if necessary, and then wash the solution into the drain with at least 50 volumes of water.[6,7]

Nitrites in aqueous solution can also be destroyed by adding about 50% excess aqueous ammonia and acidifying to pH 1 (pH paper) with hydrochloric acid. Wash the solution into the drain with 50 times its volume of water.[8]

Reactions for Spillage and Waste Disposal

$$1)\ 2KNO_2 + 3HSO_3^- + H_2O \rightarrow N_2 + 3HSO_4^- + 2KOH$$

$$2)\ KNO_2 + NH_3 \rightarrow N_2 + H_2O + KOH$$

REFERENCES

1. Merck 7734.
2. Lux 550.
3. Mellor, Vol. 2, 702, 1941.
4. Mellor, Vol. 5, 16, 1946.
5. Bergstrom, F.W. et al., *Chem. Rev.*, 12, 64, 1933.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *Laboratory Disposal Method for Potassium Nitrite,* University of Alberta, Edmonton, 2001.
7. Vogel, A.I., *A Textbook of Macro and Semimicro Qualitative Inorganic Analysis*, 4th ed., Longmans, Green and Co., Toronto, 1954, p. 338.
8. PP 84; PPL 170.

POTASSIUM PERMANGANATE $KMnO_4$

POWERFUL OXIDANT

Physical Properties

Dark purple crystals; dec, 240°C.[1]

Fire Hazard

Powerful oxidizing material that may react violently with easily oxidizable substances. Extinguish using water.[2]

Chemical Properties

Soluble in water; decomposed by ethanol.[1]

Hazardous Reactions

Acetic Acid or Acetic Anhydride. Risk of explosion.[3]

Acetone and *tert*-Butylamine. Mixture leads to violent reaction and oxidation of this amine should be run in water rather than 20% aqueous acetone.[3–5]

Alcohols and Nitric Acid. Addition of methanol, ethanol, 2-propanol, or amyl or isoamyl alcohols to a mixture of red fuming nitric acid and $KMnO_4$ caused immediate ignition.[6]

Ammonia and Sulfuric Acid. A mixture of $KMnO_4$ in H_2SO_4 oxidizes ammonia with incandescence.[7]

Ammonium Nitrate. Mixture of 0.5% $KMnO_4$ in ammonium nitrate exploded 7 hours later.[6]

Antimony or Arsenic. Antimony ignites on grinding in a mortar with $KMnO_4$ while arsenic explodes.[8]

Dichloromethylsilane. Mixture ignites on impact.[9]

Formaldehyde. Formaldehyde gas for disinfection purposes may be released from the aqueous solution by treatment with $KMnO_4$, but the quantities used must be limited to avoid the risk of fire or explosion.[10]

Glycerol. Contact of glycerol with solid $KMnO_4$ caused a vigorous fire.[10]

Hydrochloric Acid. Reaction with hydrochloric acid is occasionally explosive.[10]

Hydrogen Peroxide. Mixtures cause a fire.[11]

Hydroxylamine. Solid ignites in contact with $KMnO_4$.[12]

Nonmetals. Mixtures with phosphorus or sulfur react explosively on grinding or heating, respectively.[13]

Sulfuric Acid and Water. Explosion follows the addition of concentrated H_2SO_4 to damp $KMnO_4$.[14]

Wood. Contact between the solid material and wood, in the presence of either moisture or mechanical friction, may cause a fire.[15,16]

Physiological Properties and Health Hazards

Dilute solutions are mildly irritating; strong solutions are caustic.[1] TLV 5 mg/m^3.

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Add to

a beaker of water (about 20 mL/g of permanganate). In the fume hood, add 1 drop of concentrated sulfuric acid for every 10 mL of solution. Slowly and while stirring, add 10% sodium bisulfite until the permanganate color is discharged and the initial brown precipitate of manganese dioxide dissolves (about 13 mL of the bisulfite solution/10 mL of permanganate solution). Neutralize with sodium carbonate. Let stand until solids settle. Decant the liquid into the drain with at least 50 times its volume of water. Solid residue may be discarded with normal refuse.[17,18]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Prepare a saturated aqueous solution of the waste potassium permanganate (about 5 g of the salt dissolves in 100 mL of water at 20°C). For each 10 mL of solution, add 1 drop of concentrated sulfuric acid. In the fume hood, slowly and while stirring, add 10% sodium bisulfite solution until the permanganate color is discharged and the initial brown precipitate of manganese dioxide dissolves. About 13 mL of the bisulfite solution is required for each 10 mL of permanganate solution. Neutralize with sodium carbonate if necessary. Wash the resulting colorless solution into the drain with at least 50 times its volume of water.[17,18]

Reactions for Spillage and Waste Disposal

$$2MnO_4^{2-} + 5HSO_3^- + H^+ \rightarrow 2Mn^{2+} + 5SO_4^{2-} + 3H_2O$$

REFERENCES

1. Merck 7740.
2. NFPA 49.
3. B 1295.
4. Kornblum, N. et al., *Org. Synth.*, 43, 89, 1963.
5. Kornblum, N. et al., *Org. Synth.*, Coll. Vol. 5, 847, 1973.
6. B 1296.
7. Mellor, Vol. 1, 907, 1941.
8. Mellor, Vol. 12, 322, 1942.
9. Mueller, R. et al., J. *Prakt. Chem.*, 31, 1, 1966.
10. B 1297.
11. Anonymous, *J. Pharm. Chim.*, 6, 410, 1927.
12. Mellor, Vol. 3, 670, 1930; Vol. 8, 287, 1940.
13. Mellor, Vol. 12, 319, 1942.
14. Archer, J.R., *Chem. Eng. News*, 26, 205, 1948.
15. B 1300.
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17. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 284.
18. ITI 536.

PROCARBAZINE HYDROCHLORIDE $C_{12}H_{19}N_3O \cdot HCl$

ANTINEOPLASTIC ANIMAL CARCINOGEN

Synonyms

p-Toluamide-N-isopropyl-α-2-methylhydrazino hydrochloride, NSC 77213, matulane.

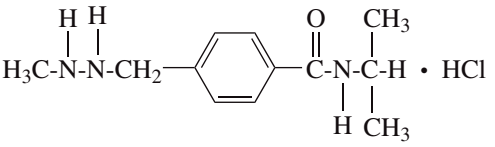
Physical Properties

White to pale yellow crystalline powder with a slight odor; mp, 233°C (dec).[1]

Chemical Properties

Soluble in water; sparingly soluble in most organic solvents.[1]

Structure



Hazardous Reactions

Slowly oxidizes in aqueous solutions containing dissolved oxygen to azoprocarbazine and hydrogen peroxide.[2]

Mode of Action

Procarbazine is an alkylating agent that reacts with RNA.[3,4]

Physiological Properties and Health Hazards

Animal carcinogen. Reasonably anticipated to be a human carcinogen and when used as a chemotherapeutic agent in combination with nitrogen mustard, a potent animal carcinogen, can result in acute nonlymphocytic leukemia.[5] Adverse effects in 50–70% of all patients treated with this drug are nausea, with vomiting, leukopenia, and thrombocytopenia. LD_{50} (oral, rat) 785 ± 34 mg/kg.[2]

Spillage Disposal

Wear protective gloves, clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container. Estimate the amount of procarbazine hydrochloride, and add sufficient water to make an approximately 1% solution. For each 100 mg of procarbazine hydrochloride, add 6 g of calcium hypochlorite and stir at room temperature for 12 hours. Decant the liquid into the drain with a large volume of water, and treat the solid residue as normal refuse.[1,6]

Waste Disposal

Wear protective gloves, clothing, and goggles. Work in the fume hood. Dissolve the waste procarbazine hydrochloride in water to make an approximately 1% solution. For each 100 mg of procarbazine hydrochloride, add 6 g of calcium hypochlorite and stir the mixture for 12 hours. The solution can be poured into the drain with a large excess of water.[1]

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2. Reed, D.J., *Handbook of Experimental Pharmacology*, 38, 1975, p. 747.
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4. Sartorelli, A.C. and Johns, D.G., Eds., *Antineoplastic and Immunosuppressive Agents*, Part I, Springer-Verlag, Berlin, New York, 1974, pp. 310–311.
5. NIEHS III–190.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

1,3-PROPANE SULTONE $C_3H_6O_3S$

ANIMAL CARCINOGEN

Synonyms

3-Hydroxy-1-propanesulfonic acid γ-sultone.

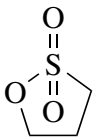
Physical Properties

Colorless liquid or white crystals[1]; mp, 30–33°C[2]; bp, 112°C.[1]

Chemical Properties

Soluble in many organic solvents (ketones, esters, and aromatic hydrocarbons); solubility in water is 100 g/L; insoluble in aliphatic hydrocarbons. Half life at 37°C in phosphate buffer (pH 7.4) is 110 minutes; hydrolyzes to 3-hydroxy-1-propanesulfonic acid.[1]

Structure



Physiological Properties and Health Hazards

Carcinogenic to rats and mice. Reasonably anticipated to be a human carcinogen.[1,3] Eye and skin exposure cause chemical burns. Mutagenic in the Ames test. LD_{50} (oral, rat) 350 mg/kg.[1] Exposure by all routes should be carefully controlled to the lowest possible levels.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. Dissolve or mix the material with a combustible solvent and package and label for disposal by burning in a chemical incinerator equipped with afterburner and scrubber.[2]

Small Quantities. Wear rubber gloves, goggles, and protective clothing. To 2 g of 1,3-propane sultone contained in a 125-mL Erlenmeyer flask, slowly add 50 mL of household bleach (5% sodium hypochlorite). Stir at room temperature for 1 hour. Wash the solution into the drain with a large volume of water.[5]

REFERENCES

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2. Ald 2951A.
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4. ACGIH 49.
5. Armour, M.A. and Renecker, D.M., *Laboratory Disposal Method for 1,3-Propane Sulfone*, University of Alberta, Edmonton, 1998.

β-PROPIOLACTONE $C_3H_4O_2$

ANIMAL CARCINOGEN, TOXIC IN CONTACT WITH SKIN, SEVERE IRRITANT

Synonyms

2-Oxetanone, hydracrylic acid β-lactone, β-propionolactone, propanolide, NSC-21626, Betaprone.

Physical Properties

Colorless liquid; bp, 162°C with decomposition.[1]

Chemical Properties

Slowly hydrolyzed in water to hydracrylic acid; miscible with alcohol, acetone, ether, and chloroform.[1]

Physiological Properties and Health Hazards

Severe skin irritant. Harmful if swallowed, inhaled, or absorbed through skin; extremely destructive to the mucous membranes and upper respiratory tract, eyes, and skin. Inhalation may be fatal as a result of spasm, inflammation, and edema of larynx and bronchi, chemical pneumonitis, and pulmonary edema. Skin cancers produced in animals.[2] An experimental carcinogen by oral, subcutaneous, intratracheal, and dermal routes.[3] Reasonably anticipated to be a human carcinogen.[4] Prevent contact with skin, eyes, and clothing. Prevent inhalation of vapor.[5] TLV-TWA 0.5 ppm (1.5 mg/m^3).[6]

Spillage Disposal

Wear eye protection, laboratory coat, butyl rubber gloves, and self-contained breathing apparatus.[2] Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to a labeled container for disposal by burning (see waste disposal procedure). Spill site should be washed thoroughly with water and soap or detergent.[2,5,7,8]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace equipped with afterburner and scrubber.[2,8]

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Add 72 mL of 2.5 M sodium hydroxide (prepared by carefully dissolving 7.2 g of NaOH in 72 mL of water) to a 100-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Place 5 mL of propiolactone (4.4 g, 0.06 mol) in the dropping funnel, and add it dropwise to the stirred basic solution at such a rate that the temperature does not exceed 45°C. When addition is complete, continue stirring for 1 hour, and then neutralize the solution with dilute acid and wash into the drain.[9]

Reactions for Spillage and Waste Disposal

$$C_3H_4O_2 + NaOH \rightarrow HOCH_2CH_2COONa$$

β-hydroxypropionic acid sodium salt

REFERENCES

1. Merck 7912.
2. Ald 2955D.
3. Sax 2907.
4. NIEHS III–192.
5. Lux 554.
6. ACGIH 50.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ*., 62, A93, 1985; Armour, M.A., *J. Chem. Educ*., 65, A64, 1988; *Haz. Mat. Spills Tec*., 29.7.
8. ITI 543.
9. PP 68.

PROPYLENEIMINE C_3H_7N

EXPERIMENTAL CARCINOGEN

Synonyms

2-Methylaziridine.

Physical Properties

Liquid[1]; bp, 66–67°C.[2]

Chemical Properties

Soluble in water and most organic solvents.[2] Emits toxic fumes of nitrogen oxides when heated to decomposition; can react with oxidizing materials.[1]

Hazardous Reactions

Acids. Polymerizes explosively in contact with acids or acidic fumes. It must be stored over solid alkali.[3]

Physiological Properties and Health Hazards

An experimental carcinogen by the dermal route. Poison by ingestion and skin contact.[1] TLV-TWA 2 ppm (4.7 mg/m^3) (skin).[4]

Spillage Disposal

Wear butyl rubber gloves, goggles, face shield, or all-purpose canister respirator and laboratory coat. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an appropriate container labeled for disposal by burning (see Waste Disposal).[2,5]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner and scrubber.[2]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. Place 7.9 g (0.12 mol) of 85% potassium hydroxide pellets in a 100-mL, three-necked, round-bottom flask equipped with a stirrer, water-cooled condenser, dropping funnel, and heating mantle or steam bath. With brisk stirring, rapidly add 31.5 mL of 95% ethanol. The potassium hydroxide dissolves within a few minutes, causing the temperature of the solution to rise to about 55°C. Heat the solution to gentle reflux and add propyleneimine (7.0 mL, 0.1 mol) dropwise at such a rate to maintain gentle reflux. Heat under reflux while stirring (to prevent bumping) for a further 2 hours. Cool, dilute the reaction mixture with water, and wash into the drain.[6]

Reactions for Spillage and Waste Disposal

$$C_3H_7N + H_2O \xrightarrow{KOH} HOCH_2CH_2CH_2NH_2$$

propanolamine

REFERENCES

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2. ITI 553.
3. B 386.
4. ACGIH 50.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.
6. PP 61.

PYRIDINE C_5H_5N

FLAMMABLE, IRRITANT

Physical Properties

Colorless liquid; characteristic disagreeable odor; sharp taste; mp, –42°C; bp, 115–116°C.[1]

Fire Hazard

Flammable.[1] Vapor forms explosive mixtures with air[2]; flash point, 20°C; explosion limits, 1.8–2.4%; auto-ignition temperature, 482°C.[3]

Chemical Properties

Forms azeotropic mixture with 3 mol of water, boiling at 92–93°C; volatile with steam; miscible with water, alcohol, acetone, ether, petroleum ether, oils, and many organic liquids; solvent for many organic and inorganic compounds.[1,4]

Hazardous Reactions

Vapors form explosive mixtures with air.[2]

Bromine Trifluoride. Reaction with bromine trifluoride gives solid that ignites when dry.[3]

Chromium Trioxide. Forms unstable complex with chromium trioxide.[2]

Dinitrogen Tetroxide. Reacts violently with liquid dinitrogen tetroxide.[5]

Fluorine. Incandesces on contact with fluorine.[3]

Formamide. Bottles containing a modified Karl Fischer reagent (pyridine, iodine, and sulfur trioxide) with formamide replacing methanol developed gas pressure during several months and exploded.[6]

Maleic Anhydride. Pyridine causes maleic anhydride to decompose exothermally.[3]

Trifluoromethyl Hypofluorite. Forms highly explosive by-product with trifluoromethyl hypofluorite in reactions when used as an acid acceptor.[7]

Physiological Properties and Health Hazards

Irritates the skin and respiratory tract. Large doses may produce kidney and liver damage.[1] Avoid contact with eyes and skin; vapor may cause headache, nausea, giddiness, and vomiting; in contact with eyes, may cause conjunctivitis; large doses taken by mouth act as a heart poison.[3] TLV-TWA 5 ppm (16 mg/m^3).[8]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a plastic pail and add enough water to dissolve the sodium carbonate. Allow the solids to settle and decant the liquid to another container. Discard the solid with normal refuse. To the liquid, add slowly (frothing will occur) 6 M sulfuric acid to pH 2. Stir into the acidified solution sufficient solid potassium permanganate so that the liquid remains purple (a drop of the liquid on a filter paper will show a purple ring). Allow the mixture to stand at room temperature for 48 hours, and then neutralize the solution by adding solid sodium carbonate (frothing will occur) or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[9]

Waste Disposal

Package Lots: Recycle or package and label for disposal by burning.[10]

Small Quantities: Wear eye protection, laboratory coat, and rubber gloves. Dissolve the pyridine (1 mL) in 50 mL of 3 M sulfuric acid (prepared by slowly adding 8 mL of concentrated sulfuric acid to 21 mL of water). Weigh 10 g of potassium permanganate and stir small portions of the solid into the pyridine solution over a period of about 1 hour. Stir the mixture at room temperature for 48 hours, and then neutralize the solution by adding solid sodium carbonate or a 10% solution of sodium hydroxide. Add solid sodium bisulfite until the solution is colorless. Decant the clear liquid into the drain and discard any brown solid with normal refuse.[9,10]

REFERENCES

1. Merck 8060.
2. ITI 557.
3. Lux 561.
4. CRC.
5. Mellor, Vol. 8, 543, 1940.
6. Anonymous, *J. Chem. Educ*., 50, A293, 1973.
7. B 127.
8. ACGIH 50.
9. Armour, M.A., *Network News* (American Chemical Society), 8, 1994, p. 1; *Haz. Mat. Spills Tec*., 29.6; Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 26, 1999.
10. Ald 2990A.

PYRIDINIUM PERCHLORATE $C_5H_5NHClO_4$

SELF-REACTIVE

Chemical Properties

Can be detonated on impact, but is normally considered a stable intermediate (mp, 288°C), suitable for purification of pyridine.[1] Occasionally explosions have occurred when the salt was disturbed,[2] which have been variously attributed to the presence of ethyl perchlorate, ammonium perchlorate, or chlorates. A safer preparative modification has been described.[3] It explodes on heating to above 335°C, or at a lower temperature if ammonium perchlorate is present.[4]

Physiological Properties and Health Hazards

Perchlorate dusts and solutions irritate the skin, eyes, and respiratory system. They are also irritating and harmful if taken internally.[5]

Hazardous Reactions

Metals. This salt may explode violently in contact with metals.[6]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[7] Dampen with water. Using a plastic scoop, cautiously transfer to a large bucket of water diluting to a concentration of less than 5%. In the fume hood, slowly and cautiously neutralize with 10% sodium hydroxide. When reaction has ceased and solids have settled, decant liquid into the drain with at least 50 times its volume of water. Solid residue may be discarded with normal refuse.[7,8] Spill site should be washed thoroughly to remove all oxidant.[5]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Carefully add solid to a large volume of water in a beaker diluting to a concentration of less than 5%. Cautiously neutralize with 10% sodium hydroxide. Flush solution down the drain with at least 50 times its volume of water.[8]

REFERENCES

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2. Anonymous, *Chemiearbeit*, 15, 19, 1963.
3. Arndt, F. et al., *Chem. Ztg.*, 74, 140, 1950.
4. B 523.
5. Lux 562.
6. Mellor, Vol. 2, Suppl. 1, 603, 1956.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
8. PPL 170.

RESORCINOL $C_6H_4(OH)_2$

IRRITANT, TOXIC

Synonyms

1,3-Benzenediol, *m*-dihydroxybenzene, resorcin.

Physical Properties

White needles. Changes to pink on exposure to light and air, or in contact with iron; mp, 110.7°C; bp, 281°C.[1]

Fire Hazard

Flammable. Flash point, 127°C; ignition temperature, 608°C; lower explosion limit, 1.4%.[2]

Chemical Properties

Soluble in water, ethanol, ether, glycerol, and benzene; slightly soluble in chloroform.[1]

Physiological Properties and Health Hazards

Irritates the skin, eyes, and mucous membranes.[1] Skin absorption, in severe cases, may result in restlessness, cyanosis, and convulsions. Swallowing may cause dizziness, drowsiness, and tremors.[3] TLV-TWA 10 ppm (45 mg/m^3); TLV-STEL 20 ppm (90 mg/m^3).[4]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Sweep up solid with brush and follow waste disposal procedure.[5,6]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning. Dissolve the compound in a flammable solvent and spray into furnace with afterburner.[5]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. A solution of 5.5 g (0.05 mol) of resorcinol in 75 mL of water is prepared in a 200-mL, three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. Ferrous sulfate heptahydrate (2.35 g, 0.0085 mol) is then dissolved in the mixture, and the pH is adjusted to 5–6 (pH paper) with dilute sulfuric acid. Then 41 mL (0.4 mol) of 30% hydrogen peroxide is added dropwise while stirring over 1 hour.

> ***CAUTION:*** The order of addition of the reagents is important. If hydrogen peroxide and ferrous sulfate are premixed, a violent reaction may occur.

Heat is evolved, and the reaction temperature is maintained at 50–60°C by adjusting the rate of addition and by using an ice bath if necessary. Stirring is continued for 2 hours while the temperature gradually falls to ambient. The solution is allowed to stand overnight and is then washed down the drain.[6]

REFERENCES

1. Merck 8240.
2. NFPA 325M.

3. Lux 563.
4. ACGIH 51.
5. ITI 560.
6. PP 63.

SELENIUM AND COMPOUNDS Se

SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING, DANGER OF CUMULATIVE EFFECTS

Physical Properties

Selenium is a steel gray or purplish powder, also fabricated into pellets, sticks, or plates. Selenium dioxide, selenous acid, and the alkali-metal selenites and selenates are colorless powders or crystals. Selenium chloride (reddish yellow), selenyl chloride (colorless or yellow) (bp, 176°C), and selenic acid (colorless) are liquids, whereas selenium tetrachloride is a cream-colored crystalline solid.[1]

Chemical Properties

Selenium powder is insoluble in water; the dioxide, selenous acid, and the alkali metal selenites are water soluble. Highly poisonous hydrogen selenide (offensive smell) is generated when an acid solution of a selenium compound is reduced by metals such as tin and zinc. Selenic acid reacts vigorously with water.[1]

Hazardous Reactions

Metal Chlorates. Selenium incandesces when mixed with any slightly moist chlorate other than alkali metal chlorates.[2]

Metals. Nickel, sodium, potassium, uranium, zinc, and platinum sponge interact with selenium with incandescence when gently heated.[3–7] The particle size of cadmium and selenium must be below a critical size to prevent explosions when making cadmium selenide; this also applies to zinc.[8]

Nitrogen Trichloride. Selenium initiates the violent and often explosive decomposition of the trichloride.[9]

Oxidants. Selenium may react explosively with BrF_5[10] or ClF_3,[11] and it ignites on contact with F_2.[12]

Oxygen and Organic Matter. Heating selenium in oxygen in the presence of traces of organic impurities may result in a vigorous explosion.[13]

Phosphorus. A warm mixture incandesces.[14]

Physiological Properties and Health Hazards

Hydrogen selenide irritates the nose, eyes, and lung tissue, and disturbs the digestive and nervous systems. Solutions of selenium compounds may burn the skin and cause severe pain by skin absorption. Selenium dioxide dust irritates the respiratory system, eyes, and skin, and is particularly harmful by skin absorption. Swallowing will cause severe irritation and poisoning. Prolonged exposure to selenium dust may cause dermatitis, fatigue, digestive upset, and bronchitis. Avoid breathing dust. Avoid contact with skin and eyes.[1] TLV-TWA 0.2 mg/m^3.[15]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Selenium powder may be mixed with sand and treated as normal refuse, as may the disulfide. Soluble selenites and selenates can be dissolved in water and run to waste, diluting with at least 50 times its volume of running water. Soda ash should be applied liberally to spills of selenium dioxide, selenic and selenous acids, and selenyl and selenium chlorides, which may then be mopped up cautiously with plenty of water; wash down the drain, diluting greatly with at least 50 times its volume of water.[16]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal in a secure landfill.

Small Quantities. Wear nitrile rubber gloves, eye protection, laboratory coat, and respirator (or work in hood). The selenium salt is dissolved in water and the solution is neutralized by the addition of 1 M NaOH (prepared by dissolving 4 g of NaOH in 100 mL of water) or 1 M sulfuric acid (prepared by cautiously adding 5 mL of concentrated acid to 85 mL of cold water). A 1 M solution of sodium sulfide (prepared by dissolving 7.8 g of Na_2S in 100 mL of water) is added to the selenium salt solution and the pH is again adjusted to neutral with 1 M sulfuric acid solution. The precipitate is separated by filtration or decantation. Wash, dry, and recycle or send for disposal in a secure landfill.[17]

Reactions for Spillage and Waste Disposal

$$Se^{2+} + Na_2S \rightarrow SeS + 2Na^+$$

$$Se^{4+} + 2Na_2S \rightarrow \underset{\substack{\text{selenium}\\ \text{sulfide}\\ \text{(insoluble)}}}{SeS_2} + 4Na^+$$

REFERENCES

1. Lux 567; see also Merck 8503–8515.
2. Mellor, Vol. 2, Suppl. 1, 583, 1956.
3. Mellor, Vol. 15, 151, 1942.
4. Mellor, Vol. 10, 766, 1947.
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6. Mellor, Vol. 4, 480, 1940.
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8. Reisman, A. et al., *J. Phys. Chem.*, 67, 22, 1963.
9. Mellor, Vol. 8, 601, 1940; Vol. 8, Suppl. 2.2, 412, 1967.
10. B 93.
11. B944.
12. B 1103.
13. Astin, S. et al., *J. Chem. Soc.*, 391, 1933.
14. B 1457.
15. ACGIH 51.
16. *RSC Chemical Safety Data Sheet*, Vol. 4b, Nos. 128–135, Royal Societry of Chemistry, Cambridge, 1991.
17. PPL 167.

SILANE SiH_4

PYROPHORIC WHEN PURE

Synonym

Silcon tetrahydride.

Physical Properties

Gas; repulsive odor; mp, –185°C; bp, –112°C.[1]

Fire Hazard

Ignites in air by raising temperature.

Chemical Properties

Stable at room temperature but decomposes at approximately 400°C. Slowly decomposes in water; practically insoluble in alcohol, ether, benzene, chloroform, silicochloroform, and silicon tetrachloride. Decomposes in potassium hydroxide solutions.[1]

Hazardous Reactions

Halogens. Burns or explodes violently on contact with bromine or chlorine or covalent chlorides.[3]

Oxygen. Very pure silane does not immediately explode with oxygen, but the decomposition products may ignite after a delay.[4]

Physiological Properties and Health Hazards

Irritating to respiratory tract.[1] TLV-TWA 5 ppm (6.6 mg/m^3).[5]

Waste Disposal

All piping and equipment for use with silane must be leak-proof. Leaks will be indicated by flames. Excess may be disposed of by controlled burning. Alternatively, seal cylinders and return to suppliers.[2]

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SILVER AZIDE AgN_3

EXPLOSIVE

Physical Properties

White crystals; mp, 252°C; bp, 297°C.[1]

Chemical Properties

While pure silver azide explodes at 340°C,[2] the presence of impurities may cause explosion at 270°C. It is also impact sensitive and explosions are usually violent.[3] Its use as a detonator has been proposed. [4] Soluble in dilute nitric acid; slightly soluble in ammonium hydroxide.[1]

Hazardous Reactions

Halogens. Contact with ethereal I_2 or nitrogen-diluted Br_2 vapor causes explosions.[5]

Photosensitizing Dyes. Many dyes cause explosions during thermolysis of dye-sensitized silver azide.[4]

Spillage Disposal

Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Using a plastic shovel, scoop into a pail of water in the fume hood (about 66 mL/g). Cautiously add aqueous 5.5% ceric ammonium nitrate (4 volumes per volume of aqueous solution) and stir for an hour. If the solution remains orange, an excess of ceric ammonium nitrate is present and the azide has been completely destroyed. The solution can be washed into the drain with at least 50 times its volume of water.[6,7] The solid residue is treated as normal refuse. A spot test for checking if azide is completely destroyed is as follows: Place a drop of the test solution in the depression of a spot plate and treat with 1 or 2 drops of dilute hydrochloric acid. Add a drop of ferric chloride solution and gently heat the spot plate. A red color indicates hydrazoic acid and incomplete decomposition.[8]

Carefully decontaminate the spill site with a 10% ceric ammonium nitrate solution. Sponge up with water. Wash into the drain with water.

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, behind a shield, cautiously add silver azide to a large excess of cold ceric ammonium nitrate solution (about 66 mL/g azide) with agitation sufficient to provide suspension of all solids. Cool the reaction. When reaction is complete (see spillage disposal for test for completeness of reaction), wash solution into the drain with water.[7] Large amounts of silver salts may be worth recovering.

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SILVER PERCHLORATE $AgClO_4$

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIAL

Physical Properties

Deliquescent crystals; decomposes at 486°C.[1]

Chemical Properties

Freely soluble in water.[1]

Hazardous Reactions

Aromatic Compounds. Forms solid complexes with aniline, pyridine, toluene, benzene, and many other aromatic hydrocarbons; benzene complex explodes violently on crushing in a mortar.[2]

Ethanol. Ethanol complex explodes on crushing.[3]

Physiological Properties and Health Hazards

Irritates the skin and mucous membranes and would be harmful if swallowed. Avoid contact with skin, eyes, and clothing.[4] TLV-TWA 0.01 mg/m^3.[5]

Spillage Disposal

Wear face shield, and goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[6] Dampen thoroughly with water. Using a plastic scoop, cautiously transfer to a large pail of water. In the fume hood, slowly and carefully neutralize with aqueous sodium hydroxide until reaction ceases. Let stand until solids settle. Decant liquid into the drain. Solid residue may be discarded with normal refuse. Large amounts of silver salts may be worth recovering.[4,6,7] Spill site should be washed thoroughly to remove all oxidant.[4]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Carefully add solid to a large quantity of water in a beaker (50–100 mL/g). Cautiously neutralize with aqueous sodium hydroxide until reaction ceases. Wash the solution into the drain.[7] For large quantities, recycling silver may be worthwhile.[4,7]

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SODIUM and SODIUM AMALGAM Na, Na(Hg)

CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GASES, CAUSES BURNS

Physical Properties

Sodium. Soft, silvery white sticks, pellets, wire, or granules; normally coated with a gray oxide or hydroxide skin.[1]

Sodium Amalgam. Silvery or gray spongy masses.[2]

Fire Hazard

Ignites spontaneously in air or oxygen; releases hydrogen on contact with water. Use sodium carbonate or dry chemical to extinguish. Fire can also be smothered with dry sand.[3]

Chemical Properties

Sodium. Reacts vigorously with water with formation of sodium hydroxide and hydrogen gas, which may ignite.[1]

Sodium Amalgam. Reacts with water forming sodium hydroxide and hydrogen gas.[1]

Preparation of Clean Sodium. The cleaning of sodium by cutting off the oxidized surfaces with a knife under xylene is tedious and wasteful. The following technique facilitates the operation, saves metal, and minimizes scrap disposal. Considerably more sodium than will be needed is placed in a wide-mouth Erlenmeyer flask or tall beaker and covered with dry xylene. The flask is heated until the sodium melts but stays in the cage of surface oxide. The heating source is then removed and the flask swirled gently to cause the sodium to flow out of the shells and form several globules. The flask is next cooled without much agitation so the sodium globules do not unite. As a precaution, dry nitrogen can be passed through the solution during cooling. When the metal has solidified, the clear globules are removed with a pointed iron rod or other device, dried rapidly with filter paper or a dry towel, and added to the weighed container used to measure the amount of sodium needed. The oxide shells contain very little sodium; the solvent is decanted, and treatment of the residue with *tert*-butyl alcohol destroys traces of active metal.[4]

Hazardous Reactions

Acids. Anhydrous hydrochloric acid, hydrogen fluoride, or sulfuric acid react slowly with sodium while aqueous solutions react explosively.[5]

Air. Dispersions of sodium in volatile solvents become pyrophoric if the solvent evaporates round the neck of a flask; serum cap closures are safer.[6]

Chloroform and Methanol. Addition of sodium to an inadequately cooled chloroform–methanol solution caused a violent explosion.[7]

Diethyl Ether. While sodium wire was being pressed into ether, the jet blocked. Increasing the pressure to free it caused ignition of the ejected sodium and explosion of the flask of ether. Pressing the sodium into xylene or toluene and subsequent transfer to the ether is recommended.[6]

N,N-Dimethylformamide. Vigorous reaction occurs on heating sodium in this solvent.[4]

Ethanol. Air must be excluded when reacting finely divided sodium with ethanol to avoid hydrogen–air mixture explosions.[8]

Halocarbons. Sodium in carbon tetrachloride,[9–11] chloroform, dichloromethane and chloromethane,[9,10] and tetrachloroethane[9,10] forms shock- and impact-sensitive mixtures. Any aliphatic halocarbon (except fully fluorinated alkanes) may be expected to behave in this way.

Halogens or Interhalogens. Ignites in fluorine gas[5,12] and in moist chlorine[5,12,13]; mixtures with iodine, liquid bromine,[8] iodobromine, or iodochlorine are shock sensitive.[5,12,14]

Hydrazine. Mixture with anhydrous hydrazine forms sodium hydrazide, which explodes on contact with air. Mixture with hydrazine hydrate causes strongly exothermic reaction to give H_2 and ammonia.[15]

Mercury. Reaction is violently exothermic.[16]

Metal Halides. Dispersions of sodium reduce many metal halides to finely powdered metals, many of which are pyrophoric.[17] Mixtures of sodium with metal halides are sensitive to mechanical shock.[9,18]

Metal Oxides. Finely dispersed sodium reduces metal oxides on heating at temperatures between 100°C and 300°C, producing pyrophoric metal powders.[19]

Nonmetal Halides. Vigorous or explosive reactions occur with phosphorus tribromide on addition of drops of water,[20] phosphorus trichloride, phosphorus pentachloride,[21] diselenium dichloride,[22] sulfur dichloride, boron tribromide, sulfur dibromide,[17] seleninyl bromide,[23] phosphoryl chloride, sulfinyl fluoride, silicon tetrachloride, and silicon tetrafluoride.[24]

Nonmetal Oxides. Mixtures with solid CO_2 are impact sensitive and explode violently.[25,26] CO_2 is unsuitable as an extinguishant for the burning metal alone, as the intensity of combustion is increased by replacing air with CO_2. Finely divided silica will often react with burning sodium, so it is not suitable as an extinguishant.[25,26]

Nonmetals. Explosions occur when carbon powder is in contact with sodium in the presence of air[27]; violent reactions occur with sulfur,[28] selenium,[29] and molten tellurium.[28]

Oxygenated Compounds. Mixtures of sodium with inorganic oxygenated compounds or oxygen-rich organic compounds are shock-sensitive explosives.[30]

Sulfides. Mixtures of sodium with carbon disulfide are shock-sensitive explosives.[30]

Water. Vigorous reaction with small pieces of Na; explosion with large lumps[13]; and violent explosion with ice.[31]

Physiological Properties and Health Hazards

Sodium reacts with skin moisture to yield sodium hydroxide, thus causing heat and caustic burns to skin and eyes. Prevent contact with skin and eyes.[2]

Spillage Disposal

Sodium Metal. Instruct others to maintain a safe distance. Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover sodium with dry sodium carbonate or calcium carbonate, shovel into dry bucket, and transport to fume hood. Behind a shield, add a little at a time to a large excess of dry butanol. Leave to stand for 24 hours and run to waste, diluting greatly with at least 50 times its volume of water.[32]

Sodium Amalgam. Cover with a large volume of water in a suitable vessel and allow to stand until there is no further reaction. The mercury may then be separated and recovered, and the sodium hydroxide solution washed into the drain.[1]

Waste Disposal

Handling techniques and safety precautions for large-scale operations have been detailed for this reactive metal.[33]

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Place small pieces of solid sodium in a three-necked, round-bottom flask equipped with a stirrer, dropping funnel, condenser, and heating mantle. Flush the flask with nitrogen. Add 95% ethanol (13 mL per gram of sodium) dropwise at a rate to cause rapid reflux. Stirring is started as soon as enough ethanol has been added to make it feasible. The mixture is stirred and heated under reflux until the sodium is dissolved. Heat is turned off and an equal volume of water is added at a rate that causes no more than mild refluxing. The solution is cooled, neutralized with 6 N sulfuric (prepared by cautiously adding 15 mL of concentrated acid to 75 mL of cold water) or hydrochloric acid (prepared by adding concentrated acid to an equal volume of cold water), and washed into the drain with at least 50 times its volume of water.[34]

Reactions for Spillage and Waste Disposal

$$2Na + 2CH_3CH_2OH \rightarrow \underset{\text{sodium ethoxide}}{2CH_3CH_2ONa} + H_2$$

$$CH_3CH_2ONa + H_2O \rightarrow NaOH + \underset{\text{ethanol}}{CH_3CH_2OH}$$

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SODIUM AMIDE $NaNH_2$

HARMFUL BY INHALATION, CAUSES BURNS

Synonym

Sodamide.

Physical Properties

White to olive green solid; mp, 210°C.[1]

Chemical Properties

Reacts violently with water, with the formation of sodium hydroxide and ammonia.[1]

Hazardous Reactions

Air. May ignite or explode on heating or grinding in air.[2–4]

Halocarbons. Interaction is explosively violent.[5]

Potassium Chlorate. Mixture explodes.[6]

Sodium Nitrite. Addition to molten amide results in violent explosion.[2]

Water. Fresh material behaves like sodium on contact with water.[5,7]

Safe Handling Procedure

Commercial sodium amide stored in a once-opened bottle absorbs moisture, decomposes, and may turn yellow and become potentially explosive; such material should be destroyed by reaction with solid ammonium chloride. Because of this instability, most procedures for use of the reagent include directions for its preparation. However, Fisher supplies material in two small sizes that come in amber bottles with wax-sealed screw caps encased in plastic-wrapped steel cans. These include a six-pack of 4-g bottles (0.1 mol) and a six-pack of 20-g bottles (0.5 mol).[8]

> ***CAUTION:*** Sodium amide is a very reactive substance; it combines with oxygen and reacts explosively with water. It is recommended that the amide be kept in sealed glass containers in an atmosphere of ammonia, or under petroleum solvents for greater convenience in handling. Specimens have been kept under this solvent for 3 years without appreciable loss in activity.

When exposed to the atmosphere, sodium amide rapidly takes up moisture and carbon dioxide. When exposed to only limited amounts, as in imperfectly sealed containers, products are formed that render the resulting mixture highly explosive. The formation of oxidation products is accompanied by the development of a yellow or brownish color. If such a change is noticed, the substance should be destroyed at once. This is conveniently accomplished by covering with a large amount of benzene toluene or kerosene and slowly adding dilute ethanol while stirring.[9]

Physiological Properties and Health Hazards

Dust severely irritates the mouth and nose, and burns the eyes. The solid in contact with moisture on the skin causes heat and caustic burns. Swallowing would cause severe internal irritation and damage. Avoid breathing dust. Prevent contact with eyes and skin.[10]

Spillage Disposal

Wear face shield, goggles, nitrile rubber gloves, and a laboratory coat. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand; shovel into an enamel or polythene bucket; and transport to fume hood. Add, a little at a time while stirring, to a large volume of 95% ethanol. After reaction is complete, run solution to waste, diluting greatly with at least 50 times its volume of running water. Dispose of solid residue as normal refuse. Wash spill site thoroughly with water.[9,11]

Waste Disposal

See safe handling procedure.

Package Lots. Place in a separate labeled container for recycling or disposal. Incinerate under controlled conditions.[12,13]

Small Quantities. Wear eye protection, nitrile rubber gloves, and laboratory coat. Work in the fume hood. Cover the sodium amide with toluene or kerosene and add 95% ethanol slowly and while stirring. When reaction is complete, place the solution in a separate labeled container for disposal by burning.[7]

Alternatively, small portions of sodium amide can be stirred into excess solid ammonium chloride in the fume hood. The resulting mixture is slowly added to a pail of cold water and the solution washed.[13,14]

Reactions for Spillage and Waste Disposal

$$1)\ NaNH_2 + CH_3CH_2OH \rightarrow CH_3CH_2O^-Na^+ + NH_3$$

$$2)\ NaNH_2 + NH_4Cl \rightarrow NaCl + 2NH_3$$

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SODIUM AZIDE NaN_3

CONTACT WITH ACIDS LIBERATES A TOXIC GAS, EXPLOSIVE

Physical Properties

Crystals decompose on heating, sometimes explosively, especially if heated rapidly.[1–3]

Chemical Properties

Soluble in water.[1] Insensitive to impact.[2]

Hazardous Reactions

Bromine. Liable to explode in presence of Br_2 vapor.[4]

Carbon Disulfide. Aqueous solution of NaN_3 forms explosive mixture with CS_2.[5]

Chromyl Chloride. Interaction of chromyl chloride and sodium azide to form chromyl azide is explosive in the absence of diluent.[6]

Heavy Metals. Contact of aqueous solutions with heavy metals (brass, Cu, and Pb) may lead to the formation of explosive heavy metal azides (e.g., in plumbing lines).[7]

Hydrochloric Acid. A serious explosion occurred when prednisone was treated with sodium azide and concentrated hydrochloric acid.[8]

Sulfuric Acid. Forms explosive solution in 50% H_2SO_4 unless acid is quite cold.[9]

Water. Violent reaction occurs when water is added to strongly heated azide.[10]

Physiological Properties and Health Hazards

Dust irritates the eyes. Solution irritates the skin and the eyes. Swallowing assumed to cause irritation and poisoning. Prevent contact with skin and eyes. Avoid breathing dust.[11] TLV-STEL-C 0.29 mg/m^3.[12]

Spillage Disposal

Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[13] Using a plastic scoop, shovel mixture into a pail of water in the fume hood (about 100 mL/g). Cautiously add aqueous 5.5% ceric ammonium nitrate (at least 4 times the volume of the azide solution) and stir for an hour. If the solution remains the orange color of ceric ammonium nitrate solution, an excess is present and the azide has been completely destroyed. Decant the solution to the drain with at least 50 times its volume of water. Treat solid residue as normal refuse.[13,14] A spot test for checking if azide is completely destroyed is as follows: A drop of the test solution is placed in the depression of a spot plate and treated with 1 or 2 drops of dilute hydrochloric acid. A drop of ferric chloride solution is added and the spot plate gently heated. A red color indicates hydrazoic acid and incomplete decomposition.[15] Carefully decontaminate the spill site with a 10% ceric ammonium nitrate solution. Sponge up with water and wash into the drain.

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, behind a shield, cautiously and slowly add the azide to a greater-than-stoichiometric amount of cold 5.5% ceric ammonium nitrate solution (153 mL/g azide) with agitation sufficient to provide suspension of all solids. Cool the reaction. When the reaction is complete (see spillage disposal for test for completeness of reaction), wash the reaction mixture into the drain.[13,14]

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SODIUM BOROHYDRIDE $NaBH_4$

IRRITANT

Physical Properties

White to pale gray hygroscopic crystals; dihydrate melts 36–37°C.[1,2]

Chemical Properties

Soluble in water (55% at 25°C)[1]; decomposes in water with evolution of hydrogen; decomposes rapidly in boiling water.[1,2]

Hazardous Reactions

Acids. Interaction with anhydrous acids (fluorophosphoric, phosphoric, or sulfuric) is very exothermic.[3]

Alkali. Alkaline solution may spontaneously heat and decompose while liberating hydrogen.[4]

Aluminum Chloride. Violent explosion resulted from mixing a 4% solution of $NaBH_4$ in diglyme with a 27% solution of $AlCl_3$ also in diglyme.[5,6]

Physiological Properties and Health Hazards

Dust irritates respiratory system. Irritating to skin and mucous membranes. Avoid breathing dust. Avoid contact with skin, eyes, and clothing.[2]

Spillage Disposal

Wear eye protection, nitrile rubber gloves, laboratory coat, and, if necessary, a self-contained breathing apparatus. Mop up with plenty of water and run to waste, diluting greatly with at least 50 times its volume of water.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, cautiously add small portions of sodium borohydride to a large volume of water such that the concentration of borohydride is less than 3%. Under a nitrogen atmosphere, add dilute acetic acid dropwise while stirring. When neutralization is complete, wash down drain with at least 50 times its volume of water.[7]

Reactions for Spillage and Waste Disposal

$$NaBH_4 + 4H_2O \xrightarrow{H_3O^+} NaOH + B(OH)_3 + 4H_2$$

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SODIUM CHLORATE $NaClO_3$

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIAL

Physical Properties

Colorless crystals or white granules; mp, 248°C.[1]

Chemical Properties

Freely soluble in water; slightly soluble in cold alcohol; soluble in boiling alcohol and glycerol.[1]

Fire Hazard

Mixtures of sodium chlorate and combustible materials are readily ignited; mixtures with finely divided combustible materials can react explosively. Extinguish fire with water spray.[2]

Hazardous Reactions

Alkenes and Potassium Osmate. Sodium chlorate should not be used in the hydroxylation of alkenes to diols in the presence of potassium osmate if the diol is to be distilled from the reaction mixture; explosive oxidation may occur.[3]

Ammonium Salts, Metals, or Nonmetals, or Sulfides. Mixtures with ammonium salts, powdered metals, phosphorus, silicon, sulfur, or sulfides are readily ignited and potentially explosive.[4]

Nitrobenzene. Mixture is powerfully explosive.[5]

Organic Matter. Mixtures with fibrous or absorbent organic materials (charcoal, flour, shellac, sawdust, and sugar) are hazardous and can be caused to explode by static friction or shock.[6]

Paper and Static Electricity. Paper impregnated with the chlorate can be ignited by static sparks.[7]

Phosphorus. Mixture explodes violently.[8]

Sulfuric Acid. Addition of concentrated H_2SO_4 causes explosion of ClO_2 generated.[9]

Wood. Wood in contact with the chlorate over a period of time may ignite or explode.[9]

Physiological Properties and Health Hazards

Dust irritates the eyes and skin. Swallowing solution or solid results in nausea, vomiting, abdominal pain, and possible kidney damage. Avoid contact with combustible materials and acids. Avoid contact with skin, eyes, and clothing.[10]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Scoop the chlorate into a container and transport to the fume hood. Add to sufficient water to give an approximately 10% solution. Follow the waste disposal procedure. Wash the spill site thoroughly with soap and water.[10]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Prepare a 10% aqueous solution of the waste sodium chlorate. For each 10 mL of solution, slowly, and while stirring, add 44 mL of a 10% solution of sodium bisulfite (this allows about 50% excess of reducing agent). The continued presence of chlorate can be detected by adding, to 3 mL of the solution, a freshly prepared solution of potassium iodide (100 mg) in 3 mL of 3 M sulfuric acid (prepared by cautiously adding 0.5 mL of concentrated acid to 2.5 mL of cold water). An amber to brown color indicates the presence of chlorate. Add sodium bisulfite solution until the test is negative. Neutralize the acidic solution with sodium carbonate and discard into the drain with at least 50 times its volume of water.[11,12]

Reactions for Spillage and Waste Disposal

$$ClO_3^- + 3HSO_3^- \rightarrow Cl^- + 3SO_4^{2-} + 3H^+$$

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SODIUM CHROMATE Na_2CrO_4

HUMAN CARCINOGEN, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

Yellow deliquescent crystals; crystallizes as tetra- or decahydrate. Decahydrate loses water; mp, 792°C (decahydrate mp, 20°C).[1]

Chemical Properties

Soluble in water.[1]

Physiological Properties and Health Hazards

Dust irritates the respiratory tract and eyes. Swallowing causes irritation and internal damage. Prolonged exposure causes ulceration, damage to liver and kidneys, and even cancer. Prevent inhalation of dust. Prevent contact with eyes and skin.[2] TLV-TWA 0.05 mg/m^3.[3]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a pail of water. Carefully acidify this solution with 3 M sulfuric acid (prepared by cautiously adding 10 mL of concentrated acid to 50 mL of cold water) until pH of 1 (to pHydrion paper) is obtained. Slowly, and while stirring, add solid sodium thiosulfate until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Filter immediately through Celite or let it stand for a week, when much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or is filtered off. The liquid may be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, dried, packaged, labeled, and sent to a secure landfill site.[4] The spill site and contaminated clothing should be washed thoroughly with soap and water to remove the oxidant.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add the chromate to a beaker of water (about 20 mL/g). Acidify with 3 M sulfuric acid (55 mL/100 mL of chromate solution). Slowly add sodium thiosulfate (about 13 g/5 g chromate). The solution will change in color from orange to olive green to blue green (cloudy). When the thiosulfate has all dissolved, neutralize with sodium carbonate (about 20 g). Filter immediately through Celite or let it stand for 1 week, when much of the supernatant can be decanted. In the latter case, the remaining liquid is allowed to evaporate or the solid filtered using Whatman No. 5 filter paper. The supernatant contains less than 0.5 ppm of chromium and can be washed into the drain. The solid residue is washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to secure landfill site.[4]

Reactions for Spillage and Waste Disposal

$$2Na_2CrO_4 + 3Na_2S_2O_3 + 2H_2O \rightarrow \underset{\substack{\text{chromium}\\\text{hydroxide}\\\text{(insoluble)}}}{2Cr(OH)_3} + 3Na_2SO_4 + 3S + 4NaOH$$

REFERENCES

1. Merck 8674.
2. Lux 304.
3. ACGIH 23.
4. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

SODIUM CYANIDE NaCN

VIOLENT POISON

Physical Properties

White granules or fused pieces. Violent poison! Odorless when perfectly dry; somewhat deliquescent in damp air and emits slight odor of HCN; mp, 563°C.[1]

Fire Hazard

Contact with acids liberates highly flammable hydrogen cyanide. Extinguish fire with dry chemical. Do not use carbon dioxide.[2]

Chemical Properties

Freely soluble in water; slightly soluble in alcohol. The aqueous solution is strongly alkaline; the solution readily dissolves gold and silver in presence of air.[1]

Hazardous Reactions

Oxidants. Oxidized violently or explosively in contact with hot oxidants.[3]

Physiological Properties and Health Hazards

Swallowing or absorption of solution through skin results in severe poisoning. The early symptoms are general weakness and heaviness of the arms and legs, increased difficulty in breathing, headache, dizziness, nausea, and vomiting, and these may be rapidly followed by pallor, unconsciousness, cessation of breathing, and death. Prevent contact with skin and eyes.[4] TLV-TWA 5 mg/m^3 (skin).[5]

Spillage Disposal

Wear breathing apparatus, eye protection, nitrile rubber gloves, and laboratory coat. Instruct others to keep a safe distance.

Sodium Cyanide Solutions. Cover the cyanide solution with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop this mixture into a container in the fume hood. Slowly add this slurry to a large pail containing household bleach (about 70 mL/g of cyanide). Proceed as per waste disposal procedure.[4,6]

Solid Sodium Cyanide. Sweep the sodium cyanide into a container and transport to the fume hood. Add to a solution of 1% sodium hydroxide (about 50 mL/g of cyanide). Slowly add the cyanide solution to household bleach as per waste disposal procedure. Wash spill site thoroughly with soap and water.[4,6]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal.

Small Quantities or Solutions. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add the sodium cyanide to a solution of 1% sodium hydroxide (about 50 mL/g of cyanide). Household bleach (about 70 mL/g of cyanide) is slowly added to the basic cyanide solution while stirring. When addition of the bleach is complete, the solution can be tested for the presence of cyanide using the Prussian blue test: To 1 mL of the solution to be tested, add 2 drops of a freshly prepared 5% aqueous ferrous sulfate solution. Boil this mixture for at least 60 seconds, cool to room temperature, and then add 2 drops of 1% ferric chloride solution. The resulting mixture is made acid to litmus with 6 M hydrochloric acid (prepared by adding

concentrated acid to an equal volume of cold water). If cyanide is present, a deep blue precipitate will form. (Concentrations of cyanide greater than 1 ppm can be detected.) If the test is positive, add more bleach to the cyanide solution, and repeat the test. Continue until no Prussian blue precipitate is formed. Wash the solution into the drain.[4,6]

Reactions for Spillage and Waste Disposal

$$CN^- + ClO^- \rightarrow \underset{\text{cyanate}}{CNO^-} + Cl^-$$

REFERENCES

1. Merck 8679.
2. NFPA 49.
3. B 1661.
4. Lux 119, 310.
5. ACGIH 36.
6. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 285.

SODIUM DICHROMATE $Na_2Cr_2O_7$

HUMAN CARCINOGEN, CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

Dihydrate. Reddish to bright orange, somewhat deliquescent crystals; loses water on prolonged heating at about 100°C.

Anhydrous. mp, 356.7°C.[1]

Chemical Properties

Very soluble in water; oxidizing agent.[1]

Hazardous Reactions

Acetic Anhydride. Addition of the dehydrated salt to the anhydride may cause a reaction that accelerates to explosion.[2]

Boron. Mixtures in the presence or absence of silicon may ignite.[3]

Ethanol and Sulfuric Acid. Oxidation of ethanol with acid dichromate may result in minor explosion.[4]

Hydroxylamine. Violent reaction between sodium dichromate dihydrate and aqueous hydroxylamine may accelerate to explosion. Anhydrous hydroxylamine explodes violently on contact with sodium dichromate.[5]

Organic Residues, Oxidizable Substances, and Sulfuric Acid. Vessels containing tarry residues or traces of oxidizable substances should not be placed in dichromate and sulfuric acid cleaning solutions; violent reactions may result.[4]

Trinitrotoluene. In the oxidation of trinitrotoluene in sulfuric acid to trinitrobenzene, the reaction mixture must be stirred vigorously to prevent the addition of a portion of sodium dichromate that will cause local ignition.[6]

Physiological Properties and Health Hazards

Dust irritates the respiratory tract and eyes. Swallowing causes irritation and internal damage. Prolonged exposure to dust causes skin ulceration, damage to liver and kidneys, and even cancer. Prevent inhaling dust. Prevent contact with eyes and skin.[7] TLV-TWA 0.05 mg/m^3.[8]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop mixture into a container of water in the fume hood (about 100 mL/5 g dichromate). Carefully acidify the solution with 3 M sulfuric acid (prepared by cautiously adding 9 mL of concentrated acid to 46 mL of cold water) until a pH of 1 (to pHydrion paper) is obtained. Slowly, and while stirring, add solid sodium thiosulfate until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Filter immediately through Celite or let it stand for a week, when much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or filtered off. The liquid can be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, dried, packaged, labeled, and sent to a secure landfill site.[9,10] The spill site and contaminated clothing should be washed thoroughly with soap and water to remove the oxidant.[7]

Waste Disposal

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add the solid dichromate to a container of water (about 100 mL/5 g). Acidify with 3 M sulfuric acid (about 40–60 mL). Slowly, and while stirring, add solid sodium thiosulfate (about 15 g) until the solution becomes cloudy and blue colored. Neutralize the solution with sodium carbonate. After a few minutes, a blue-gray flocculent precipitate is formed. Filter immediately through Celite or let it stand for a week, when much of the supernatant can be decanted. The remaining liquid is allowed to evaporate or is filtered off. The liquid can be washed into the drain. The solid residue should be washed with hot water to remove sodium sulfate, and then dried, packaged, labeled, and sent to a secure landfill site.[9,10]

Reactions for Spillage and Waste Disposal

$$Cr_2O_7^{2-} + 3S_2O_3^{2-} + 2H_3O^+ \rightarrow \underset{\substack{\text{chromium}\\ \text{hydroxide (insoluble)}}}{2Cr(OH)_3} + 3SO_4^{2-} + 3S$$

REFERENCES

1. Merck 8683.
2. B 1076.
3. B 56.
4. B 1077.
5. Mellor, Vol. 3, 670, 1941; Vol. 8, 287, 1940.
6. Clarke, H.T. et al., *Org. Synth.*, Coll. Vol. 1, 543, 1941.
7. Lux 304.
8. ACGIH 23.
9. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
10. ITI 585.

SODIUM ETHOXIDE $NaOCH_2CH_3$
and SODIUM METHOXIDE $NaOCH_3$

HIGHLY FLAMMABLE, CAUSE BURNS

Synonyms

Sodium ethylate; Sodium methylate.

Although these compounds are sometimes used industrially as solids, they are more frequently prepared in the laboratory by reacting sodium metal with ethanol and methanol. The hazards of these solutions are dealt with here.

Physical Properties

Sodium Ethoxide. White or yellowish hygroscopic powder.[1]

Sodium Methoxide. White, free-flowing powder.[2]

Chemical Properties

Sodium Ethoxide. Decomposed by water to sodium hydroxide and ethanol. Soluble in ethanol.[1]

Sodium Methoxide. Decomposed by water to sodium hydroxide and methanol. Soluble in methanol.[2]

Hazardous Reactions

Chloroform. Mixture of solid sodium methoxide, methanol, and chloroform explodes.[3]

Physiological Properties and Health Hazards

Solutions burn skin and eyes. Swallowing causes severe internal irritation and damage. Prevent contact with skin and eyes.[4]

Spillage Disposal

Shut off all possible sources of ignition. Wear face shield, goggles, laboratory coat, and butyl rubber gloves. Cover spill of alkoxide solution with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to a pail of water in the fume hood. Neutralize the solution with 6 M hydrochloric acid (prepared by cautiously adding concentrated acid to an equal volume of cold water). Decant liquid to drain with at least 50 times its volume of water. Solid residue may be discarded with normal refuse. Ventilate area of spillage well to evaporate remaining liquid and dispel vapor.[4,5]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[6]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. Slowly add sodium alkoxide to a pail of water. Neutralize with 6 M hydrochloric acid (prepared by cautiously adding a volume of concentrated acid to an equal volume of cold water), and then wash into the drain.[5]

Reactions for Spillage and Waste Disposal

$$NaOCH_2CH_3 + HCl \rightarrow \underset{\text{chloride}}{NaCl} + \underset{\text{ethanol}}{CH_3CH_2OH}$$
sodium

$$NaOCH_3 + HCl \rightarrow \underset{\text{chloride}}{NaCl} + \underset{\text{methanol}}{CH_3OH}$$
sodium

REFERENCES

1. Merck 8687.
2. Merck 8594.
3. Kaufmann, W.E. et al., *Org. Synth.*, Coll. Vol. 1, 258, 1944.
4. Lux 578.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.6.
6. Ald 3140A, 3146D.

SODIUM FLUORIDE NaF

POISONOUS

Physical Properties

Clear, lustrous crystals of cubic tetragonal form or white powder; mp, 993°C.[1]

Chemical Properties

Soluble in water; insoluble in alcohol. Aqueous solutions have alkaline reaction and etch glass.[1]

Physiological Properties and Health Hazards

Dust irritates tissues of the respiratory tract, eyes, and skin. Highly toxic. Swallowing results in nausea, stomach pains, vomiting, and diarrhea. Prolonged exposure causes shortness of breath, cough, and cyanosis.[2] TLV-STEL-C (as F) 3 ppm.[3]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, face shield, and goggles. Sweep sodium fluoride into a large beaker. In the fume hood, add water (about 18 mL/g of fluoride) and solid calcium hydroxide (about 0.9 g/g of fluoride) to the beaker and stir for 24 hours. Filter. Wash filtrate into the drain. The solid residue can be discarded with normal refuse.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. In a beaker, add 0.9 g of calcium hydroxide and 18 mL of water for every gram of sodium fluoride. Stir the resulting mixture for 24 hours. Filter. Wash filtrate into the drain. The solid residue can be discarded with normal refuse.[4]

Reactions for Spillage and Waste Disposal

$$2NaF + Ca(OH)_2 \rightarrow \underset{\substack{\text{calcium}\\\text{fluoride}\\\text{(insoluble)}}}{CaF_2} + 2NaOH$$

REFERENCES

1. Merck 8691.
2. Lux 578.
3. ACGIH 36.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988.

SODIUM HYDRIDE NaH

CONTACT WITH WATER LIBERATES HIGHLY FLAMMABLE GAS, TOXIC

Physical Properties

Silvery needles; the commercial product is a gray-white powder; dec, 425°C.[1]

Fire Hazard

Flammable solid; may ignite in moist air. Do not use water, carbon dioxide, dry chemical, or halogenated extinguishers. Extinguish fire by smothering with dry sand or class D extinguisher.[2]

Chemical Properties

Reacts explosively with water, and violently with lower alcohols; ignites spontaneously on standing in moist air.[1]

Hazardous Reactions

Acetylene. Reacts vigorously in the presence of moisture even at –60°C.[3]

Air. The finely divided dry powder ignites in dry air.[4]

Dimethyl Sulfoxide. Mixture explodes if heated above 70°C.[5]

Halogens. Incandesces in chlorine or fluorine at room temperature and in iodine at 100°C.[3]

Sulfur Dioxide. Reacts explosively with sulfur dioxide.[6]

Water. Addition to a small amount of water causes explosion.[7]

Physiological Properties and Health Hazards

Highly toxic and an irritant.[8]

Spillage Disposal

Wear nitrile rubber gloves, fireproof clothing, goggles, and face shield. Eliminate all sources of ignition. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Carefully transfer to a pail and, in the fume hood, slowly and cautiously add butanol (about 10 mol/mol hydride or about 38 mL/g of hydride). When the reaction ceases, add water very carefully until remaining hydride is destroyed. Let stand until solids settle. Decant liquid into the drain. The solid residue may be discarded with normal refuse.[9,10]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile gloves. In the fume hood, mix the hydride with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Slowly and cautiously add butanol (about 38 mL/g of hydride) until the reaction ceases. Then add water very carefully until all of the hydride is destroyed. Let stand until solids settle. Decant liquid into the drain. The solid residue may be discarded with normal refuse.[9,10]

Reactions for Spillage and Waste Disposal

$$NaH + CH_3CH_2CH_2CH_2OH \rightarrow \underset{\text{sodium butoxide}}{NaOCH_2CH_2CH_2CH_3} + H_2$$

$$NaOCH_2CH_2CH_2CH_3 + H_2O \rightarrow NaOH + HOCH_2CH_2CH_2CH_3$$

REFERENCES

1. Merck 8699.
2. NFPA 49.
3. Mellor, Vol. 2, 483, 1941.
4. B 1180.
5. French, F.A., *Chem. Eng. News*, 44, 48, 1966; Olson, G.L., *Chem. Eng. News*, 44, 7, 1966; Russel, G.A. et al., *J. Org. Chem.*, 31, 248, 1966.
6. Moissan, H., *Compt. Rend.*, 135, 647, 1902.
7. Braidech, M.M., *J. Chem. Educ.*, 44, 321, 1967.
8. ITI 589.
9. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
10. PPL 164.

SODIUM HYDROSULFITE $Na_2S_2O_4 \cdot 2H_2O$, also $Na_2S_2O_4$

CONTACT WITH MOIST COMBUSTIBLE MATERIALS MAY CAUSE FIRE

Synonyms

Sodium dithionite, sodium hyposulfite.

Physical Properties

White or grayish white crystalline powder.[1]

Fire Hazard

Fires resulting from contact with combustible materials are best extinguished by means of carbon dioxide, dry chemical, or sand. Avoid water unless flooding amounts are available.[2]

Chemical Properties

Very soluble in water; slightly soluble in alcohol.[1] Addition of small amounts of water to the salt causes hazardous reaction.[3] Oxidizes in air.[1]

Hazardous Reactions

Acids. Contact with acids liberates toxic gas.[3]

Oxidants. Violent decomposition can occur in the presence of oxidants.[4]

Sodium Chlorite. Mixture ignites.[5]

Water. Addition of 10% of water caused heating and spontaneous ignition.[3]

Physiological Properties and Health Hazards

Avoid contact with moist flammable materials including clothing. Harmful if swallowed.[3]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Cautiously add to a very large volume of water in a pail. Slowly add household bleach (containing about 5% sodium hypochlorite) until the reaction is complete (presence of hydrosulfite can be detected by adding silver nitrate solution; a black precipitate indicates hydrosulfite).[8] Let stand until solids settle. Decant the liquid into the drain. The solid residue may be discarded with normal refuse.[6,7] The spill site and contaminated clothing should be washed thoroughly with soap and water to remove all traces of the hydrosulfite.[3]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add to an equal volume of sodium carbonate or calcium carbonate, and then add to a very large volume of water. Cautiously add calcium or sodium hypochlorite solution (household bleach). When reaction is complete (test with silver nitrate for completeness of reaction),[8] dilute and neutralize. Let stand until solids settle. Liquid may be decanted into the drain with at least 50 times its volume of water. Solid residue may be discarded with normal refuse.[6,7]

Reactions for Spillage and Waste Disposal

$$S_2O_4^{2-} + 3OCl^- + 2OH^- \rightarrow 2SO_4^{2-} + 3Cl^- + H_2O$$

REFERENCES

1. Merck 8700.
2. NFPA 49.
3. Lux 577.
4. B 1385.
5. B 987.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
7. ITI 590.
8. Vogel, A.I., *A Textbook of Macro and Semimicro Qualitative Inorganic Analysis,* 4th ed., Longmans, Green and Co., Toronto, 1954, p. 415.

SODIUM HYDROXIDE NaOH

CORROSIVE, CAUSES SEVERE BURNS

Physical Properties

White lumps, sticks, or pellets; mp, 318°C.[1]

Chemical Properties

Very soluble in water; rapidly absorbs carbon dioxide and water from the air.[1]

Hazardous Reactions

Bromine. Addition of bromine to sodium hydroxide solution should be slow with adequate mixing to prevent the formation of two layers; otherwise, a violent eruption may occur.[2]

Chloroform and Methanol. Sodium hydroxide reacts vigorously with a mixture of chloroform and methanol.[3]

Cinnamaldehyde. May form peroxide in contact with solid sodium hydroxide.[4]

Cyanogen Azide. Forms explosive solid (sodium 5-azidotetrazolide) with 10% sodium hydroxide.[5]

Maleic Anhydride. Decomposes exothermally in the presence of low concentrations of sodium hydroxide.[6]

Nitrobenzene. A mixture of nitrobenzene, flake sodium hydroxide, and some water may explode when heated.[7]

Sodium Borohydride. Alkaline sodium borohydride solutions decompose rapidly with the evolution of hydrogen when the pH is below 10.5.[8]

1,2,4,5-Tetrachlorobenzene. The alkaline hydrolysis of tetrachlorobenzene to yield 2,4,5-trichlorophenol may proceed explosively even at atmospheric pressure in ethylene glycol.[9]

2,2,2-Trichloroethanol. Addition of 50% sodium hydroxide solution to trichloroethanol causes an explosion.[10]

Trichloronitromethane. Reacts violently with alcoholic sodium hydroxide.[11]

Water. Very exothermic reaction with limited amounts of water.[12]

Zinc. A mixture of flake sodium hydroxide and zinc dust may ignite.[13]

Zirconium. Heating with sodium hydroxide results in explosion.[14]

Physiological Properties and Health Hazards

Corrosive to all tissues. Inhalation of the dust causes damage to the respiratory tract. Swallowing causes severe internal irritation and injury. Solutions as weak as 2.5 M can cause severe eye damage.[15] TLV-STEL-C 2 mg/m^3.[16]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves.[17] Shovel into a bucket and add, a little at a time and while stirring, to a large volume of ice water. Neutralize with 5% hydrochloric acid and wash into the drain.[17] Wash spill site thoroughly with water.[15]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Add slowly, while stirring, to a large volume of ice water. Neutralize with 5% hydrochloric acid and wash into the drain.[18]

Reactions for Spillage and Waste Disposal

$$NaOH + HCl \rightarrow \underset{\text{sodium chloride}}{NaCl} + H_2O$$

REFERENCES

1. Merck 8701.
2. B 102.
3. B 133.
4. B 780.
5. Marsh, F.D., *J. Org. Chem.*, 37, 2966, 1972.
6. Davie, W.R., *Chem. Eng. News*, 42, 41, 1964.
7. B 603.
8. Anonymous, *Angew. Chem.* (*Nachr.*), 8, 238, 1960.
9. B 562.
10. B 246.
11. B 121.
12. B 1184.
13. B 1472.
14. B 1474.
15. Lux 187, 580.
16. ACGIH 53.
17. LSS.
18. Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.4; Armour, M.A., Ashick, D., and Konrad, J., *Chemical Health and Safety*, 6, 25, 1999.

SODIUM HYPOCHLORITE SOLUTION (Containing over 5% Active Chlorine) $NaClO \cdot 5H_2O$

CAUSES BURNS

Synonyms

Clorox®, Javex, bleach.

Physical Properties

Colorless or yellow solution smelling of chlorine.[1]

Chemical Properties

Oxidant.[2]

Fire Hazard

Anhydrous material is explosive. May cause ignition in contact with organic materials.[3]

Hazardous Reactions

Oxygen is evolved slowly during storage; bottles should have vent caps and material should be stored at 15–18°C and out of direct sunlight.[4]

Amines. Primary aliphatic or aromatic amines form explosive N-mono- or di-chloroamines with sodium hypochlorite.[5]

Ammonium Acetate, Carbonate, Nitrate, Oxalate, and Phosphate. All induce rapid decomposition of sodium hypochlorite.[6]

Cellulose. Reacts violently with sodium hypochlorite.[7]

Ethyleneimine. Forms explosive 1-chloroethyleneimine with sodium hypochlorite.[8]

Methanol. Several explosions involving methanol and sodium hypochlorite were attributed to formation of methyl hypochlorite, especially in presence of acid or other esterification catalyst.[4]

Phenylacetonitrile. Use of sodium hypochlorite to destroy acidified benzyl cyanide residues caused a violent explosion, which was thought to have been due to formation of nitrogen trichloride.[4]

Physiological Properties and Health Hazards

Irritates and burns the eyes and skin. Swallowing causes internal irritation and damage. Avoid contact with skin, eyes, and clothing.[9] Inhalation may produce severe bronchial irritation or pulmonary edema.[1]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Mop with plenty of water and wash into the drain with at least 50 times its volume of water.[9]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add to a pail of cold water. Wash into the drain.[9]

REFERENCES

1. Merck 8702.
2. PPL 170.
3. ITI 592.
4. B 986.
5. B 984.
6. NFPA 491M.
7. Sax 3088.
8. NFPA 491M.
9. Lux 187, 580.

SODIUM NITRATE $NaNO_3$

EXPLOSIVE WHEN MIXED WITH COMBUSTIBLE MATERIALS

Physical Properties

Colorless crystals; deliquescent in moist air; mp, 308°C.[1]

Fire Hazard

Mixtures of sodium nitrate and combustible materials are readily ignited; mixtures with finely divided combustible materials can react explosively. Extinguish fire with flooding amounts of water, using caution (avoid splattering!) if the nitrate has melted.[2]

Chemical Properties

Freely soluble in water.[1]

Hazardous Reactions

Acetic Anhydride. Mixture reacts violently.[3]

Aluminum or Aluminum Oxide. Forms explosive mixtures.[4]

Antimony. Mixture with powdered antimony explodes.[5]

Fibrous Material. May ignite fibrous organic material (e.g., jute, wood, and similar cellulosic material) on heating.[6]

Nonmetals. Mixture with charcoal ignites on heating.[7]

Performic Acid. Contact with the nitrate may lead to explosive decomposition.[8]

Phenol and Trifluoroacetic Acid. Rapid exothermic reaction occurs in mixture.[9]

Sodium. An explosive compound is formed by interaction with Na.[10]

Sodium Thiosulfate or Sodium Phosphinate. Mixture explodes on heating.[11]

Physiological Properties and Health Hazards

Ingestion may cause gastroenteritis, abdominal pains, vomiting, muscular weakness, irregular pulse, convulsions, and collapse; 15–30 g in a single dose may be fatal. Avoid contact with combustible materials.[12]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Shovel into bucket of water and wash the solution into the drain with water. Spill site should be washed thoroughly to remove all oxidant, which is liable to render any organic matter (particularly wood, paper, and textiles) with which it comes into contact dangerously combustible when dry. Clothing wetted with the solution should be washed thoroughly.[12,13]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Dissolve in a large excess of water. Wash into the drain with water.[13]

REFERENCES

1. Merck 8720.
2. NFPA 491M.
3. Davey, W. et al., *Chem. Ind.*, 814, 1948.
4. B 1337.
5. Mellor, Vol. 9, 382, 1939.
6. Mellor, Vol. 2, Suppl. 2.1, 1244, 1961.
7. Mellor, Vol. 2, 820, 1941.
8. D'Ans, J. et al., *Chemische Berichte*, 48, 1139, 1915.
9. Spitzer, U.A. et al., *J. Org. Chem.*, 39, 3936, 1974.
10. Mellor, Vol. 2, Suppl. 2.1, 518, 1961.
11. Mellor, Vol. 2, 820, 1941.
12. Lux 187, 581.
13. PPL 171.

SODIUM NITRITE $NaNO_2$

CONTACT WITH COMBUSTIBLE MATERIAL MAY CAUSE FIRE

Physical Properties

White or slightly yellow granules, rods, or powder; hygroscopic; mp, 271°C.[1]

Fire Hazard

Mixtures of sodium nitrite and combustible materials are readily ignited; mixtures with finely divided combustible materials can react explosively. Extinguish fire with water spray.[2]

Chemical Properties

Soluble in water. Poisonous nitrous fumes produced by the action of acids. Avoid contact with combustible materials or acids.[2]

Physiological Properties and Health Hazards

Toxic if swallowed.[2]

Hazardous Reactions

Amidosulfates. Warming with metal amidosulfates (sulfamates) may become explosively violent.[3]

Aminoguanidine Salts. Sensitive explosive formed in the absence of acid.[4]

Ammonium Salts. Violent explosion occurs on heating a mixture.[4,5]

Metal Cyanides. Mixtures explode on heating.[6,7]

Phenol. Mixture explodes violently on heating.[8]

Phthalic Acid or Phthalic Anhydride. Mixtures explode violently on heating.[8]

Sodium Amide. Addition of the nitrite to molten amide results in violent explosion.[9]

Sodium Thiocyanate. Mixture explodes on heating.[6]

Sodium Thiosulfate. Mixture of solutions does not react violently; solid mixture explodes on heating.[6,10]

Urea. Conditions for fusion with urea must be exactly as described to avoid risk of explosion.[11]

Wood. Wood impregnated with solutions of the nitrite over a long period will burn fiercely if accidentally ignited.[12]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, a face shield, and goggles. Scoop the solid into a container and transport to the fume hood. Slowly add to a beaker of cold water. While stirring, gradually add freshly prepared 10% aqueous sodium bisulfite or metabisulfite solution to the aqueous nitrite solution (about 50 or 75 mL/g nitrite, respectively).[13] Test for the presence of nitrite using starch-iodide paper dipped in dilute acetic acid.[14] When the nitrite is completely destroyed, decant the liquid to the drain. The spill site should be washed thoroughly to remove all oxidant, which is liable to render any organic matter (e.g., wood, paper, and textiles) dangerously combustible when dry. Clothing wetted with the solution should be removed and washed immediately.[2]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, add nitrite to a beaker of water (30–50 mL/g). Slowly and while stirring, add freshly prepared 10% aqueous sodium bisulfite or metabisulfite solution (about 50 or 75 mL/g, respectively) until reaction is complete (5–10 min).[13] Test for presence of nitrite with starch-iodide paper dipped in dilute acetic acid.[14] When reaction is complete, neutralize with sodium carbonate and then wash solution into the drain.[13]

Nitrites in aqueous solution can also be destroyed by adding about 50% excess aqueous ammonia and acidifying to pH 1 (pH paper) with hydrochloric acid.[15]

Reactions for Spillage and Waste Disposal

$$1)\ 2NaNO_2 + 3HSO_3^- + H_2O \rightarrow N_2 + 3HSO_4^- + 2KOH$$

$$2)\ NO_2^- + NH_3 \rightarrow N_2 + 2H_2O$$

REFERENCES

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2. Lux 187, 582.
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6. Mellor, Vol. 8, 478, 1940.
7. Greenwood, P.H.S., *J. Proc. R. Inst. Chem.*, 137, 1947; Elson, C.H.R., *J. Proc. R. Inst. Chem.*, 19, 1947.
8. B 1336.
9. Bergstrom, F.W. et al., *Chem. Rev.*, 12, 64, 1933.
10. Mellor, Vol. 10, 501, 1947.
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12. B 1337.
13. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
14. Vogel, A.I., *A Textbook of Macro and Semimicro Qualitative Inorganic Analysis*, 4th ed., Longmans, Green and Co., Toronto, 1954, p. 338.
15. PPL 171.

SODIUM PERIODATE $NaIO_4$

CONTACT WITH COMBUSTIBLE MATERIALS MAY CAUSE FIRE

Synonyms

Sodium metaperiodate.

Physical Properties

White crystals; dec, 300°C.[1]

Fire Hazard

Mixtures with combustible materials are readily ignited; mixtures with finely divided combustible materials can react explosively. Extinguish fire with water spray.[1]

Chemical Properties

Soluble in water[1]; oxidizing agent.[2]

Physiological Properties and Health Hazards

Toxicity not documented but may be compared to that of potassium periodate, which is an irritant and is toxic by ingestion. Avoid contact with combustible materials. Avoid contact with skin, eyes, and clothing.[1]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Scoop the solid into a container and add to water to give a saturated solution. Follow waste disposal procedure. Spill site should be washed thoroughly to remove all oxidant, which is liable to render any organic matter (particularly wood, paper, and textiles) with which it comes into contact, dangerously combustible when dry. Clothing wetted with the solution should be washed thoroughly.[1]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Prepare a saturated solution of waste sodium periodate in water. About 9.5 g of the salt will dissolve in 100 mL of water at 20°C. In the fume hood, slowly and while stirring, add a 10% aqueous solution of sodium bisulfite until the initially formed black precipitate dissolves and a clear and then a pale yellow solution is formed. Test for the presence of unreduced periodate: To 3 mL of solution, add a freshly prepared solution of potassium iodide (100 mg) in 3 mL of 3 M sulfuric acid (prepared by cautiously adding 0.5 mL of concentrated acid to 2.5 mL of cold water). An amber/brown color indicates the presence of oxidizing agent. Add sodium bisulfite solution until the test is negative. About 20 mL of bisulfite solution will be required for each 10 mL of periodate solution. Neutralize the solution with sodium carbonate and wash into the drain.[3]

Reactions for Spillage and Waste Disposal

$$IO_4^- + 4HSO_3^- \rightarrow I^- + 4SO_4^{2-} + 4H^+$$

REFERENCES

1. Lux 582; Merck 8714.
2. Ald 3150C.
3. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; Armour, M.A., Chemical treatment methods to minimize waste, in *Pollution Prevention and Waste Minimization in Laboratories*, Reinhardt, P.A. et al., Eds., Lewis Publishers, Boca Raton, FL, 1995, p. 284.

SODIUM PEROXIDE Na_2O_2

CONTACT WITH COMBUSTIBLE MATERIALS MAY CAUSE FIRE, CAUSES SEVERE BURNS

Physical Properties

Yellowish-white powder; absorbs water and carbon dioxide from the air.[1]

Fire Hazard

If in contact, or mixed, with organic or other oxidizable substances, ignition or explosion may take place. Use flooding quantities of water or dry chemical extinguisher to fight fire.[2]

Chemical Properties

Reacts violently with water forming sodium hydroxide and oxygen.[3]

Hazardous Reactions

Acetic Acid. Mixture explodes.[4]

Ammonium Peroxydisulfate. Mixture explodes on heating above 75°C, grinding in a mortar, or exposure to drops of water or carbon dioxide.[5]

Calcium Acetylide. Mixture is liable to explode.[6]

Fibrous Materials. Contact with moist cloth, paper, or wood often causes ignition.[4]

Hydrogen Sulfide. Gas ignites immediately on contact with solid peroxide.[7]

Hydroxy Compounds. Mixture with ethanol, glycerol, sugar, or acetic acid usually results in fire or explosion.[8]

Metals and Carbon Dioxide. Mixtures with aluminum, magnesium, or tin powders ignite in moist air and explode in carbon dioxide.[9]

Nonmetal Halides. Violent interaction with diselenium[10] or disulfur dichlorides[10] and phosphorus trichloride.[11]

Nonmetals. Explosive mixtures formed with carbon,[8] phosphorus,[8] antimony, arsenic, boron, sulfur, and selenium.[12]

Organic Liquids and Water. Ignites on simultaneous contact with water and aniline, benzene, diethyl ether, or glycerol.[13]

Water. Reacts vigorously or explosively, depending on relative quantities. Contact of the peroxide with combustible materials and traces of water may cause ignition.[13,14]

Physiological Properties and Health Hazards

Dust irritates the respiratory system and eyes. Causes both thermal and caustic burns on moist skin. Swallowing would cause severe internal irritation and damage. Do not breathe dust. Prevent contact with skin and eyes.[3]

Spillage Disposal

Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Carefully shovel into an enamel or plastic bucket and transport to the fume hood. Slowly and cautiously add to a large volume of water (resulting in a less-than-5% solution). Acidify with dilute sulfuric acid to pH of less than 3. Slowly and while stirring, add a 50% excess of aqueous bisulfite. An increase in temperature indicates that a reaction is occurring. If the reaction has not begun after addition of

about 10% of the bisulfite, lowering the pH may help. When reaction is complete, neutralize and let the solids settle. Decant solution to drain. Solid residues may be discarded with normal refuse.[15,16]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, cover with at least double the amount of a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Mix completely and uniformly. Behind a shield, cautiously and slowly add to a large volume of water giving a less-than-5% solution. Acidify with dilute sulfuric acid to a pH of less than 3. Slowly and while stirring, add a 50% excess of aqueous sodium bisulfite. An increase in temperature indicates that a reaction is occurring. If the reaction has not begun after addition of about 10% of the bisulfite, lowering the pH may help. When reaction is complete, neutralize and then let the solids settle. Wash the solution to the drain. Solid residues may be treated as normal refuse.[15,16]

Reactions for Spillage and Waste Disposal

$$Na_2O_2 + NaHSO_3 + H_2O \rightarrow 2NaOH + NaHSO_4$$

REFERENCES

1. Merck 8728.
2. NFPA 49.
3. Lux 583.
4. B 1380.
5. Mellor, Vol. 10, 464, 1947.
6. Mellor, Vol. 2, 490, 1941.
7. Barrs, C.E., *J. R. Inst. Chem.*, 79, 43, 1955.
8. B 1382.
9. Mellor, Vol. 2, 490, 1941; Vol. 2, Suppl. 2.1, 633, 1961; Vol. 5, 217, 1946.
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12. Mellor, Vol. 2, 490, 1941.
13. B 1383.
14. Friend, J.N. et al., *Nature*, 134, 778, 1934; Cheeseman, G.H. et al., *Nature*, 134, 971, 1934.
15. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
16. PPL 170.

SODIUM SULFIDE Na_2S

LIBERATES HIGHLY TOXIC HYDROGEN SULFIDE ON CONTACT WITH ACID, FLAMMABLE SOLID

Synonyms

Sodium monosulfide, sodium sulfuret.[1]

Physical Properties

Cubic crystals or granules. Extremely hygroscopic. Discolors on exposure to air.[1]

Pentahydrate, $Na_2S \cdot 5H_2O$. Flat, shiny, four-sided, prismatic crystals. Loses three molecules of water at 100°C; mp, 120°C (with loss of all water of crystallization).

Nonahydrate, $NaS_2 \cdot 9H_2O$. Tetragonal deliquescent crystals. Odor of hydrogen sulfide. Discolors to yellow, and then brownish black on exposure to light and air; mp, ~50°C.[1]

Fire Hazard

Combustible solid. Finely divided material may explode in air. Reaction with water or acid produces hydrogen sulfide. Carbon dioxide causes release of hydrogen sulfide. Combustion may produce irritants and toxic gases.[2]

Chemical Properties

Soluble in water; slightly soluble in alcohol; insoluble in ether. Aqueous solutions are strongly alkaline.[1]

Pentahydrate. Freely soluble in water; soluble in alcohol; insoluble in ether. Aqueous solutions are strongly alkaline. Decomposed by acids with evolution of H_2S.[1]

Nonahydrate. Soluble in water; slightly soluble in alcohol; insoluble in ether. Decomposed by acids, even by carbonic acid. Aqueous solutions are very alkaline and upon standing in contact with air are slowly converted to sodium thiosulfate. Exposure of the crystal to air produces hydrogen sulfide. Keep well closed in a cool place. Do not handle with bare hands.[1]

Hazardous Reactions

Fused sodium sulfide in small lumps is liable to spontaneous heating when exposed to moisture and air.[3]

Acids. Extremely toxic hydrogen sulfide released on contact with acids.[1]

Carbon. Mixtures with finely divided carbon may heat up on exposure to air.[3]

Diazonium Sulfides. May form explosive compounds with diazonium salts.[4]

Physiological Properties and Health Hazards

The solid and solution are irritant and corrosive to skin and eyes. Irritant and poisonous if taken by mouth.[5]

Spillage Disposal

For spills of aqueous solutions of sodium sulfide, wear nitrile rubber gloves, laboratory coat, and eye protection. If there is any risk of hydrogen sulfide being formed, a self-contained breathing apparatus is necessary. Cover the spill with a 1:1:1 mixture of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all of the liquid has been absorbed, scoop the mixture into a plastic pail and place in the fume hood. Add sufficient 1% aqueous sodium hydroxide solution to

dissolve the sodium carbonate in the spill mix and to ensure that the liquid is basic. Estimate the volume of sodium sulfide solution that was spilled and add 175 mL of household chlorine bleach containing 5.25% sodium hypochlorite for each 10 mL of commercial sodium sulfide solution (commercial sodium sulfide solution is about 20%; this volume of bleach allows a 25% excess). Allow the mixture to stand in the fume hood overnight and decant the liquid into the drain. Dispose of the solid (sand and calcium bentonite) in the normal refuse.[6]

Waste Disposal

Package Lots. Place in labeled containers for recycling or disposal in accordance with local regulations.

Small Quantities. Sodium sulfide can be converted to sodium sulfate by oxidation with household bleach. For each 10 mL of commercial sodium sulfide solution, add 100 mL of 1% aqueous sodium hydroxide solution and 175 mL of household bleach. Allow the solution to stand overnight, and then pour into the drain.[6]

Reactions for Spillage and Waste Disposal

$$Na_2S + 4NaOCl \rightarrow Na_2SO_4 + 4NaCl$$

REFERENCES

1. Merck 8756.
2. NFPA 49.
3. B 1386.
4. B 1565
5. Lux 584.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; Armour, M.A., *Laboratory Disposal Method for Sodium Sulfide,* University of Alberta, Edmonton, 1997; *Haz. Mat. Spills Tec.*, 29.1.

STIBINE SbH_3

VERY TOXIC

Synonyms

Antimony trihydride.

Physical Properties

Colorless gas; disagreeable odor; decomposes slowly at room temperature; mp, –88°C; bp, –18.4°C.[1]

Fire Hazard

Ignites on heating.[2]

Chemical Properties

Quickly decomposes at 200°C; slightly soluble in water; soluble in alcohol, carbon disulfide, and other organic solvents.[1]

Hazardous Reactions

May decompose explosively, even at low temperatures.[3]

Ammonia. A mixture with ammonia explodes on heating.[4]

Chlorine. Reacts explosively with chlorine and chlorine water.[5]

Nitric Acid. Oxidized explosively by concentrated acid.[4,6]

Ozone. An explosion resulted when oxygen containing 2% ozone was passed through stibine at –90°C.[7]

Physiological Properties and Health Hazards

Highly toxic vapor. Symptoms, often latent for up to 2 days, include headache, vomiting, anorexia, numbness, abdominal cramping, chills, and nausea. Death results from renal failure and pulmonary edema (see Arsine).[1,2] TLV-TWA 0.1 ppm (0.51 mg/m^3).[8]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, goggles, face shield, and self-contained breathing apparatus. Remove the leaking cylinder to a safe area outdoors or to an efficient fume hood. Fit the cylinder with an appropriate control valve and length of hose. Discharge the gas slowly into 15% aqueous sodium hydroxide. Adjust the pH of the sodium hydroxide solution (pH 7–8) with 1 M sulfuric acid (prepared by cautiously adding 5 mL of concentrated acid to 85 mL of cold water) or 1 M sodium hydroxide (prepared by dissolving 4 g of NaOH in 100 mL of cold water). The antimony hydroxide is insoluble in strong base. The precipitate is separated by filtration and packaged for disposal in a secure landfill. The aqueous solution is neutralized and then washed into the drain.[9]

Reactions for Spillage and Waste Disposal

$$SbH_3 + 3H_2O \rightarrow Sb(OH)_3 + 3H_2$$

REFERENCES

1. Merck 8889.
2. ITI 056, 063.
3. Mellor, Vol. 9, 394, 1939.
4. *ibid*., 397.
5. *ibid*., 396.
6. Mellor, Vol. 5, 36, 1946; Vol. 6, 814, 1940.
7. Stock, A. et al., *Chemische Berichte*, 38, 3837, 1905.
8. ACGIH 15.
9. PPL 166.

STREPTOZOTOCIN $C_8H_{15}N_3O_7$

ANIMAL CARCINOGEN

Synonyms

2-Deoxy-2-{[(methylnitrosamino)carbonyl]amino}-D-glucopyranose, 2-deoxy-2-(3-methyl-3-nitrosoureido)-D-glucopyranose, streptozocin.[1]

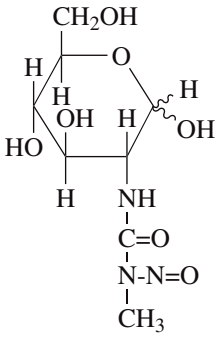
Physical Properties

Pointed platelets or prisms from 95% ethanol; mp, 115°C (decomposition).[1] A mixture of α and β stereoisomers; aqueous solutions rapidly undergo mutarotation to an equilibrium value of $[\alpha]_D^{25}$ + 39°.[1]

Chemical Properties

Soluble in water, lower alcohols, and ketones; insoluble in nonpolar organic solvents. Decomposes to diazomethane in alkaline solutions at 0°C.[2]

Structure



Mechanism of Action

Direct inhibition of DNA, RNA, and protein synthesis. The drug also inhibits the progress of cells into mitosis from G_2 of the cell cycle.[3]

Physiological Properties and Health Hazards

Animal carcinogen. Reasonably anticipated to be a human carcinogen.[4] Produces experimental diabetes in laboratory animals.[1] Adverse effects in patients including nausea, vomiting, diarrhea, and reversible liver function abnormalities. The most serious toxic effects are renal toxicity.[3] LD_{50} i.v. in rats, 137.7 mg/kg; in dogs, 25–50 mg/kg.[1]

Spillage Disposal

Solutions. Wear protective gloves, clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Wash the spill area with soap and water. Estimate the weight of absorbing mixture used, add to the mixture a 3 M solution of sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate, allowing

12 mL of solution per 48 mg of streptozotocin. Allow to stand overnight, neutralize by careful addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite until a colorless liquid is formed. Decant the liquid into the drain with water and discard the residue as normal refuse.[2,5]

Waste Disposal

Wear rubber gloves, protective clothing, and goggles. Work in the fume hood. To 10 mL of an aqueous solution containing 48 mg of streptozotocin, add 2 mL of concentrated sulfuric acid and 2.0 g of potassium permanganate. Continue stirring for 12 hours. Neutralize the solution by the slow addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite until a colorless liquid is formed. Wash into the drain with water.[2,5]

REFERENCES

1. Merck 8912.
2. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents,* No. 73, IARC Scientific Publications, Lyon, 1985, p. 123.
3. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, MA, 1982, pp. 148–150.
4. NIEHS III–198.
5. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 103; *Haz. Mat. Spills Tec.*, 29.7.

STRONTIUM Sr

IGNITES IN AIR WHEN FINELY DIVIDED

Physical Properties

Silvery-white metal; rapidly becomes yellow on exposure to air.[1]

Chemical Properties

Vigorous reaction with water.[1]

Hazardous Reactions

Air. The finely divided metal may ignite in air.[2]

Halogens. Incandesces in chlorine at 300°C and ignites in bromine at 400°C.[2]

Water. Vigorous interaction.[2]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover the spill with a 1:1:1 mixture by weight of sodium or calcium carbonate, clay cat litter (bentonite), and sand. Sweep the mixture into a container and transport to the fume hood. Slowly add to a large container of water and mix. Neutralize with dilute hydrochloric acid. Wash into the drain.[3,4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Work in the fume hood. Wear nitrile rubber gloves, eye protection, and laboratory coat. Dissolve waste in a minimum volume of 6 M hydrochloric acid (prepared by cautiously adding concentrated acid to an equal volume of cold water) and filter. Neutralize with 6 M ammonium hydroxide (prepared by cautiously adding 36 mL of concentrated base to 69 mL of cold water; use pHydrion paper) and precipitate with excess sodium carbonate. Filter, wash, and dry the precipitate. Recycle or send for disposal.[3]

REFERENCES

1. Merck 8915.
2. B 1460.
3. ITI 605.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.

SULFAMIC ACID HSO_3NH_2

IRRITATING TO SKIN AND EYES

Synonyms

Sulfamidic acid, amidosulfuric acid.

Physical Properties

White crystals melting at about 205°C with decomposition.[1]

Chemical Properties

One part dissolves in about six parts water at 0°C, and in about two parts at 80°C. Sparingly soluble in alcohol; slightly soluble in acetone; insoluble in ether.[1]

Hazardous Reactions

Nitrates or Nitrites. Heating with mixtures of sodium or potassium nitrates or nitrites leads to reactions that may be explosive.[2]

Nitric Acid. Mixing with fuming nitric acid results in violent release of nitrous oxide.[3]

Physiological Properties and Health Hazards

Dust or solution irritates the eyes, skin, and mucous membranes. Prolonged exposure of skin to the acid may cause irritation. Avoid contact with skin and eyes.[4]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Clean up with dust pan and brush. May be flushed away to waste with at least 50 volumes of water or mixed with sand and disposed of as normal refuse.[4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Sift slowly into a large container of cold water, with agitation. When dissolved, neutralize with sodium carbonate and pour into the drain with at least 50 times its volume of water.[4]

REFERENCES

1. Merck 9006.
2. Heubel, J. et al., *Compt. Rend.* [3], 257, 684, 1963.
3. Mellor, Vol. 8, Suppl. 2, 316, 1977.
4. Lux 187, 210.

SULFURIC ACID H_2SO_4

CAUSES SEVERE BURNS

Physical Properties

Concentrated sulfuric acid is a colorless, oily liquid; bp, 290°C.[1]

Fire Hazard

May ignite finely divided combustible materials on contact.[2] Do not use water to extinguish fire.[2]

Chemical Properties

Reacts vigorously with water.[1,2]

Hazardous Reactions

Acetaldehyde. Polymerized violently by concentrated acid.[3]

Acetone and Nitric Acid. Acetone is oxidized violently by a nitric acid and sulfuric acid mixture.[4]

Acetonitrile. Strongly exothermic reaction on warming.[5]

Acrylonitrile. Vigorous reaction on contact with even small quantities of strong acid.[6]

Alkyl Nitrates. Violent reaction with sulfuric acid.[7]

Benzyl Alcohol. Decomposes explosively at about 180°C in the presence of 58% sulfuric acid.[8]

1-Chloro-2,3-epoxypropane. Interaction is violent.[9]

2-Cyano-2-propanol. Vigorous reaction due to exothermic polymerization of the dehydration product, methacrylonitrile.[10]

Cyclopentadiene. Violent or explosive reaction with concentrated acid.[11]

Cyclopentanone Oxime. Violent reaction occurred on heating the oxime with 85% sulfuric acid to effect a Beckmann rearrangement.[12]

1,3-Diazidobenzene. Ignites and explodes mildly on contact with concentrated acid.[13]

Metal Acetylides or Carbides. Monocesium and monorubidium acetylides ignite on contact with concentrated sulfuric acid.[14] Hazardous reactions occur with other carbides.[12]

Metal Chlorates. May explode violently with concentrated sulfuric acid.[15]

Metal Perchlorates. Explosive perchloric acid is formed in contact with concentrated sulfuric acid.[16]

Nitric Acid and Organic Matter. A violent explosion may occur when organic residues are dissolved in the mixed concentrated acids.[17]

Nitric Acid and Toluene. Nitration of toluene with mixed concentrated acids must be carefully controlled or an explosion can result.[18]

Nitroaryl Bases and Derivatives. *o*-Nitroaniline reacts almost explosively and *p*-nitroaniline, *p*-nitroacetanilide, aminonitrodiphenyls, aminonitronaphthalenes, and some nitro-N-heterocycles react vigorously when heated with sulfuric acid above 200°C.[19–21]

Nitromethane. Mixture with sulfuric acid is susceptible to detonation.[22]

Nitromethylamine. Decomposes explosively with concentrated sulfuric acid.[23]

p-Nitrotoluene. Decomposes violently when heated with concentrated sulfuric acid to 160°C.[24]

Permanganates. Explosion may occur with concentrated acid.[25]

Phosphorus Trioxide. Ignites on addition of sulfuric acid.[26]

Potassium. Explosive interaction.[27]

Sodium. Slow reaction with anhydrous acid; explosive reaction with aqueous acid.[28]

Sodium Borohydride. Reaction with concentrated sulfuric acid to yield diborane may be dangerously violent.[29]

Water. Dilution of the acid with water is vigorously exothermic; the acid must always be added to water to avoid local boiling.[30]

Physiological Properties and Health Hazards

Concentrated acid causes severe burns to the eyes and skin. Dilute acid irritates the eyes and skin. Swallowing concentrated acid would cause severe internal damage. Prevent contact with skin and eyes.[31] TLV-TWA 1 mg/m^3; TLV-STEL 3 mg/m^3;[32] LD_{50} (rats) 2.14 g/kg.[1]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves.[33] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a pail in the fume hood. Slowly and cautiously add this mixture to a large volume of water in a pail. Neutralize with sodium carbonate if necessary. Let stand until solids settle. Decant liquid into the drain. Solid residue may be discarded with normal refuse.[34,35]

Waste Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Cautiously and slowly add to a large volume of ice-cold water. Slowly and carefully add sodium carbonate until neutralization is complete. Wash into the drain.[34,35]

Reactions for Spillage and Waste Disposal

$$H_2SO_4 + Na_2CO_3 \rightarrow \underset{\text{sodium sulfate}}{Na_2SO_4} + H_2O + CO_2$$

REFERENCES

1. Merck 9064.
2. NFPA 49.
3. B 272.
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27. B 1292.
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SULFURYL CHLORIDE SO_2Cl_2

CAUSES SEVERE BURNS, IRRITANT

Physical Properties

Colorless or yellow, fuming liquid with pungent odor; bp, 69°C.[1]

Chemical Properties

Decomposed by water with formation of hydrochloric and sulfuric acids.[1]

Hazardous Reactions

Alkalies. Reaction may be explosively violent.[2]

Diethyl Ether. Decomposes vigorously in ether, especially if peroxides are present.[3]

Dimethyl Sulfoxide. Reacts violently or explosively in dimethyl sulfoxide,[4] probably due to the formation and polymerization of formaldehyde.[5]

Dinitrogen Pentoxide. Reacts explosively.[6]

Lead Dioxide. Reacts explosively.[7]

Phosphorus. Red phosphorus reacts vigorously on warming.[8]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. Liquid causes burns. Swallowing would cause severe internal irritation and damage. Avoid breathing vapor. Prevent contact with skin and eyes. Do not put water into container.[9]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the sulfuryl chloride has been absorbed, scoop the mixture into a plastic pail and, in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[10]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the sulfuryl chloride into a large evaporating dish. Cover the sulfuryl chloride with excess solid soda ash or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.[10]

Reactions for Spillage and Waste Disposal

$$SO_2Cl_2 + 2Na_2CO_3 \rightarrow \underset{\text{sodium sulfate}}{Na_2SO_4} + \underset{\text{sodium chloride}}{2NaCl} + 2CO_2$$

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TANNIC ACID $C_{76}H_{52}O_{46}$

ANIMAL CARCINOGEN

Physical Properties

Yellowish-white to light brown, amorphous, bulky powder, flakes, or spongy masses; faint characteristic odor; astringent taste; gradually darkens on exposure to air and light; dec, 210–215°C.[1]

Chemical Properties

One gram dissolves in 0.35 mL water and 1 mL of glycerol; very soluble in alcohol and acetone; practically insoluble in benzene, chloroform, ether, petroleum ether, carbon disulfide, and carbon tetrachloride.[1]

Physiological Properties and Health Hazards

Irritant.[3] An experimental carcinogen of the liver by subcutaneous route.[2,3]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to an appropriate labeled container for disposal by burning.[3,4]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning.[3]

REFERENCES

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3. Ald 3211
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sym-TETRABROMOETHANE $C_2H_2Br_4$

HIGHLY TOXIC, IRRITANT

Synonyms

1,1,2,2-Tetrabromoethane, acetylene tetrabromide, Muthmann's liquid.[1]

Physical Properties

Yellowish, heavy, very refractive liquid; odor of camphor and iodoform; bp_{54}, 151°C; mp, 0°C.[1]

Chemical Properties

Insoluble in water. Miscible with alcohol, chloroform, ether, aniline, and glacial acetic acid.[1]

Hazardous Reactions

Incompatible with strong oxidizing agents (such as nitric acid and potassium permanganate) and strong bases (such as sodium and potassium hydroxides).[2]

Physiological Properties and Health Hazards

Irritant, narcotic.[1,4] Irritant to the upper respiratory tract, producing changes in the liver, kidneys, and central nervous system.[3,4] Very potent mutagen.[5] TLV-TWA 1 ppm (14 mg/m^3).[6]

Spillage Disposal

Wear nitrile rubber gloves, a laboratory coat, and self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (calcium bentonite), and sand. When all liquid has been absorbed, scoop the mixture into an appropriate container and package and label for disposal by burning.[7]

Waste Disposal

Package and label for disposal by burning.[2]

REFERENCES

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4. Sax 45.
6. ACGIH 13.
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TETRACHLOROSILANE $SiCl_4$

VIOLENT REACTION WITH WATER

Synonyms

Silicon tetrachloride.

Physical Properties

Colorless, clear, mobile, fuming liquid; suffocating odor; mp, –70°C; bp, 59°C.[1]

Chemical Properties

Miscible with benzene, ether, chloroform, petroleum, and ether[1]; decomposes in alcohol.[2]

Hazardous Reactions

Sodium. Mixture is shock-sensitive explosive.[3]

Water. Violent reaction.[4]

Physiological Properties and Health Hazards

Vapor irritates the respiratory system and eyes. Liquid burns eyes and skin. Severe internal damage results from ingestion.[4]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, face shield, and goggles. Cover spill with a 1:1:1 mixture of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Cautiously and slowly add to a large volume of water. Decant the solution into the drain. The solid may be treated as normal refuse.[4,5]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Place a 1-L, three-necked, round-bottom flask, equipped with a stirrer, dropping funnel, and thermometer, on a steam bath. Place in the flask 600 mL (1.5 mol, 50% excess) of a 2.5 M sodium hydroxide solution (prepared by slowly dissolving 60 g of NaOH in 600 mL of cold water) and add tetrachlorosilane (42.5 g or 28 mL; 0.25 mol) dropwise at such a rate to maintain gentle reflux. The rate of the reaction must be carefully controlled. After addition is complete, heat the mixture until a clear solution is obtained. Cool the mixture to room temperature, neutralize with dilute hydrochloric acid and wash into the drain.[6]

Reactions for Spillage and Waste Disposal

$$SiCl_4 + 4OH^- \rightarrow Si(OH)_4 + 4Cl^-$$

REFERENCES

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3. B 1376.

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TETRAHYDROFURAN C_4H_8O

EXTREMELY FLAMMABLE, MAY FORM EXPLOSIVE PEROXIDES

Physical Properties

Colorless, volatile liquid with ethereal odor; bp, 66°C.[1]

Fire Hazard

Flash point, –14°C; explosive limits, 2–11.8%; ignition temperature, 321°C. Extinguish fire with dry chemical or carbon dioxide. Water may be ineffective unless as a spray.[2]

Chemical Properties

Miscible with water, alcohol, ketones, esters, ethers, and hydrocarbons.[1] Liable to form explosive peroxides on exposure to light and air. These should be decomposed before distilling to small volume.[3]

Hazardous Reactions

Air. Readily forms peroxides by auto-oxidation. Initially the 2-hydroperoxide is formed. This decomposes smoothly but if allowed to accumulate, it may be transformed into other peroxides that decompose violently. Procedures for testing for and removing peroxides have been published.[4–7] The use of cuprous chloride is recommended for removal of trace quantities of peroxides.[5]

Alkali. Material containing peroxides should not be dried with KOH or NaOH because explosions may occur.[5]

Lithium Aluminum Hydride. The hydride should only be used to remove the last traces of water from tetrahydrofuran, which is peroxide-free.[8]

Purification. A purification method has been published.[9] Peroxidized material should not be dried with sodium or potassium hydroxides as explosions may occur.[3]

Physiological Properties and Health Hazards

Vapor irritates the eyes and respiratory system; high concentrations have a narcotic effect. Liver damage may result from skin absorption or ingestion. Avoid breathing vapor. Avoid contact with eyes.[3] TLV-TWA 200 ppm (590 mg/m^3); TLV-STEL 250 ppm (737 mg/m^3).[10]

Spillage Disposal

Shut off all possible sources of ignition. Instruct others to maintain a safe distance. Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves[11]. Cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transfer to a large dish and let evaporate in fume hood. Ventilate area well to evaporate remaining liquid and dispel vapor. Treat solid residue as normal refuse.[3,12]

Waste Disposal

Containers of tetrahydrofuran that have been opened and are more than 1 year old may contain hazardous quantities of peroxides. Especially if they have screw caps, these should not be opened but should be disposed of by the appropriate authorities. When the container can be opened safely, check for peroxides as follows: Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Dissolve 100 mg of potassium iodide in 1 mL of glacial acetic acid. Add to 1 mL

of tetrahydrofuran. A pale yellow color indicates a low concentration (0.001–0.005%), and a bright yellow or brown color a high concentration (0.01% and hazardous) of peroxide in the sample.[13] To remove peroxides, wear butyl rubber gloves, laboratory coat, and eye protection. Pour the tetrahydrofuran (100 mL) into a separatory funnel and shake with a freshly prepared 50% aqueous solution of sodium metabisulfite (20 mL) for 3 minutes. Release the pressure in the funnel at 10-second intervals. Separate the aqueous layer. Retest the tetrahydrofuran for the continued presence of small amounts of dialkyl peroxides that are not reduced by the metabisulfite treatment. If peroxides are absent, the ether can be dried for re-use or packaged for disposal by burning. If peroxides are still present, in the fume hood place the tetrahydrofuran in a 250-mL round-bottom flask equipped with a condenser and add a solution of 100 mg of potassium iodide in 5 mL of glacial acetic acid and 1 drop of concentrated hydrochloric acid. Reflux gently on the steam bath for 1 hour.[12] Package the tetrahydrofuran for disposal by burning.[14]

REFERENCES

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3. Lux 187, 600.
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7. Davies, A.G., *J. R. Inst. Chem.*, 80, 386, 1956.
8. Moffett, R.B., *Chem. Eng. News*, 32, 4328, 1954.
9. *Org. Synth.*, Coll. Vol. 5, 976, 1973.
10. ACGIH 56.
11. LSS.
12. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
13. Jackson, H.L. et al., *J. Chem. Educ.*, 3, 114, 1974.
14. Ald 3273C; ITI 626.

TETRAMETHYLSILANE $(CH_3)_4Si$

FLAMMABLE

Physical Properties

Colorless flammable liquid;[1] mp, –27°C; bp, 26–28°C.[2]

Fire Hazard

Flammable liquid.[2] Flash point –27°C.[1,3]

Chemical Properties

Soluble in ether.[1] Moisture sensitive.[2]

Hazardous Reactions

In the preparation of chlorotrimethylsilane by the antimony trichloride-catalyzed chlorination of tetramethylsilane, the reaction is explosive at 100°C, but controllable below 30°C.[4]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into an open container in the fume hood and allow to evaporate. Ventilate spill site well to evaporate remaining liquid and dispel vapor.[5]

Waste Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Absorb the liquid on vermiculite and allow it to evaporate or cover it with sodium carbonate and package and label for incineration.[2]

REFERENCES

1. Sax 3247.
2. Ald 3303B.
3. B 1844.
4. Bush, R.P., British Patent 1 388 991, 1975 (*Chem. Abs.*, 83, 59026e, 1975).
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.

THALLIUM AND SALTS Tl^{2+}

POISONOUS DUST, SERIOUS RISK OF POISONING BY INHALATION OR SWALLOWING, DANGER OF CUMULATIVE EFFECTS

Physical Properties

Thallium is a soft bluish-white metal; mp, 303.5°C. The carbonate and sulfate are white crystalline solids; the oxide is black.[1]

Chemical Properties

The carbonate and sulfate are water soluble; the oxide forms hydroxide in water.[1]

Physiological Properties and Health Hazards

The dusts irritate the nose and eyes and may cause nausea and abdominal pain by absorption. The metal on contact with moist skin produces a white film of the hydroxide. Skin absorption of the soluble salts and ingestion may cause nausea, vomiting, abdominal pains, weakness of the legs, and mental confusion. Prolonged exposure to small amounts of the dust or solutions may result in loss of appetite, loss of hair, pain or weakness of limbs, insomnia, and mental disturbance. Avoid breathing dust. Avoid contact with skin and eyes.[2] TLV-TWA 0.1 mg/m^3.[3]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear nitrile rubber gloves, laboratory coat, a face shield, and goggles. Work in the fume hood. Dissolve the thallium salt in water. Add a 10% aqueous solution of sodium sulfide until no further precipitation occurs. Filter the precipitated thallium sulfide, dry, package, and send for disposal in a secure landfill site. Destroy excess sulfide in the filtrate by the addition of laundry bleach, neutralize the solution with 6 M hydrochloric acid (prepared by cautiously adding a volume of concentrated acid to an equal volume of cold water), and wash into the drain.[4]

Reactions for Spillage and Waste Disposal

$$Tl^{2+} + H_2S \rightarrow \underset{\substack{\text{thallium}\\\text{sulfide}\\\text{(insoluble)}}}{TlS} + 2H^+$$

REFERENCES

1. Merck 9327, 9330, 9337, 9340.
2. Lux 604.
3. ACGIH 57.
4. Ald 3315A-3317C.

6-THIOGUANINE $C_5H_5N_5S$

ANTINEOPLASTIC

Synonyms

Lanvis, 6-TG.

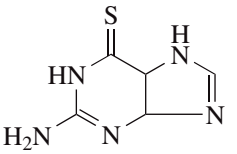
Physical Properties

Pale yellow crystalline powder or needles from water; mp, above 360°C.[1]

Chemical Properties

Insoluble in water, alcohol, and chloroform. Very soluble in dilute solutions of alkali hydroxides.[2]

Structure



Mode of Action

This compound is an analogue of guanine. It is converted into a nucleotide and incorporated into DNA. It also inhibits purine-synthesizing agents.[3]

Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, anorexia, diarrhea, rash, and bone marrow depression.[3]

Waste Disposal

Wear protective gloves, clothing, and goggles. Dissolve 6-thioguanine (40 mg) in 80 mL of 3 M sulfuric acid (13.6 mL of concentrated sulfuric acid slowly added to 66.4 mL of cold water). While stirring, add 0.5 g of potassium permanganate in small portions over a period of about 10 minutes.

Stir the mixture at room temperature for 12 hours and neutralize by slow and careful addition of soda ash or 10% sodium hydroxide solution. While stirring, add a saturated aqueous solution of sodium bisulfite until a colorless liquid is formed. Wash into the drain with water. Immerse vials and glassware containing 6-thioguanine residues in a solution of 0.5 g of potassium permanganate in 80 mL of 3 M sulfuric acid (13.6 mL of concentrated sulfuric acid slowly added to 66.4 mL of cold water) and allow to stand overnight. Rinse the glassware thoroughly with water and discard or re-use. Neutralize the cleaning solution by slow and careful addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite until a colorless liquid is formed. Wash into the drain with water.[4]

To 1 mL of an aqueous solution containing 3.2 mg of thioguanine, add 25 mL of household bleach. Stir the mixture at room temperature for 90 minutes. When reaction is complete, wash the solution into the drain with water.[4]

REFERENCES

1. Merck 9411.
2. Castegnaro, M. et al., *Laboratory decontamination and destruction of carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 81.
3. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, MA, 1982, p. 155.
4. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 104; *Haz. Mat. Spills Tec.*, 29.7.

THIONYL CHLORIDE $SOCl_2$

CAUSES BURNS, SEVERE IRRITANT

Physical Properties

Colorless to pale yellow or reddish fuming liquid; suffocating odor; bp, 76°C.[1]

Chemical Properties

Reacts with water to form hydrochloric acid and sulfur dioxide.[1]

Hazardous Reactions

Ammonia. Addition of concentrated ammonia may cause an explosion.[2]

Bis(dimethylamino) sulfoxide. Interaction causes decomposition that may become explosive above 80°C.[3]

N,N-Dimethylformamide. A mixture decomposes vigorously at room temperature in the presence of traces of iron or zinc.[4]

Dimethyl sulfoxide. Violent or explosive reaction occurs due to the formation and exothermic polymerization of formaldehyde.[5,6]

Linseed Oil and Quinoline. These reagents, used in purifying thionyl chloride, must be added to the chloride. If the chloride is added to them, vigorous decomposition may occur.[7]

Water. Violent interaction produces hydrochloric acid and SO_2.[8]

Physiological Properties and Health Hazards

Avoid breathing vapor. Prevent contact with skin and eyes. Destructive to tissues of respiratory system; causes burns. Harmful by inhalation, ingestion, or absorption through the skin.[9] TLV-STEL-C 1 ppm (4.9 mg/m^3).[10]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, eye protection, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. When the thionyl chloride has been absorbed, scoop the mixture into a plastic pail, and in the fume hood, very slowly add the mixture to a pail of cold water. Allow to stand 24 hours. Test the pH of the solution and neutralize if necessary with sodium carbonate. Decant the solution to the drain. Treat the solid residue as normal refuse.[11]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear butyl rubber gloves, laboratory coat, and eye protection. In the fume hood, pour the thionyl chloride into a large evaporating dish. Cover the thionyl chloride with excess solid sodium carbonate or calcium carbonate. When the reaction has subsided, very slowly add the mixture to a pail of cold water. Allow to stand for 24 hours. Test the pH of the solution and neutralize if necessary. Wash the solution into the drain.[11]

Reactions for Spillage and Waste Disposal

$$SOCl_2 + Na_2CO_3 \rightarrow SO_2 + 2NaCl + CO_2$$

REFERENCES

1. Merck 9423.
2. B 1023.
3. Armitage, D.A. et al., *J. Inorg. Nucl. Chem.*, 36, 993, 1974.
4. Spitulnik, M.J., *Chem. Eng. News*, 55, 31, 1977.
5. Buckley, A., *J. Chem. Educ.*, 42, 674, 1965.
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7. B 1024.
8. B 1025.
9. Lux 605.
10. ACGIH 57.
11. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.3.

THIOUREA H_2NCSNH_2

ANIMAL CARCINOGEN

Synonyms

Thiocarbamide.

Physical Properties

Crystals; mp, 176–178°C.[1]

Chemical Properties

Soluble in water and alcohol; sparingly soluble in ether. Forms addition compounds with metallic salts.[1]

Hazardous Reactions

Strong oxidizing agents and strong acids. Reaction may be vigorous.[2]

Acrylaldehyde. Heat is evolved in a vigorous reaction and polymerization may occur.[3]

Hydrogen Peroxide and Nitric Acid. A solid peroxide may be formed that can decompose violently.[4]

Physiological Properties and Health Hazards

Harmful if inhaled, swallowed, or absorbed through the skin. May cause irritation.[5] Animal carcinogen; reasonably anticipated to be a human carcinogen.[6] LD_{50} (oral-rat) 125 mg/kg.[2]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and goggles. Sweep up the solid into a container and place in the fume hood. Wash the spill area thoroughly with soapy water. Add sufficient water to the solid so that it dissolves. Estimate the weight of thiourea spilled and add 100 mL of household bleach (5% sodium hypochlorite solution) for each 1 g of thiourea. Allow to stand overnight. Pour the liquid into the drain.[7]

Waste Disposal

Package Lots. Package and label for disposal by burning.[2]

Small Quantities. Follow spillage disposal procedure.[7]

REFERENCES

1. Merck 9433.
2. Ald 3338B.
3. B 358.
4. B 1210.
5. Lux 606.
6. NIEHS III–204.
7. Armour, M.A., *Laboratory Disposal Method for Thiourea*, University of Alberta, Edmonton, 1997.

TIN(IV) CHLORIDE $SnCl_4$

CAUSES BURNS, IRRITANT

Synonyms

Stannic chloride, tin tetrachloride.

Physical Properties

Colorless fuming liquid; bp, 114°C.[1]

Chemical Properties

Reacts with water forming hydrochloric acid.[2]

Hazardous Reactions

Potassium. Mixture explodes violently on impact.[3]

Sodium. Mixture explodes violently on impact.[4]

Turpentine. Reaction is strongly exothermic and may ignite.[5]

Physiological Properties and Health Hazards

The vapor irritates the respiratory system and eyes. The liquid irritates the eyes and skin and may cause burns. Swallowing will result in internal irritation and damage. Avoid contact with skin and eyes. Do not put water into container.[2] TLV-TWA 2 mg/m^3.[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, goggles, and a face shield. A self-contained breathing apparatus may be necessary, depending on the size of the spill. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), or sand. When the tin chloride has been absorbed, scoop the mixture (some fuming will occur when the solid mass is broken up) into a plastic pail in the fume hood, and then very slowly add the mixture to a pail of cold water. Let stand for 24 hours. Neutralize if necessary with sodium carbonate or dilute hydrochloric acid. Decant the solution into the drain. Treat the solid residue as normal refuse.[7]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In the fume hood, cautiously and slowly add to a large container of water. Slowly stir in sodium carbonate until the solution is neutral to litmus, and a thick, white precipitate has formed. Let stand for 24 hours. Wash the liquid into the drain. Treat the solid as normal refuse.[7]

Reactions for Spillage and Waste Disposal

$$SnCl_4 + 4H_2O \rightarrow Sn(OH)_4 + 4HCl$$

REFERENCES

1. Merck 8852.
2. Lux 607.

3. Mellor, Vol. 2, Suppl. 3, 1571, 1963.
4. Mellor, Vol. 2, Suppl. 2, 497, 1961.
5. Mellor, Vol. 7, 446, 1941.
6. ACGIH 57.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.

TITANIUM TETRACHLORIDE $TiCl_4$

VIOLENT REACTION WITH WATER

Physical Properties

Colorless liquid; fumes in moist air; mp, –24.1°C; bp, 136.4°C; penetrating acid odor.[1]

Chemical Properties

Soluble in cold water, alcohol, and dilute hydrochloric acid.[2] Decomposes in hot water.[1]

Hazardous Reactions

Hydrogen Fluoride. Violent reaction.[3]

Potassium. Violent reaction.[3]

Urea. Hexaurea complex formed after 6 weeks at 80°C; decomposes violently at 90°C.[4]

Water. Violent reaction with water liberating HCl.[5]

Physiological Properties and Health Hazards

Irritates the skin, eyes, and respiratory system.[5] Liquid irritates the skin. Assumed to cause severe internal irritation if ingested. Avoid contact with skin and eyes.[5]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, goggles, and a face shield. Cover the spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Slowly and cautiously, add the mixture to a pail of cold water. Test the pH of the solution and, if acidic, neutralize with 10% sodium hydroxide solution. Decant the solution into the drain. Treat the solid residue (which contains the highly insoluble titanium oxide) as normal refuse.[6]

Waste Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Work in the fume hood. Add the titanium tetrachloride (10 mL) dropwise from a dropping funnel to a stirred 5% solution of sodium hydroxide (250 mL) cooled in an ice bath. Filter the solid and treat as normal refuse. Wash the filtrate into the drain.[6]

Reactions for Spillage and Waste Disposal

$$TiCl_4 + 2H_2O \rightarrow \underset{\substack{\text{titanium}\\\text{dioxide}\\\text{(insoluble)}}}{TiO_2} + 4HCl$$

REFERENCES

1. Merck 9555.
2. CRC.
3. Sax 3302.
4. Ionova, E.A. et al., *Chem. Abs.*, 64, 9219b, 1966.

5. Lux 609.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; Armour, M.A. and Young, R., *Laboratory Disposal Method for Titanium Tetrachloride*, University of Alberta, Edmonton, 1996; *Haz. Mat. Spills Tec.*, 29.1.

o-TOLIDINE $(CH_3(NH_2)C_6H_3\text{-})_2$
and *o*-TOLIDINE DIHYDROCHLORIDE $(CH_3(NH_2)C_6H_3\text{-})_2 \cdot 2HCl$

ANIMAL CARCINOGEN, POISONOUS DUST, TOXIC IN CONTACT WITH SKIN, DANGER OF CUMULATIVE EFFECTS

Synonyms

3,3'-Dimethylbenzidine.

Physical Properties

White to reddish crystals or powder; mp, 129–131°C (base).[1]

Chemical Properties

Base is slightly soluble in water; soluble in alcohol, ether, and dilute acids.[1]

Physiological Properties and Health Hazards

The dihydrochloride and its solutions irritate the skin and eyes. Prevent inhalation of dust. Prevent contact with skin and eyes.[2] No TLV. Animal carcinogen; reasonably anticipated to be a human carcinogen.[3]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Moisten and shovel mixture into pail for dispersion in an excess of dilute hydrochloric acid (1 volume of concentrated acid diluted with 2 volumes of water). Allow to stand, with occasional stirring, for 24 hours, and then run solution to waste, diluting with at least 50 times its volume of water. The solid residue can then be treated as normal refuse. The spill site should be washed with water and soap or detergent.[2,4]

Waste Disposal

Package Lots. Place in a separate labeled container for recycling or disposal by burning.[5]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. To a 100-mL, three-necked, round-bottom flask equipped with a stirrer, thermometer, and dropping funnel, add 2.5 mL of water, 7.5 mL of concentrated hydrochloric acid and 4.24 g (0.02 mol) of *o*-tolidine. Maintain the temperature −5 to 0°C by a cooling bath, while dissolving 1.5 g (0.0211 mol) of 97% sodium nitrite in 3.5 mL of water by adding dropwise to the mixture of *o*-tolidine hydrochloride. Continue stirring an additional 30 minutes after addition is complete. While maintaining the temperature at −5 to 0°C, add 41.6 mL (0.4 mol) of 50% hypophosphorous acid (precooled to 0°C) over 10–15 minutes. Continue stirring for 1 hour. Allow the mixture to stand at room temperature for 24 hours, and then extract with 2×10 mL portions of toluene. Package the toluene extract of the deamination product, dimethylbiphenyl, for disposal by burning. Wash the aqueous phase into the drain.[6]

Reactions for Spillage and Waste Disposal

$$(CH_3(NH_2)C_6H_3)_2 \cdot 2HCl \xrightarrow[2)\ 2H_3PO_2]{1)\ 2HNO_2} (CH_3C_6H_4)_2 + 2HCl$$

dimethylbiphenyl

REFERENCES

1. Merck 9591.
2. Lux 187, 610.
3. NIEHS III–118; ACGIH 57.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.
5. ITI 075.
6. PP 70.

TOLUENE $C_6H_5CH_3$

HIGHLY FLAMMABLE, HARMFUL VAPOR

Synonyms

Toluol, methylbenzene, phenylmethane.

Physical Properties

Colorless liquid with characteristic odor; bp, 111°C.[1]

Fire Hazard

Flash point, 4°C; explosive limits, 1.2–7.1%; ignition temperature, 480°C. Extinguish fire with foam, dry powder, or carbon dioxide.[2]

Chemical Properties

Very slightly soluble in water; miscible with alcohol, chloroform, ether, acetone, glacial acetic acid, and carbon disulfide.[1]

Hazardous Reactions

Bromine Trifluoride. Frozen toluene reacts violently with bromine trifluoride at −80°C.[3]

Dinitrogen Tetroxide. Mixture of toluene and the tetroxide is liable to explode.[4]

Nitric Acid and Sulfuric Acid. Inadequate control in nitration of toluene with mixed acids may lead to a runaway or explosive reaction.[5]

Tetranitromethane. Forms highly explosive mixture with toluene.[6]

Uranium Hexafluoride. Vigorous interaction with toluene.[7]

Physiological Properties and Health Hazards

Vapor irritates the eyes and mucous membranes, and may cause dizziness, headache, nausea, and mental confusion. The liquid irritates the eyes and mucous membranes and may cause dermatitis. Swallowing or absorption through the skin would cause poisoning. Avoid breathing vapor. Avoid contact with skin and eyes.[8] TLV-TWA 50 ppm (188 mg/m^3).[9]

Spillage Disposal

Shut off all possible sources of ignition. Wear eye protection, laboratory coat, and Viton® gloves.[10] Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Transport to fume hood for evaporation. Ventilate spill site well to evaporate remaining liquid and dispel vapor.[9–11]

Waste Disposal

Place in nonhalogenated solvent disposal container for recycling or disposal by burning. Incineration will be easier by mixing with a more flammable solvent.[12]

REFERENCES

1. Merck 9607.
2. NFPA 170.

3. Simons, J.H., *Inorg. Synth.*, 3, 185, 1950.
4. Mellor, Vol. 8, Suppl. 2.2, 264, 1967.
5. B 1165.
6. B 189.
7. B 1126.
8. Lux 610.
9. ACGIH 57.
10. LSS.
11. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
12. ITI 648.

p-TOLUENESULFONIC ACID $CH_3C_6H_4SO_3H$

HIGHLY TOXIC

Physical Properties

Colorless monoclinic leaflets or prisms; crystalline solid; mp (anhydrous), 106–107°C; bp_{20}, 140°C.[1,2]

Chemical Properties

Soluble in water, alcohol, and ether.[1] Hygroscopic.[2]

Hazardous Reactions

When heated to decomposition, emits highly toxic fumes of sulfur dioxide.[3]

Acetic Acid, Acetic Anhydride, and Water. Explosions can occur when using an analytical method involving sequential addition of acetic acid, aqueous *p*-toluenesulfonic acid, and acetic anhydride to serum.[4]

Physiological Properties and Health Hazards

Irritating to the skin, eyes, and mucous membranes.[2] Highly toxic if swallowed. Animal experiments show moderate systemic toxicity and high irritation.[3]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Scoop into a pail of cold water. Neutralize with sodium carbonate and wash into the drain.[2]

Waste Disposal

Dissolve in a hydrocarbon solvent and burn in an incinerator equipped with afterburner and scrubber.[2]

Reactions for Spillage and Waste Disposal

$$2CH_3C_6H_4SO_3H + Na_2CO_3 \rightarrow 2CH_3C_6H_4SO_3Na + CO_2 + H_2O$$

REFERENCES

1. Merck 9611.
2. Ald 3366D.
3. Sax 3314.
4. B 452.
5. Aldrich 1361, 1474.

p-TOLUENESULFONYL CHLORIDE $CH_3C_6H_4SO_2Cl$

CORROSIVE

Physical Properties

Crystals; mp, 69–71°C; bp_{15}, 146°C.[1]

Chemical Properties

Freely soluble in alcohol, benzene, and ether; insoluble in water.[1] Moisture sensitive.[2]

Physiological Properties and Health Hazards

Corrosive.[2]

Spillage Disposal

Wearing eye protection, laboratory coat, and nitrile rubber gloves, scoop the solid into a container. Follow waste disposal procedure. Wash the spill site thoroughly with soap and water.

Waste Disposal

Package Lots. Place in a separate container labeled for disposal by burning. Dissolve in an appropriate solvent and burn in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Add 60 mL of 2.5 M sodium hydroxide (prepared by carefully dissolving 6 g of NaOH in 60 mL of cold water) to a 100-mL, three-necked, round-bottom flask equipped with a stirrer and thermometer, placed on a steam bath. The *p*-toluenesulfonyl chloride (0.05 mol, 9.5 g) is added in small portions. If the reaction is sluggish (no dissolution or rise in temperature), heat the mixture to about 90°C. When the initially added material has dissolved, add the remainder in small portions. After the addition is complete, continue heating until a clear solution is obtained. Cool the mixture to room temperature, neutralize with dilute hydrochloric or sulfuric acid, and then wash into the drain.[3]

Reactions for Spillage and Waste Disposal

$$CH_3C_6H_4SO_2Cl + NaOH \rightarrow CH_3C_6H_4SO_3Na + HCl$$

REFERENCES

1. Merck 9612.
2. Ald 3367C.
3. PPL 161.

TRIALLATE $C_{10}H_{16}Cl_3NOS$

HERBICIDE

Synonyms

Avadex BW®, Far-Go®, S-2,3,3-trichloroallyl diisopropylthiocarbamate.

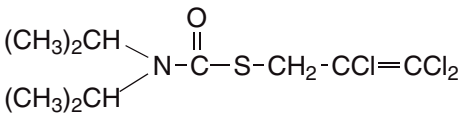
Physical Properties

Colorless crystals or amber liquid; bp, 148–149°C(9 mm); mp, 29–30°C.[1]

Chemical Properties

Soluble in acetone, benzene, ethanol, and heptane. Solubility in water, 4 mg/L.[1,2]

Structure



Physiological Properties and Health Hazards

LD_{50} (oral, rat) 1100 mg/kg[1]; LC_{50} (rainbow trout) 1.2 mg/L.[2]

Formulations

Emulsifiable concentrate, liquid, and granular.[1,2]

Spillage Disposal

Wear gloves and eye protection. The granular formulation may be swept up and discarded as for waste disposal. If liquid is spilled, cover with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a pail of cold water. Slowly and carefully add 2 M sulfuric acid (11 mL of concentrated acid added to 90 mL of water) until the solution has a pH of 1. Foaming may occur during the addition. Estimate the quantity of Triallate in the spilled material, and for each 1 g, add 2.5 g of potassium permanganate. Stir until the permanganate is dissolved and leave at room temperature overnight. If the purple color of the solution disappears, add another 2.55 portion of potassium permanganate, stir to dissolve, and leave overnight. Reduce the excess permanganate by adding solid sodium bisulfite until no purple color remains. Neutralize the solution with 10% sodium hydroxide solution and decant the liquid into the drain. Discard the solid as regular refuse.[3]

Waste Disposal

Wear gloves and eye protection. This waste disposal procedure is for Triallate in the form of granules containing 10% of the active ingredient. To 0.3 g of the formulation, add 50 mL of 2 M sulfuric acid (5.5 mL of concentrated sulfuric acid added to 45 mL of water) containing 0.5 g of potassium permanganate. Stir the mixture at room temperature for 24 hours. If the purple color of the solution disappears, add another 0.5-g portion of potassium permanganate and continue stirring for another 24 hours. Reduce the excess permanganate by adding solid sodium bisulfite, and neutralize the

solution with 10% sodium hydroxide solution. Pour the liquid into the drain, and discard any residual brown precipitate of manganese dioxide in regular refuse.[3]

Decontamination of Contaminated Clothing

Protect your hands; handle contaminated clothing with unlined nitrile or neoprene gloves. Store contaminated work clothing separately in a disposable plastic bag until ready to wash; launder contaminated work clothing daily. Do not wash contaminated clothing with the regular family wash; launder alone to avoid the transfer of pesticide to other clothes. Use the recommended amount of heavy-duty detergent; for heavily soiled clothing, use 1.5 times the recommended amount of detergent. Use the high water level and a 12-minute wash. After washing, hang to dry; to avoid dryer contamination, do not use a dryer. Clean the washer by running the machine through a complete wash cycle with detergent (but without clothing) before washing other clothes.

Emulsifiable concentrate formulation of Triallate, diluted to field strength concentration (1.8% a.i.), was used to contaminate cotton twill fabric. The best laundry procedure is to pretreat the work clothing with a pre-wash commercial soil and stain remover containing a blend of surfactants and solvents, launder in hot water (60°C), and then launder a second time. Approximately 18% of the pesticide remains in the fabric using the above procedure; after a single wash when no pretreatment and warm water (50°C) were used, as much as 48% of the Triallate remained.

Do not wash clothing that has been contaminated with concentrated Triallate. The clothing should be placed in a plastic bag and taken to an approved landfill site.[4,5]

REFERENCES

1. Agro. Handbook, A0403; Merck 9669.
2. Agro. Desk Ref., 409.
3. Armour, M.A., et al., Treatment of spills and small quantities of waste pesicides, *Proceedings of the 3rd Asian Conference on Academic Activity for Waste Treatment*, Bangkok, Thailand, 1996, p. 53.
4. Armour, M.A., et al., Decontamination of spills and residues of pesticides and protective clothing worn during their handling, *Proceedings of the Pacific Basin Conference on Hazardous Waste*, Kuala Lumpur, Malaysia, 1996, p. 44.
5. Rigakis, K.B., Martin-Scott, S., Crown, E.M., Kerr, N., and Eggerston, B., *Agric. For. Bull.*, 10, 24, 1987.

TRIBUTYLPHOSPHINE $(C_4H_9)_3P$

PYROPHORIC, CORROSIVE, LACHRYMATOR

Physical Properties

Colorless liquid with garlic odor; bp_{50}, 240°C.[1]

Fire Hazard

Flammable; flash point, 37°C[2]; auto-ignition temperature, 200°C.[3]

Chemical Properties

Almost insoluble in water; miscible with ether, methanol, ethanol, and benzene.[1]

Physiological Properties and Health Hazards

Corrosive; lachrymator.[2] Acute toxic data: LD_{50} (oral, rat) 750 mg/kg.[3]

Spillage Disposal

Wear eye protection, laboratory coat, and butyl rubber gloves. Cover liquid with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container and transport to the fume hood. Slowly add the solid to laundry bleach (66 mL per 1 g of tributylphosphine). Decant the liquid into the drain and package the solid for disposal by burning.[4,5]

Waste Disposal

Package Lots. Package securely in a suitable container labeled for disposal by burning. Burn in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. Add tributylphosphine (10.1 g, 0.05 mol) dropwise while stirring to a 25% excess of hypochlorite (670 mL of laundry bleach or 55 g calcium hypochlorite in 200 mL of water). The temperature of the reaction should be monitored. After addition is complete and the reaction has subsided, filter the solid and package it for disposal by burning. Wash the aqueous solution into the drain.[5]

Reactions for Spillage and Waste Disposal

$$(C_4H_9)_3P + 4NaOCl + 3H_2O \rightarrow H_3PO_4 + 3C_4H_9OH + 4NaCl$$

REFERENCES

1. Sax 5, 1042.
2. Ald 3398C.
3. B 1850.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
5. PP 95.

TRICHLOROSILANE $SiHCl_3$

FLAMMABLE

Physical Properties

Colorless,[1] volatile, mobile liquid; mp, –126.5°C; bp, 31.8°C.[2]

Fire Hazard

Supports combustion.[2] Flash point, –28°C; auto-ignition temperature, 104°C.[3]

Chemical Properties

Fumes in air. Decomposed by water. Soluble in benzene, carbon disulfide, chloroform, and carbon tetrachloride.[2]

Hazardous Reactions

Technical material is ignitable (possibly due to presence of dichlorosilane).[4]

Oxidizing Materials. Can react vigorously on contact with oxidizing materials.[1]

Water. Will react with water or steam to produce heat and toxic and corrosive fumes.[1]

Physiological Properties and Health Hazards

Irritating to the mucous membranes and skin.[1] Corrosive.[5]

Spillage Disposal

Wear butyl rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and transport to the fume hood. Slowly add the solid to a pail of cold water. If necessary, neutralize the solution with sodium carbonate. Decant the solution into the drain. Treat the solid as normal refuse.[6]

Waste Disposal

Package Lots. Where possible, the compound should be redistilled or otherwise repurified and reused.

Small Quantities. Wear eye protection, laboratory coat, and butyl rubber gloves. Work in the fume hood. To dispose of small quantities, equip a 1-L, three-necked, round-bottom flask with a dropping funnel, stirrer, and thermometer. Place 500 mL of water in the flask and cool it in an ice-water bath. Place 10 mL of trichlorosilane in the dropping funnel and add dropwise to the stirred water. Do not allow the temperature to rise above 20°C. When addition is complete, neutralize the solution with sodium carbonate and wash into the drain.[6]

Reactions for Spillage and Waste Disposal

$$SiHCl_3 + 3H_2O \rightarrow HSi(OH)_3 + 3HCl$$

REFERENCES

1. Sax 3361.
2. Merck 9719.

3. B 1850.
4. Mueller, R. et al., *J. Prakt. Chem.*, 31, 1, 1966.
5. Ald 3421A.
6. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.

TRIETHYLALUMINUM $Al(C_2H_5)_3$

PYROPHORIC, VIOLENT REACTION WITH WATER

Physical Properties

Colorless volatile liquid; bp, 194°C.[1]

Fire Hazard

Extremely pyrophoric.[2] Flash point, below –53°C; auto-ignition temperature, below –53°C.[3] Use carbon dioxide or dry chemical fire extinguisher. Do not use water.[4]

Chemical Properties

Moisture sensitive.[2]

Hazardous Reactions

Ignites in air.[5]

Alcohols. Explosive reaction with methanol, ethanol, and 2-propanol. Vigorous reaction with *tert*-butanol.[5]

N,N-Dimethylformamide. Mixture explodes when heated.[6]

Halogenated Hydrocarbons. Violent reactions may occur when aluminum alkyls are exposed to halogenated hydrocarbons.[7]

Water. Triethylaluminum explodes violently in water.[7]

Physiological Properties and Health Hazards

Irritant to the skin, eyes, and mucous membranes. Harmful by inhalation, ingestion, or absorption through the skin.[8] TLV-TWA (as Al) 2 mg/m^3.[9]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container and transport to the fume hood. Slowly add the solid to a large beaker of butanol. When reaction has ceased, decant the liquid to the drain. Treat the solid as normal refuse.[10]

Waste Disposal

Package Lots. Package for burning. Dissolve in an appropriate solvent and incinerate.[11]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Place a solution of the triethylaluminum (dissolved under nitrogen in a dry hydrocarbon solvent at less than 5% concentration) in a hydrocarbon or an ether in a three-necked, round-bottom flask equipped with a stirrer, dropping funnel, nitrogen inlet, and ice bath. Dissolve a 10% excess of *tert*-butyl alcohol (3 mL alcohol/g alkylaluminum) in a hydrocarbon (5% solution) by adding dropwise to the well-stirred solution under nitrogen. Follow with addition of cold water, and then enough 5% hydrochloric acid to neutralize the aqueous phase. The aqueous and organic phases are separated and the organic phase is packaged and sent for disposal by burning. The aqueous phase is washed into the drain.[11]

Reactions for Spillage and Waste Disposal

$$Al(C_2H_5)_3 + 3(CH_3)_3COH \rightarrow Al(OC(CH_3)_3)_3 + 3C_2H_6$$

$$Al(OC(CH_3)_3)_3 + 3H_2O \rightarrow Al(OH)_3 + 3(CH_3)_3COH$$

REFERENCES

1. Merck 324.
2. Ald 3428A.
3. B 1847.
4. NFPA 49.
5. Mirviss, S.B. et al., *Ind. Eng. Chem.*, 53, 54A, 1961.
6. Dimethylformamide Brochure, Imperial Chemical Industries, Billingham, UK, 1965.
7. NFPA 491M.
8. Lux 622.
9. ACGIH 14.
10. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
11. ITI 669.
12. PP 95.

TRIETHYLPHOSPHINE $(C_2H_5)_3P$

PYROPHORIC

Physical Properties

Colorless liquid; odor of hyacinths; bp, 127–128°C.[1] Isopropyl alcohol solution (67% by weight) has strong odor.[2]

Fire Hazard

Pyrophoric.[2] Ignites spontaneously on contact with oxidizable material.[3]

Chemical Properties

Practically insoluble in water; miscible with alcohol and ether.[1]

Hazardous Reactions

Oxygen. Action of oxygen at low temperature on triethylphosphine produces an explosive product.[4]

Physiological Properties and Health Hazards

Probably highly toxic.[4]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover liquid with a 1:1:1 mixture by weight of sodium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into a container and transport to the fume hood. Slowly add the solid to laundry bleach (contains about 5% sodium hypochlorite; 75 mL per 1 g of triethylphosphine). Maintain a temperature of 45–50°C during addition, and then stir for another 2 hours. Decant the liquid into the drain. Package the solid for disposal by burning.[5,6]

Waste Disposal

Package Lots. Place in an appropriate container labeled for disposal by burning. Burn in a furnace equipped with afterburner and scrubber.[2]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Add triethylphosphine (9.0 g, 0.05 mol) dropwise while stirring to a 25% excess of hypochlorite (670 mL of laundry bleach or 55 g of calcium hypochlorite in 200 mL of water) in a three-necked, round-bottom flask equipped with a stirrer, dropping funnel, and thermometer. The temperature of the reaction should be maintained at 45–50°C during addition. After addition is complete, stir for 2 hours. The solid is filtered and packaged for disposal by burning. The aqueous solution is washed into the drain.[6]

Reactions for Spillage and Waste Disposal

$$(C_2H_5)_3P + 4NaOCl + 3H_2O \rightarrow H_3PO_4 + 3C_2H_5OH + 4NaCl$$

REFERENCES

1. Merck 9748.
2. Ald 3435C.

3. B 667.
4. Sax 3374.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
6. PP 95.

TRIFLUOROACETIC ACID CF_3COOH
and TRIFLUOROACETIC ANHYDRIDE $(CF_3CO)_2O$

SERIOUS RISK OF POISONING BY INHALATION, SWALLOWING, OR SKIN CONTACT; CAUSES SEVERE BURNS

Physical Properties

Colorless liquids with pungent odor; bp, 72°C and 40°C, respectively.[1]

Chemical Properties

The acid is miscible with water. The anhydride reacts with water, forming the acid.[1]

Hazardous Reactions

Lithium Aluminum Hydride. Reacts violently with trifluoroacetic acid.[2]

Dimethyl Sulfoxide. Reacts explosively with trifluoroacetic anhydride.[3]

Physiological Properties and Health Hazards

The vapor irritates the eyes and respiratory system. The liquid burns the eyes and quickly penetrates the skin to cause deep-seated burns. Assumed to cause severe burning and damage if taken by mouth. There is no reported toxic effect due to the presence of fluorine, as in the case of highly toxic fluoroacetic acid. Avoid breathing vapor. Prevent contact with skin and eyes.[1]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, goggles, a face shield, and, if necessary, a self-contained breathing apparatus. Cover the spill with a 1:1:1 mixture by weight of soda ash or calcium carbonate, clay cat litter (bentonite), and sand. When the trifluoroacetic acid (anhydride) has been absorbed, scoop the mixture into an appropriately labeled container for recycling or disposal by burning.[4–6]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning.[5,6]

REFERENCES

1. Lux 626; Merck 9754.
2. Karo, W., *Chem. Eng. News*, 33, 1368, 1955.
3. Sharma, A.K. et al., *Tetrahedron Lett.*, 1503–1506, 1974.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
5. Ald 3439A.
6. PP 60.

TRIISOBUTYLALUMINUM $Al(i\text{-}C_4H_9)_3$

PYROPHORIC, VIOLENT REACTION WITH WATER

Synonyms

TIBAL.

Physical Properties

Colorless flammable liquid; mp, 6°C; bp_{10}, 86°C.[1]

Fire Hazard

Undiluted material ignites in air; flash point, below 0°C; auto-ignition temperature, below 4°C.[2] Use carbon dioxide or dry chemical fire extinguisher.[3]

Chemical Properties

Soluble in saturated hydrocarbons. Solutions stable at normal handling temperatures. Decomposes at above 50°C when undiluted.[2] Stereospecific reducing agent.[1]

Hazardous Reactions

Powerful reductant supplied as hydrocarbon solution.[2] Violent reaction with acids, air, alcohols, amines, and water.[3]

Physiological Properties and Health Hazards

Extremely destructive to living tissue.[4] TLV-TWA (as Al) 2 mg/m^3.

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, goggles, and a face shield. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Slowly add the solid to a large beaker of butanol. When reaction has ceased, decant the solution to the drain. Treat the solid as normal refuse.[5]

Waste Disposal

Package Lots. Package for disposal by burning.[6]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. A solution of the triisobutylaluminum (dissolved under nitrogen in dry hydrocarbon solvent at less than 5% concentration) is placed in a three-necked, round-bottom flask equipped with a stirrer, dropping funnel, nitrogen inlet, and ice bath. A 10% excess of *tert*-butyl alcohol (2 mL alcohol/g of alkylaluminum) dissolved in a hydrocarbon (approximately 5% solution) is added dropwise to the well-stirred solution under nitrogen. This is followed by addition of cold water and then enough 5% hydrochloric acid to neutralize the aqueous phase. The aqueous and organic phases are separated and the organic phase is packaged and sent for disposal by burning. The aqueous phase is washed into the drain.[7]

Reactions for Spillage and Waste Disposal

$$Al(i\text{-}C_4H_9)_3 + 3(CH_3)_3COH \rightarrow Al(O(CH_3)_3) + 3i\text{-}C_4H_{10}$$

$$Al(O(CH_3)_3) + H_2O \rightarrow \underset{\substack{\text{aluminum}\\\text{hydroxide}\\\text{(insoluble)}}}{Al(OH)_3} + (CH_3)_3COH$$

REFERENCES

1. Merck 324.
2. B 1850.
3. NFPA 49.
4. Sax 3391; ACGIH 14.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
6. ITI 673.
7. PP 95.

TRIPHENYLPHOSPHINE $(C_6H_5)_3P$

REACTS VIGOROUSLY WITH OXIDIZING MATERIALS

Physical Properties

Odorless monoclinic platelets or prisms; mp, 80.5°C; bp, 360°C (in inert gas).[1]

Fire Hazard

Combustible when exposed to heat or flame. Flash point, 180°C.[2]

Chemical Properties

Triboluminescent; soluble in ether, benzene, chloroform, and glacial acetic acid; less soluble in alcohol; practically insoluble in water.[1]

Hazardous Reactions

Chlorobenzene and Phosphorus Trichloride. In the preparation of triphenylphosphine from chlorobenzene, phosphorus trichloride, and sodium dispersed in toluene or xylene, explosions may be avoided by adding about 1 mol of a lower alcohol per 100g of sodium used.[3]

Oxidizing materials. Can react vigorously with oxidizing materials.[2]

Physiological Properties and Health Hazards

Irritant.[4] Acute toxic data: LD_{50} (oral, rat) 800 mg/kg; LC_{50} (inhalation, rat) 1135 ppm for 4 hours.[2]

Spillage Disposal

Wear eye protection, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop the solid into an appropriate labeled container for disposal by burning.[4,5]

Waste Disposal

Package Lots. Package securely and send for disposal by burning in a furnace equipped with afterburner and scrubber.[4]

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. Work in the fume hood. Triphenylphosphine (1.3 g, 0.005 mol) is dissolved in tetrahydrofuran (30 mL). The solution is slowly added while stirring to household bleach (70 mL) or a calcium hypochlorite solution (5.5 g in 20 mL of water). After stirring for 1 hour, the solution is placed in an open container in the fume hood to allow the tetrahydrofuran to evaporate. The aqueous solution is washed into the drain.[6]

Reactions for Spillage and Waste Disposal

$$(C_6H_5)_3P + 4NaOCl + 3H_2O \rightarrow \underset{\text{phosphoric acid}}{H_3PO_4} + \underset{\text{phenol}}{3C_6H_5OH} + 4NaCl$$

REFERENCES

1. Merck 9814.
2. Sax 3431.

3. B 890, 1372.
4. Ald 3519D.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ*., 62, A93, 1985; Armour, M.A., *J. Chem. Educ*., 65, A64, 1988; *Haz. Mat. Spills Tec*., 29.1.
6. PP 95.

URETHAN $H_2NCO_2C_2H_5$

ANIMAL CARCINOGEN

Synonyms

Carbamic acid ethyl ester, ethyl carbamate, ethyl urethan.

Physical Properties

Colorless, odorless crystal[1]; cooling saline taste; mp, 48–50°C; bp, 182–189°C; sublimes readily at 103°C and 54 mm pressure.[2]

Chemical Properties

One gram is soluble in 0.5 mL of water, 0.8 mL of alcohol, 0.9 mL of chloroform, 1.5 mL of ether, 2.5 mL of glycerol, and 32 mL of olive oil.[2]

Hazardous Reactions

Emits toxic fumes when heated.[1]

Physiological Properties and Health Hazards

Animal carcinogen. Reasonably anticipated to be a human carcinogen.[3] Causes depression of bone marrow and occasionally focal degeneration in the brain. Can produce central nervous system depression, nausea, and vomiting.[1] LD_{50} (i.p., rat) 1500 mg/kg; LD_{50} (oral, mouse) 2500 kg/kg (s.c., mouse) 1750 mg/kg.[1]

Spillage Disposal

Wear a dust mask, rubber gloves, protective clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a beaker and transport to a fume hood. Estimate the quantity of urethan spilled and for each 1 g, add at least 100 mL of household bleach (5% sodium hypochlorite). Allow to stand overnight; decant the supernatant liquid into the drain with water and discard the solid residue as normal refuse.[4,5]

Waste Disposal

Package Lots. Place in a separate labeled container for disposal by burning. This combustible material should be packaged and labeled for burning in a furnace equipped with afterburner and scrubber.[6]

Small Quantities. To urethan (1 g) contained in a 125-mL Erlenmeyer flask equipped with a magnetic stirrer, slowly and cautiously add 100 mL of household bleach (5% sodium hypochlorite). Stir for 2 hours. Wash the solution into the drain with water.[5]

REFERENCES

1. Sax 3471.
2. Merck 9942.
3. NIEHS III–214.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.1.
5. Armour, M.A. and Renecker, D.M., *Laboratory Disposal Method for Urethan,* University of Alberta, Edmonton, 1996..
6. Ald 3580D.

VINBLASTINE SULFATE $C_{46}H_{58}N_4O_9 \cdot H_2SO_4$

ANTINEOPLASTIC

Synonyms

Velbe, Velban, Exal, Vincaleukoblastine sulfate.

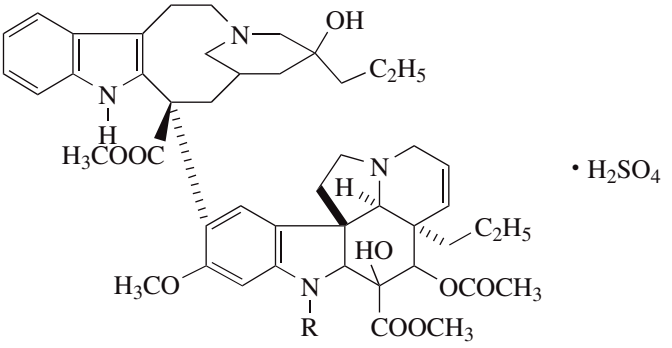
Physical Properties

White to slightly yellow in color, odorless, hygroscopic, amorphous or crystalline powder; mp, 284–285°C (dec).[1]

Chemical Properties

Soluble in water (1 in 10), ethanol (1 in 12200), and chloroform and methanol (1 in 50). Insoluble in ether.[1]

Structure



R = CH_3

Mode of Action

Vinblastine disrupts mitosis of the cell by interfering with microtubular proteins.[2]

Physiological Properties and Health Hazards

Adverse effects in patients include nausea, vomiting, anorexia, diarrhea, abdominal pain, pharyngitis, stomatitis, enterocolitis, rectal bleeding, blood anomalies, paresthesias, peripheral neuritis, mental depression, loss of tendon reflexes, headache, and convulsions.[2]

Spillage Disposal

Solutions. Wear protective gloves, clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Wash the spill area with soap and water. Estimate the weight of absorbing mixture used and add to it a 3 M solution of sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate per 100 mL, allowing 20 mL of permanganate solution per 1 g of absorbing mixture. After standing at room temperature overnight, neutralize by careful addition of soda ash or 10% sodium hydroxide, decant the liquid into the drain with water, and discard the residue as normal refuse.[3–5]

Waste Disposal

Wear rubber gloves, protective clothing, and goggles. Work in the fume hood. To 10 mL of an aqueous solution containing 10 mg of vinblastine sulfate, add 10 mL of 6 M sulfuric acid (3.4 mL of concentrated sulfuric acid slowly added to 6.6 mL of cold water). Add potassium permanganate (1 g). Continue stirring for 2 hours. Neutralize the solution by the slow addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite (approximately 1 g of sodium bisulfite per 3.5 mL of water) until a colorless liquid is formed. Wash into the drain with water.[3,4]

Acid Hydrolysis. To 10 mL of aqueous solution containing 10 mg of vinblastine sulfate, add 40 mL of 50% of sulfuric acid (20 mL of concentrated sulfuric acid slowly added to 20 mL of cold water). Boil the mixture gently for 2 hours. When hydrolysis is complete, the solution, at room temperature, is neutralized by the careful addition of 10% sodium hydroxide, and then washed into the drain with water.[4,5]

REFERENCES

1. Martindale, W., *The Extra Pharmacopoeia,* 27th ed., edited by A. Wade, Pharmaceutical Press, London, 1977.
2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, MA, 1982.
3. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 75.
4. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 103.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

VINCRISTINE SULFATE $C_{46}H_{56}N_4O_{10}\cdot H_2SO_4$

ANTINEOPLASTIC

Synonyms

Kyocristine, Oncovin, Leurocristine, LCR, VCR.

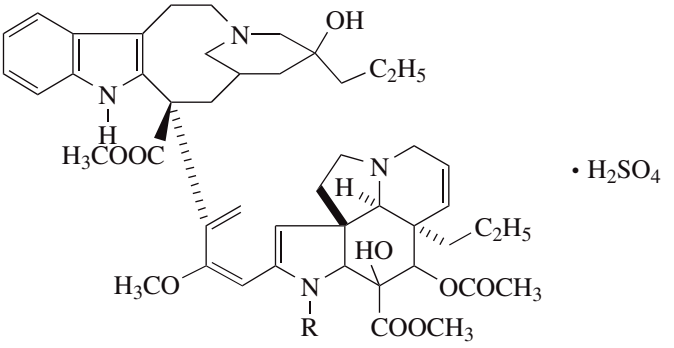
Physical Properties

White to slightly yellow, very hygroscopic, amorphous or crystalline powder[1]; mp, 273–281°C (after recrystallization from absolute ethanol).[2]

Chemical Properties

Soluble in water (1 in 2), ethanol (1 in 600), and chloroform and methanol (1 in 30). Insoluble in ether.[1]

Structure



R = CHO

Mode of Action

Vincristine is thought to disrupt cell replication by interfering with microtubular proteins.[3]

Physiological Properties and Health Hazards

Causes neurotoxicity, autonomic and central nervous system toxicity, alopecia, mild leukopenia, mild anemia, and mild thrombocytopenia, as well as nausea, vomiting, diarrhea, constipation, stomatitis, and oral ulceration.[3]

Spillage Disposal

Solutions. Wear protective gloves, clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a container and transport to the fume hood. Wash the spill area with soap and water. Estimate the weight of absorbing mixture used and add to it a 3 M solution of sulfuric acid (17 mL of concentrated sulfuric acid slowly added to 83 mL of cold water) containing 4.7 g of potassium permanganate per 100 mL. Allow 20 mL of permanganate solution per gram of mixture. After standing at room temperature overnight, neutralize by careful addition of soda ash or 10% sodium hydroxide, decant the liquid into the drain with water, and discard the residue as normal refuse.[2,4,5]

Waste Disposal

Wear rubber gloves, protective clothing, and goggles. Work in the fume hood. To 10 mL of an aqueous solution containing 10 mg of vincristine sulfate, add 10 mL of 6 M sulfuric acid (3.4 mL of concentrated sulfuric acid slowly added to 6.6 mL of cold water). Slowly, while stirring, add potassium permanganate (1 g). Continue stirring for 2 hours. Neutralize the solution by the slow addition of soda ash or 10% sodium hydroxide solution, and then add, while stirring, a saturated aqueous solution of sodium bisulfite (approximately 1 g in 3.5 mL of water) until a colorless liquid is formed. Wash into the drain with water.[2,4]

Acid hydrolysis. To 10 mL of an aqueous solution containing 10 mg of vincristine sulfate in a 100-mL beaker, add 40 mL of 50% sulfuric acid (20 mL of concentrated sulfuric acid is slowly added to 20 mL of cold water). Cover the beaker with a watch glass, and gently boil the mixture on a hot plate for 2 hours. After cooling, the solution is neutralized by slow addition of soda ash or 10% sodium hydroxide solution. The liquid is washed into the drain with water.[3,4]

REFERENCES

1. Merck 10048; Martindale, W., *The Extra Pharmacopoeia*, 27th ed., edited by A. Wade, Pharmaceutical Press, London, 1977.
2. Castegnaro, M. et al., *Laboratory Decontamination and Destruction of Carcinogens in Laboratory Wastes: Some Antineoplastic Agents*, No. 73, IARC Scientific Publications, Lyon, 1985, p. 126.
3. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, MA, 1982.
4. Armour, M.A., Laboratory methods for disposal of toxic inorganic and organic chemicals, in *Hazardous Waste Control in Research and Education*, Korenaga, T. et al., Eds., Lewis Publishers, Boca Raton, FL, 1994, p. 103.
5. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

VINDESINE SULFATE $C_{43}H_{57}N_5O_{11}S$

ANTINEOPLASTIC

Synonyms

Eldisine, LY 099094.

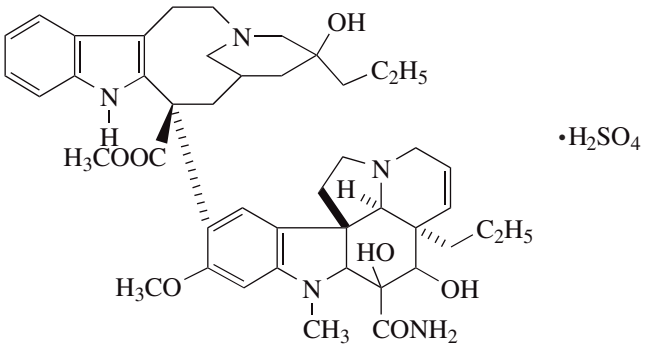
Physical Properties

Amorphous solid from ethanol-isopropyl alcohol; mp, above 250°C.[1]

Mode of Action

The drug is thought to disrupt cell mitosis by interfering with microtubular proteins.[2]

Structure



Physiological Properties and Health Hazards

Adverse effects include nausea, vomiting, alopecia, lethargy, neurologic toxicities, loss of deep tendon reflexes, and hallucinations.[2] LD_{50} (i.v., mouse) 6.3 ± 0.6 mg/kg; LD_{50} (i.v., rat) 2.0 ± 0.2 mg/kg.[1]

Spillage Disposal

Solutions. Wear protective gloves, clothing, and goggles. Cover the spill with a 1:1:1 mixture by weight of soda ash, clay cat litter (bentonite), and sand. Scoop the mixture into a beaker and transport to the fume hood. Slowly and while stirring, add 50% sulfuric acid (5 mL of concentrated sulfuric acid slowly added to 5 mL of cold water), allowing 10 mL of acid for each 1 g of absorbing mixture used. Place the beaker on a hotplate and boil gently for 2 hours. Cool, and neutralize the solution by careful addition of soda ash or 10% sodium hydroxide solution. Decant the liquid into the drain with water and discard the solid as normal refuse.[3,4]

Waste Disposal

Wear rubber gloves, protective clothing, and goggles. Work in the fume hood. To 5 mL of an aqueous solution containing 5 mg of vindescine sulfate in a 100-mL beaker, add 20 mL of 50% sulfuric acid (10 mL of concentrated sulfuric acid slowly added to 10 mL of cold water; caution: highly corrosive liquid). Cover the beaker with a watch glass and gently boil the mixture on a hot plate for 2 hours. Cool, neutralize the solution by slow addition of soda ash or 10% sodium

hydroxide solution, and then, while stirring, add a saturated aqueous solution of sodium bisulfite (approximately 1 g in 3.5 mL of water) until a colorless solution is formed. The liquid is washed into the drain with water.[3]

REFERENCES

1. Merck 10049.
2. Preston, J.D. et al., *Cancer Chemotherapeutic Agents: Handbook of Clinical Data*, 2nd ed., G.K. Hall Medical Publishers, Boston, MA, 1982.
3. Armour, M.A., Ayer, K., McKenzie, P., *Laboratory Disposal Method for Vindesine Sulfate,* University of Alberta, Edmonton, 1991.
4. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.7.

VINYL BROMIDE CH_2=CHBr

SUSPECTED CANCER AGENT, EXTREMELY FLAMMABLE, VERY TOXIC BY INHALATION

Synonyms

Bromoethylene.

Physical Properties

Colorless liquid or gas; bp, 16°C.[1]

Fire Hazard

Flammable. Flash point, below –8°C. Extinguish fire with water spray, foam, dry powder, or carbon dioxide.[1]

Chemical Properties

Insoluble in water.[1]

Hazardous Reactions

Oxidants. The highly flammable gas reacts violently with oxidants.
Sunlight. Vinyl bromide may polymerize in sunlight.[2]

Physiological Properties and Health Hazards

Inhalation of vapor in high concentrations may produce dizziness and narcosis. The liquid irritates the eyes and may irritate the skin by its defatting action; it is assumed to be harmful if taken by mouth. In view of the recent observation that vinyl chloride can cause liver cancer, it must be assumed that vinyl bromide may behave similarly. Prevent inhalation of vapor. Prevent contact with skin and eyes.[1] TLV-TWA 0.5 ppm (2.2 mg/m^3).[3]

Spillage Disposal

Evacuate area and allow to evaporate.[1]

Waste Disposal

Send unopened labeled container for recycling or disposal by burning.[4]

REFERENCES

1. Lux 640.
2. B 243.
3. ACGIH 60.
4. ITI 686; PP 60.

VINYL CHLORIDE $CH_2=CHCl$

SUSPECTED CANCER AGENT, EXTREMELY FLAMMABLE, VERY TOXIC BY INHALATION, DANGER OF VERY SERIOUS IRREVERSIBLE EFFECTS

Synonyms

Chloroethylene.

Physical Properties

Colorless gas with pleasant sweet odor; bp, –14°C.[1]

Fire Hazard

Flammable. Flash point, –78°C; explosive limits, 4–33%; ignition temperature, 472°C. Because the gas is supplied in a cylinder, turning off the valve will reduce any fire involving it; if possible, cylinders should be removed quickly from an area in which a fire has developed. Fight fire with water, dry chemical, or carbon dioxide.[2]

Chemical Properties

Slightly soluble in water; soluble in alcohol, ether, carbon tetrachloride, and benzene. Polymerizes in light or in the presence of a catalyst.[1]

Hazardous Reactions

Discharge of a spray of vapor and liquid under pressure from a cylinder into a fume hood caused ignition of the vapor due to static electricity. Discharge of the gas only did not cause static build-up.

Traces of peroxide can cause explosive polymerization.[3]

Nitrogen Oxides. An explosion in a valve in a liquid monomer line was ascribed to traces of nitrogen oxides remaining after the valve had been passivated by treatment with nitric acid.

Oxygen. The stability of the monomer to heat and the resultant intensity of the isotherm at 90–100°C depends on the partial pressure of oxygen.[3]

Physiological Properties and Health Hazards

Inhalation of vapor in high concentrations produces dizziness and narcosis. The liquid may irritate and burn the skin, the latter due to its freezing action. As a result of observations made in the U.S. (1974), exposure to vinyl chloride monomer (VCM) in the workplace has been shown to cause a rare liver cancer, angiosarcoma. This may not manifest itself until more than 20 years after initial exposure. Prevent inhalation of gas. Prevent contact with liquid.[4] TLV-TWA 1 ppm.[5] Listed as known human carcinogen.[6]

Spillage Disposal

Surplus gas or leaking cylinder can be burned off through a suitable burner in a fume hood.[4]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning.[4]

REFERENCES

1. Merck 10055.
2. NFPA 49.
3. B 244.
4. Lux 641.
5. ACGIH 60.
6. NIEHS III–61.

VINYLIDENE CHLORIDE $CH_2=CCl_2$

EXTREMELY FLAMMABLE, MAY FORM EXPLOSIVE PEROXIDES, HARMFUL VAPOR

Synonyms

1,1-Dichloroethylene.

Physical Properties

Colorless, volatile liquid with chloroform-like odor; bp, 32°C.[1]

Fire Hazard

Flammable. Flash point, –18°C (closed cup); explosive limits, 7.3–16%; ignition temperature, 570°C. Extinguish fire with foam, dry chemical, or carbon dioxide. Water may be ineffective unless as spray.[2]

Chemical Properties

Almost insoluble in water; soluble in organic solvents. Above 0°C polymerizes rapidly.[1]

Hazardous Reactions

Air. In the absence of inhibitor (e.g., hindered phenol), rapidly absorbs oxygen from air, forming explosive peroxide.[3]

Chlorotrifluoroethylene. Violent polymerization can occur on heating at 180°C in an autoclave.[4]

Ozone. Reaction products with ozone are particularly dangerous.[5]

Physiological Properties and Health Hazards

Breathing vapor may cause drowsiness. Prolonged inhalation of small quantities may cause liver and kidney damage. Liquid irritates the skin and eyes. Swallowing would cause poisoning. Avoid breathing vapor. Avoid contact with skin and eyes.[6] TLV-TWA 5 ppm (20 mg/m^3).[7]

Spillage Disposal

Wear breathing apparatus, eye protection, laboratory coat, and butyl rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand.[8] Shovel into bucket and place in fume hood to evaporate, or dispose of by burning in a furnace equipped with afterburner and scrubber.[9]

Waste Disposal

Place in a separate labeled container for recycling or disposal by burning. The compound should be burned in a furnace equipped with afterburner and scrubber.[9]

REFERENCES

1. Merck 10057.
2. NFPA 49.
3. Reinhardt, R.C., *Chem. Eng. News*, 25, 2136, 1947.
4. Raasch, M.S. et al., *Org. Synth.*, 42, 46, 1962.

5. Dow Chemical Company, Vinylidene Chloride Monomer, 9, Dow Chemical Company, Midland, MI, 1966, p. B 232.
6. Lux 333.
7. ACGIH 60.
8. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
9. ITI 688.

XYLENES $C_6H_4(CH_3)_2$

HARMFUL VAPOR, FLAMMABLE

Synonyms

Xylols.

Physical Properties

Colorless liquids; bp, 144°C (*o*-), 139°C (*m*-), and 138°C (*p*-).[1]

Fire Hazard

Flammable. Flash point, 17°C (*o*-), and 25°C (*m*- and *p*-); explosive limits, approximately 1–7%; ignition temperature, 463°C (*o*-), 527°C (*m*-), and 528°C (*p*-). Extinguish fire with foam, dry powder, carbon dioxide, or vaporizing liquid.[2]

Chemical Properties

Immiscible with water; miscible with alcohol, ether, and many other organic solvents.[1]

Hazardous Reactions

Air and Acetic Acid. The oxidation of *p*-xylene in acetic acid to form terephthalic acid can result in an explosion.[3]

Nitric Acid. May detonate immediately on contact with concentrated nitric acid.[4]

Physiological Properties and Health Hazards

Vapor irritates the eyes and mucous membranes and may cause dizziness, headache, nausea, and mental confusion. Liquid irritates the eyes and mucous membranes. Swallowing or absorption through the skin would cause poisoning. Prolonged exposure to skin contact may result in dermatitis. Avoid breathing vapor. Avoid contact with skin and eyes.[5] TLV-TWA 100 ppm (434 mg/m^3); TLV-STEL 150 ppm (651 mg/m^3).[6]

Spillage Disposal

Shut off all possible sources of ignition. Wear face shield, goggles, laboratory coat, and nitrile rubber gloves. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand, and then shovel into bucket and transport to fume hood for atmospheric evaporation. Ventilate spill site well to evaporate remaining liquid and dispel vapor.[5,7]

Waste Disposal

Place waste in nonhalogenated solvent disposal container for disposal by burning.[8]

REFERENCES

1. Merck 10136.
2. NFPA 49.
3. B 748.
4. B 1165.
5. Lux 642.
6. ACGIH 61.

7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.
8. Ald 3618C, 3619A, 3619D; ITI 691–693.

ZIRCONIUM TETRACHLORIDE $ZrCl_4$

HIGHLY IRRITATING FUMES, MOISTURE SENSITIVE

Synonyms

Zirconium chloride.

Physical Properties

Lustrous crystals; sublimes at 331°C; mp_{25}, 437°C; extremely hygroscopic.[1]

Fire Hazard

Self-ignites in air.[2]

Chemical Properties

Fumes in moist air, forms hydrochloric acid vapor; soluble in water with extensive decomposition. Soluble in alcohol, ether, and concentrated hydrochloric acid.[3] Strong Lewis acid.[1]

Hazardous Reactions

Air. Forms HCl vapor in moist air.[1]

Ethanol. Violent reaction.[4]

Lithium. Lithium strip ignites in contact with $ZrCl_4$ in nitrogen atmosphere in glove box.[5]

Water. Violent reaction.[4]

Physiological Properties and Health Hazards

Fumes highly irritating to the eyes, skin, and respiratory system. TLV-TWA 5 mg/m^3.[6]

Spillage Disposal

Wear nitrile rubber gloves, laboratory coat, and eye protection. Cover spill with a 1:1:1 mixture by weight of sodium carbonate or calcium carbonate, clay cat litter (bentonite), and sand. Scoop into a container and remove to fume hood. Slowly add the solid mixture to a large volume of stirred water and crushed ice in a beaker. When the reaction is complete, neutralize with sodium carbonate or dilute hydrochloric acid. Decant the liquid portion to the drain with at least 50 times its volume of water. The solid residue may be sent to the landfill.[7,8]

Waste Disposal

Package Lots. Place in a separate container labeled for disposal by burning.

Small Quantities. Wear eye protection, laboratory coat, and nitrile rubber gloves. In a fume hood, add the zirconium tetrachloride in small portions to a large volume of stirred water and crushed ice in a beaker. When the reaction is complete, neutralize with sodium carbonate or dilute hydrochloric acid. The liquid portion of the reaction mixture may be washed into the drain, while the solid residue may be treated as normal refuse.[8]

Reactions for Spillage and Waste Disposal

$$ZrCl_4 + 4H_2O \rightarrow \underset{\substack{\text{zirconium}\\ \text{hydroxide}\\ \text{(insoluble)}}}{Zr(OH)_4} + 4HCl$$

REFERENCES

1. Merck 10227.
2. Sax 3548.
3. CRC.
4. Rosenheim, A. et al., *Chemische Berichte*, 40, 811, 1907.
5. Anonymous, British Chemical Industries Safety Council, *Quarterly Safety Summaries*, 40, 16, 1969.
6. ACGIH 61.
7. Armour, M.A., Browne, L.M., and Weir, G.L., *J. Chem. Educ.*, 62, A93, 1985; Armour, M.A., *J. Chem. Educ.*, 65, A64, 1988; *Haz. Mat. Spills Tec.*, 29.5.