GEOLOGICAL SURVEY CIRCULAR 827

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The Handling, Hazards, and Maintenance of Heavy Liquids in The Geologic Laboratory

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CONTENTS

ILLUSTRATIONS

TABLE

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ABSTRACT

In geologic laboratories the organic heavy liquids bromoform, methylene iodide, tetrabromoethane, and clerici compounds have been used for years in mineral separation processes. Because the volume of use of these compounds is low, insufficient data is available on their toxic properties. This report is an attempt to summarize the known data from published and industry sources. The physical properties, hazards of handling, proper storage facilities, and adequate protective clothing are discussed for each compound as well as for their common and less-common solvents. Toxicity data for these materials is listed along with exposure symptoms and suggested first aid treatments. Safety for the worker is emphasized. Three reclamation methods which recover the solvent used as a dilutant and purify the heavy liquid are discussed and illustrated. These include: the water cascade, refluxing-distillation-condensation, and flash evaporation methods. Various techniques for restoration and stabilization of these heavy liquids are also included.

INTRODUCTION

The heavy liquids bromoform ($CHBr_3$), methylene iodide (CH_2I_2), tetrabromoethane ($(CHBr_2)_2$), and clerici compounds are organic chemicals of primary importance in the mineral separation processes followed in many geologic laboratories. For years geologists and their technicians have used and abused these highly toxic substances. Perhaps this misuse can be attributed to a lack of organized and properly distributed information relative to the exact hazards of these materials. It is the intention of this report to consolidate data on heavy liquids from literature sources and from personal communications with scientists and medical personnel working in the geologic and chemical disciplines. Any trade names and trademarks in this paper are used for descriptive purposes only and do not constitute endorsement by the U.S. Geological Survey.

The physical properties of each chemical will be listed as well as toxicity data. The indirect and obvious hazards of exposure to heavy liquids will also be discussed along with first aid treatments. Procedures for handling and storage, with emphasis on safety of the worker, are itemized. A section on the various methods used to reclaim and stabilize heavy liquids and their solvents has also been included with an appendix containing data on some of the more exotic solvents potentially used as heavy liquid dilutants.

The authors have extensively researched these substances. However, any additional data or references from readers of this report would be most appreciated.

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Dr. M. W. Anders, Department of Pharmacology, University of Minnesota, Minneapolis, Minnesota 55455.

Richard Doll, E. B. Knight Co., Toms River, New Jersey 08753.

James Svoboda, Columbia Organic Chemicals, P.O. Box 9096, Columbia, South Carolina 29290.

Jerry Swimmer, National Biochemical Co., Geoliquids Division, 3127 West Lake Street, Chicago, Illinois 60612.

SECTION I: PROPERTIES, HAZARDS, EXPOSURE SYMPTOMS, AND TREATMENT

BROMOFORM (tribromomethane)—Properties, hazards, exposure symptoms, and treatment

Chemistry:	$CHBr_3$
Physical properties:	
Description:	Colorless liquid or hexagonal crystals. Odor similar to chloroform (when chloroform is used as preservative).
Molecular weight:	252.77
Melting point:	6°-7°C
Boiling point:	149.5°C
Density:	2.89 at 20°C
	Density varies due to preservative added. Pure bromoform is unstable and would decompose in several weeks. Preservatives are added to inhibit this decomposition. Ethanol and chloroform are commonly used. They are added in quantities of 1-2 percent which lowers the density to 2.85-2.88 (J. Swimmer, oral commun., 1978). Propylene oxide may also be used (R. Doll, oral commun., 1978). It requires only 0.5 percent by volume giving a somewhat higher specific gravity than does bromoform stabilized by ethanol.
Flash point:	None (nonflammable).
Weight per liter:	(6.36 lbs) = 2.88 kg/liter (Sax, 1978).
Vapor pressure:	15 mm at 41°C (Weast, 1970).
Common dilutants:	Acetone, alcohols, benzene.
Less-common dilutants:	Carbon tetrachloride, triethylorthophosphate N,N-dimethylformamide, isoamylacetate dimethylsulfoxide (Zussman, 1977).

Toxicity

The permissible exposure level for bromoform, based on its toxicity is established by Federal standards. One method of measuring this tolerance level is the TLV (Threshold Limit Value). The TLV of bromoform is 0.5 ppm, which is 5 milligrams per cubic meter of air. The American Conference of Government and Industrial Hygienists (ACGIH) cautions the use of TLV as a hazard or toxicity index as TLV is intended for use as a positive indicator of exposure levels.

Bromoform is listed as a suspected carcinogen (cancer-causing agent) by the National Institute of Occupational Safety and Health (NIOSH) (1976). The chlorination of drinking water can lead to the formation, if appropriate precursors are present, of compounds known as trihalomethanes, of which bromoform is one (Bunn and others, 1975; Horvath, 1976). Because of this association, bromoform and other trihalomethanes were tested and bromoform was found to be mutagenic (Simmer, 1977; Simmon and others, 1977) which is apparently one criteria to suspect carcinogenic tendencies. Bromoform, when tested by Theiss and colleagues (1977, p. 2717) was found to produce "a pulmonary adenoma response" or tumors in mice. It is currently being investigated in a 2-year test program by the National Cancer Institute (NCI representative, oral communication, December, 1979).

The LD_{50} for bromoform (that amount which will kill 50 percent of a test animal population within 14 days) is 1,820 mg/kg of body weight. This determination was done on mice. The lowest published Lethal Dose data (LD_{LO}), obtained on rabbits is given as 410 mg/kg of body weight (NIOSH, 1977).

Bromoform is a metabolic poison that may be absorbed through the skin and is potentially cumulative in the body. It is considered a Class B poison (Sax, 1968).

Byproducts of bromoform decomposition also have toxic properties. If overheated to the point of decomposition (indicated by a color change), carbonyl bromide ($COBr_2$) is produced which is highly toxic (Sax, 1968). A second byproduct of decomposition, hydrobromic acid (HBr) is a pale yellow liquid which is highly corrosive and releases toxic vapors that attack metal (O'Connell, 1963). Both compounds pose a hazard if inhaled.

Hazards

Perhaps the greatest hazard of bromoform and similar "heavy liquids" is that the exact nature of the hazards involved with their use is only vaguely known. Sufficient toxicological research has not been done on these compounds to adequately determine their long-range effects on the human body. Therefore every precaution must be taken in their use.

Due to the slow evaporation rate of bromoform, which is a function of its low boiling point, the risk of atmospheric contamination can be reduced but not removed. Because of its fairly high vapor pressure (15 mm at 41°C), bromoform is transformed into vapor with relative ease as compared to methylene iodide (1.01 mm at 25.3°C), and even acetylene tetrabromide (10 mm at 114°C). Therefore, additional respiratory precautions combined with adequate ventilation are essential because of the toxic fumes released during the use of bromoform. A major consideration is the fact that once bromoform is smelled in the air, it is an indication that the threshold limit for a person for an 8-hour day (0.5 ppm or 5 mg/m^3) may have been reached or is being reached (Sax, 1968). A person subjected to the odor of bromoform may become accustomed to it and not notice the subsequent saturation of the air until more unpleasant side effects occur. However, please note that the sense of smell is not a reliable medium for the determination of exposures.

An insidious hazard of bromoform is that it metabolizes to carbon monoxide and can substantially raise the level of CO in the bloodstream (Anders and others, 1978; Ahmed, Kubic, and Anders, 1977; Stevens and Anders, 1978). Carbon monoxide has 200 times more affinity for the iron in hemoglobin than oxygen and therefore diminishes the ability of the blood to carry oxygen. An additional problem is that in the presence of CO hemoglobin is more reluctant to give up the oxygen it already carries. As CO saturation of the red blood cells occurs a person becomes light-headed, dizzy, and eventually loses consciousness. Fifty-percent saturation is extremely serious; close to 60 percent can cause death. Carbon monoxide produced biosynthetically, as from the bromoform interaction, tends to remain in the body longer than does CO from more primary sources. Smoking can accelerate the effects of carbon monoxide overexposure as smokers have approximately a 7-percent CO saturation initially before any exposure to bromoform can add to this. The preceding information comes from a discussion with Dr. Anders of the University of Minnesota. His research to date has been done with rats.

Bromoform is a contact poison and can be introduced into the body by external exposure. Hydrocarbons such as bromoform have an affinity for body oils and tend to also move into fatty tissues (Bowman, Borzelleca, and Munson, 1978).

The decomposition byproducts, carbonyl bromide and hydrobromic acid, produce dangerous vapors and require immediate ventilation.

When bromoform is dissolved with acetone, the mixture becomes highly flammable. Fires from this solution are extremely hazardous because as the bromoform is heated, and therefore decomposes, toxic fumes are liberated.

Geologists using or recovering bromoform in fieldwork must use extra care that proper ventilation is provided—especially when working in air-conditioned trailers or with quantities in open funnels.

Exposure Symptoms

A chemical may have an acute effect on the body which is an immediate or almost immediate manifestation of exposure symptoms and it may also cause chronic or long term effects from exposure. As can be seen from the cited references, research programs to determine precisely what deleterious effects bromoform has on the human system have only recently been undertaken. Some data has been extrapolated from similar compounds. Unfortunately, when internal organs such as the liver and kidneys are involved, definitive data on humans is many times only available after an autopsy. With a chemical as exotic as bromoform the appropriate tests are rarely performed. It is not always possible to determine if the damage is caused by the passage of a compound through the system or its retention therein. It is the experimentally unsubstantiated opinion of several researchers in this area that, although bromoform is retained within the system for a longer period of time than the "average" alien substance, it is eventually flushed from the body; but that the amount acquired from overexposure is such that the bromoform appears to be perhaps indefinitely retained.

Careless use of bromoform, failure of ventilation systems, or spills can cause exposure over the limits considered acceptable. Some of the symptoms of overexposure are: tearing, headaches, excessive saliva flow, nausea, reddening of the face, dizziness, drowsiness or a state of apparent inebriation, or, with prolonged exposure, deep narcosis and death (Sax, 1968). Skin contact may be followed by burns that appear as second-degree inflammations.

It is important to note that the consumption of alcoholic beverages before, during, or after exposure to bromoform may enhance the exposure symptoms and increase the effects of the combined exposures (National Biochemical Co., 1975; Sax, 1968). As noted in the previous section, smoking may also escalate the effects of bromoform on the system causing dizziness, headaches, and even loss of consciousness (Dr. Anders, oral commun., December 1979).

Bromoform can cause systemic damage. Repeated exposure can lead to excessive damage to the liver, kidneys, heart, and lungs. Experiments with rats by Bowman, Borzelleca, and Munson (1978) of the Virginia Commonwealth University, showed that bromoform infiltrates the fatty tissue of the liver and also can cause hemorrhaging in the kidneys, adrenals, lung, and brain of the test animals.

Bromoform is potentially cumulative in the body organs and, therefore, repeated overdoses of it are highly dangerous. An internal lethal dose is 2-4 ounces for a 150-pound man (Carver, 1971).

An additional problem relative to determining the "safe" limits of exposure to bromoform is the fact that each person can react differently to the same volume of vapors. Workers whose prior working history shows that they are overly sensitive to bromoform vapors should not be allowed to work where exposure is assured or likely.

Treatment

Countermeasures for overexposure victims are: remove the victim to fresh air, administer artificial respiration and oxygen if needed, and get medical assistance immediately.

For skin contact, flush thoroughly with water and wash with soap. Skin oils will absorb the bromoform and therefore they must be dissolved with a strong soap and washed away. Remove contaminated clothing and wash it before reuse.

If the victim exhibits advanced inhalation exposure symptoms, tests for carbon monoxide poisoning should be considered. This is perhaps easier to determine than the presence of bromoform, bromides, or bromine in the blood. Bromine blood content can apparently be tested for by electron capture gas chromatography (Dr. Anders, oral commun., December 1979).

First aid measures given by the American National Red Cross (1973, p. 101-102) for inhaled poisons are:

1. Remove the victim from the poison source. Be sure you do not become a victim yourself.
2. Have oxygen brought to the scene.
3. Loosen the victim's clothing, give artificial respiration if needed.
4. Be sure to remove any saturated clothing from the area.

Cases of overexposure should be referred to a physician as soon as possible. The rescuers should be sure to protect themselves and to wear protective gear when helping others.

METHYLENE IODIDE (diiodomethane)—Properties, hazards, exposure symptoms, and treatment

Chemistry:	CH_2I_2
Physical properties:	
Description:	Straw-colored, clear liquid.
Molecular weight:	267.9
Melting point:	5°-6°C
Boiling point:	181°C
Density:	3.325 at 20°C
Vapor pressure:	1.01 mm at 25.3°C (J. Swimmer, oral commun., 1979).
Flash point:	None. (Vapor will burn if exposed to open flame.)
Weight per liter:	3.32 kg/liter (Sax, 1968; Weast, 1970).
Common dilutants:	Acetone, alcohols, benzene.
Less-common dilutants:	Carbon tetrachloride, triethylorthophosphate, N,N-dimethylformamide, ortho-dichlorobenzene (Zussman, 1977).

Toxicity

A literature search reveals that there is minimal documented data on methylene iodide (MEI). This lack of data may be a function of limited use of MEI in industry and the sciences. (See National Biochemical Co. Bull. No. 31, 1975, for list of physical properties and arbitrary precautions used by that company in their manufacturing processes.)

The TLV is estimated to be 50 ppm per 8-hour working day. This value is experimentally unsupported and its use as a standard is therefore suspect. This is much higher than 0.5 ppm TLV for bromoform determined by the ACGIH (American Conference Governmental Industrial Hygienists). Until further tests are conducted on MEI and a specific standardized TLV is established, precaution is advised. Due to the high vapor density and low vapor pressure, MEI can perhaps be used relatively safely with adequate ventilation as compared with other halogenated hydrocarbons. A TLV of 25 ppm is suggested by Jerry Swimmer (written commun., 1979) based on personal experience within his company. An LD$_{50}$ for methylene iodide is given in NIOSH (1977) as 830 mg/kg. The determination was done on a mouse.

Hazards

The hazards of MEI are mostly unknown. It is, however, a probable irritant and a possible narcotic in high doses. It has been known to cause local anesthesia of nasal mucous areas from moderate and high vapor concentrations. Mr. Swimmer indicates that in his facility and within his experience in the industry no one to his knowledge has lost consciousness due to methylene iodide vapors (J. Swimmer, written commun., 1979).

Like bromoform, methylene iodide has been shown to metabolize carbon monoxide and raise the level of CO in the blood with all the attendant symptoms and problems described in the bromoform section (Kubic and others, 1974; Kubic and Anders, 1975).

Methylene iodide will decompose upon exposure to light and heat and will emit toxic iodine fumes. MEI itself is not flammable, but the vapor will burn if exposed to open flames, releasing large amounts of free iodine (Sax, 1968). Iodine possesses toxic properties similar to those of methylene iodide.

MEI is often diluted with acetone—as a washing agent or to lower the density of MEI. It must be remembered that, although MEI is not itself flammable, it becomes so when mixed with acetone. A fire will pose added danger as MEI iodine vapors will be released into the air during combustion.

Exposure Symptoms

When using MEI, the proper precautions for working with a toxic liquid should be followed. These precautions include adequate ventilation and protective laboratory clothing. MEI should be considered to be an anesthetic in high vapor concentrations—adequate ventilation is necessary. Overexposure can result in respiratory complications. Excessive overexposure may bring about unconsciousness. MEI is rated as a narcotic by Sax (1975). This classification is refuted by Jerry Swimmer (written commun., 1979) who cites loss of consciousness as a criteria of a narcotic substance. Because the vapor pressure of MEI is so low, the chances are slight of MEI vapors saturating the atmosphere sufficiently to cause overexposure to the point of unconsciousness in the worker.

Treatment

The general procedures listed in the bromoform section under "Treatment" can be considered to apply to MEI exposures also.

ACETONE (dimethyl ketone, ketone propane, propanone)--Properties, hazards, exposure symptoms, and treatment

Chemistry:	CH_3COCH_3
Physical properties:	
Description:	Clear, colorless liquid having a mintlike odor and a sweet taste; in the context of the present report, acetone is referred to in its use as a dilutant for bromoform and methylene iodide.
Molecular weight:	58.08
Melting point:	-94.6°C
Boiling point:	56.48°C
Soluble in:	Water, alcohol, ethyl ether, benzene.
Density:	0.797
Flash point:	-17.8°C
Autoignition:	538°C (1,000°F).
Vapor pressure:	400 mm at 39.5°C
Weight per liter:	0.79 kg/liter (6.59 lbs/gallon).
Explosive limits:	Lower, 2.6 percent; upper, 12.8 percent (Sax, 1968; Weast, 1970).

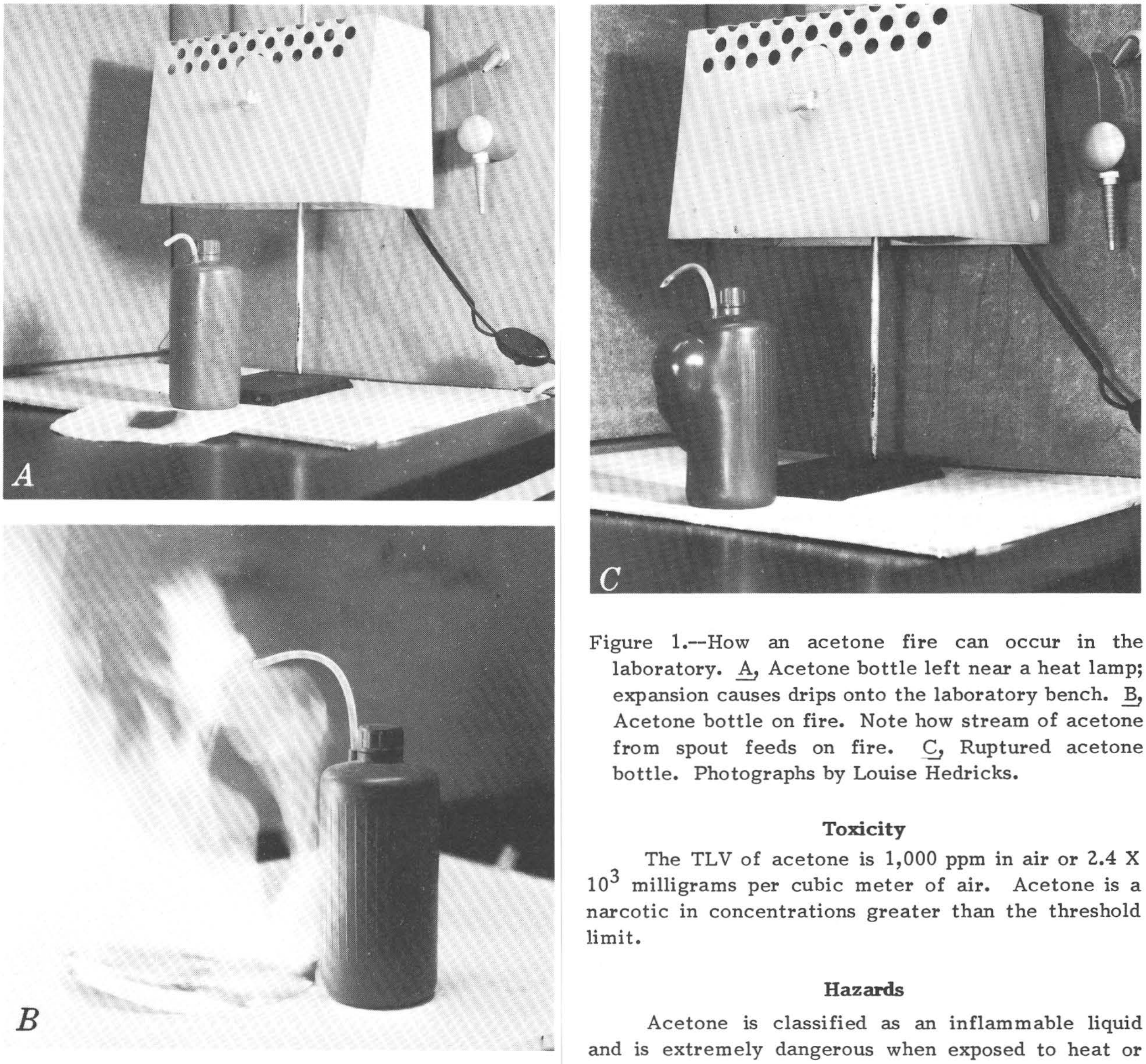

Figure 1.--How an acetone fire can occur in the laboratory. A, Acetone bottle left near a heat lamp; expansion causes drips onto the laboratory bench. B, Acetone bottle on fire. Note how stream of acetone from spout feeds on fire. C, Ruptured acetone bottle. Photographs by Louise Hedricks.

Toxicity

The TLV of acetone is 1,000 ppm in air or 2.4 X 10^3 milligrams per cubic meter of air. Acetone is a narcotic in concentrations greater than the threshold limit.

Hazards

Acetone is classified as an inflammable liquid and is extremely dangerous when exposed to heat or flame. Even mixed with water, a solution of greater than 10 percent acetone is flammable, having a flash point of 27°C. The fact that acetone is extremely volatile adds to its hazard potential. Explosive vapors can soon saturate the air in the event of a spill. Keep acetone away from any heat or possible ignition source. Heat lamps used to dry samples in the laboratory are a very real danger. Acetone placed close to a heat lamp will volatilize and spill all over the laboratory counter. Figure 1 shows how an acetone fire can occur.

Acetone may also react vigorously with oxidizing materials (Sax, 1968).

Exposure Symptoms

The volatile nature of acetone poses a problem if acetone is used in a poorly ventilated area. The symptoms of overexposure are dizziness, headache, coughing, irritation of the respiratory tract and, in more extreme cases, unconsciousness (Sax, 1968). The symptoms of slight exposure (dizziness, headache) are quickly relieved by removing the victim to fresh air. The effects will disappear in about 2 hours. Prolonged exposure will cause more acute headache and dizziness and may result in the loss of consciousness.

Treatment

Remove the victim to fresh air and administer artificial respiration if necessary; immediate medical help is imperative.

For external exposure: acetone will dissolve skin fat upon contact and cause dryness. Use of hand lotion will help avoid cracking and relieve irritation.

If acetone is splashed in the eye, irrigate with large amounts of water and call a physician.

TETRABROMOETHANE (acetylene tetrabromide)—Properties, hazards, exposure symptoms, and treatment

Chemistry:	$(CHBr_2)_2$
Physical properties:	
Description:	Colorless to yellow liquid.
Molecular weight:	345.7
Melting point:	-1°C
Boiling point:	151°C at 54 mm (Sax, 1975). 239.42°C at 760 mm (Weast, 1970).
Density:	2.96 at 25°C.
Flash point:	None.
Weight per liter:	2.96 kg/liter (Sax, 1968; O'Connell, 1963).
Vapor pressure:	10 mm at 114°C (Weast, 1970).
Common dilutants:	Acetone, alcohols, benzene.
Less-common dilutants:	Carbon tetrachloride, triethylorthophosphate (Zussman, 1977).

Toxicity

The TLV of tetrabromoethane is set by the ACGIH at 1.0 ppm (14 mg/m^3 air). This substance, like bromoform, is a metabolic poison and a narcotic in high concentrations (Sax, 1968).

One of the byproducts of tetrabromoethane when heated to decomposition is carbonyl bromide (carbon oxybromide), $COBr_2$. It is listed (Sax, 1975) as extremely poisonous, and is present as a vapor. Also formed in the presence of water is hydrobromic acid, HBr, an extreme corrosive.

Hazards

The hazards are similar to those of bromoform, with adequate ventilation imperative for workers' safety. Both internal and external exposure pose hazards because the substance is both an irritant and a metabolic poison. The odor may not readily be noticed by workers as they will become accustomed to it. Hooded ventilation systems are necessary when working with tetrabromoethane.

Exposure Symptoms

Exposure over the accepted limits to the fumes can cause systemic problems of varying degrees. Slight exposure causes tearing and drowsiness. Single exposures of this type pose little danger of permanent injury. Repeated exposure, however, becomes a severe hazard and can result in long-term injury or death. Major damage can be done to the kidneys and liver, where toxins accumulate during blood filtration. If the toxic material is introduced faster than it can be expelled, poisoning will occur. This explains the danger of cumulative poisoning by repeated slight exposure.

Exposure to the skin is not serious if the tetrobromoethane is washed off immediately. If not, it can cause severe burns, and absorption through the skin may cause systemic poisoning.

Treatment

Treatments for overexposure are the same as those for bromoform. Tetrabromoethane effects are also enhanced by the use of alcoholic beverages.

CLERICI SOLUTION (thallium-malonate-formate)—Properties, hazards, exposure symptoms, and treatment

Chemistry: $CH_2(COOTl)_2 \cdot HCOOTl \cdot H_2O$

Physical properties:

Description: Clerici solution is made up of equal weights of thallus formate ($CH_2(COOTl)_2$) and thallus malonate (HCOOTl) in distilled water. A solution of approximately 135 ml is made up from 300 g of each thallium compound dissolved in 40 g of distilled water. Several other methods of preparation for clerici solution are described by Vassar (1925). Thallus monalate and formate are available either as salts or in solution (Jahns, 1939). Odorless liquid, clear to amber in color. Highly mobile liquid (pours as water).

Density: 4.28 at 20°C. Density increases with temperature and thallium salt concentration to approximately 5.0 at 90°C in a saturated solution.

Boiling point: 125°C at 1 atmosphere (Sax, 1968; Rosenblum, 1974).

Common dilutant: Distilled water.

Toxicity

The threshold limit value for thallium compounds has been set by the ACGIH at 0.1 mg/m^3 air.

Hazards

Thallium compounds carry a hazard reputation that is indeed sobering. Thallium compounds are used in rat poisoning. The risks of atmospheric contamination can be limited with proper ventilation during use and storage. Because the compounds are odorless and usually colorless, they give no warning of their presence.

Heating clerici solution to vary its density gives off highly toxic, heavy-metal fumes. The danger of exposure cannot be overstressed. Clerici solution should never be exposed to an open flame. It should be heated only over a steam bath. The high mobility of clerici solution may create additional hazards; if spilled, the solution can move rapidly into small areas and cracks and remain there undetected.

Clerici compounds should be used only as a last resort when all other methods of separation are ineffectual. If the compounds are used, extra precaution should be instituted in the working area. These precautions are discussed in detail in the later section on handling.

Exposure Symptoms

The greatest dangers to the laboratory user are inhalation and exposure to the skin. Repeated exposure to thallium compounds is cumulative in the body and can cause serious physical impairment or death. Thallium and its salts are absorbed through the skin.

External exposure to thallium salts is immediately irritating to areas of contact. Because clerici is odorless, atmospheric contamination may go unnoticed until acute internal exposure has taken place. The symptoms of thallium poisoning resemble those of lead and other heavy-metal poisoning. Some symptoms are: swelling of the lower extremities, joint pain, abnormal sensitivity in feet or hands (burning and (or) tingling), mental confusion, paralysis, inflammation of the kidneys, abnormal white blood count, and loss of hair of the head and body. Fatal poisoning has been known but is not common. (Sax, 1968).

A white deposit may appear on the skin after a slight contact with thallium salts. If the skin is properly and promptly washed with soap and water, this deposit will usually disappear within several days without any apparent injurious effects (Engineered Materials, 1969).

Treatment

Remove the victim from the area of contamination and obtain immediate medical assistance. If any of the before-mentioned symptoms appear, medical care is imperative.

Wash the skin exposed to the chemical immediately with soap and water. If the chemical is ingested, force the victim to drink large volumes of fluids until medical help is available. Give first aid treatments only after medical help has been called for (McElwee, 1979).

BROMOFORM—Storage, handling, and spills

Storage

Bromoform, like methylene iodide, is a halogenated hydrocarbon and will break down and darken due to free halogens emitted by exposure to heat and light (J. Swimmer, written commun., 1978). It should, therefore, be stored in amber glass bottles in a cool, dry place; preferably in an area of good ventilation.

Handling

Any time heavy liquids are handled, it is imperative to wear gloves, a rubber apron, and goggles or safety glasses (fig. 2). The gloves should be checked for cuts or tears. Rubber gloves are advised; most plastics will deteriorate with prolonged exposure to a heavy liquid. Work with bromoform should be performed under a fume hood that should draw 160 feet per minute (fpm) face velocity. Less draw violates the OSHA regulation that if the TLV is exceeded, the hood is not venting properly. The additional precaution of wearing a filtered respirator is also advised. Filter paper and absorbent towels or tissues contaminated with bromoform should be

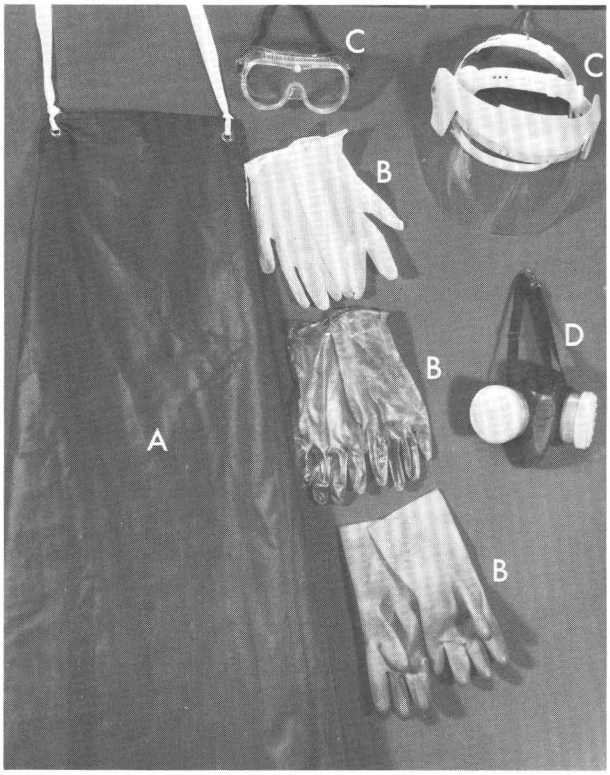

Figure 2.—Protective clothing recommended for heavy-liquid work. A, Rubber apron; B, rubber gloves; C, safety goggles; D, respirator with replaceable filters. Photograph by Louise Hedricks.

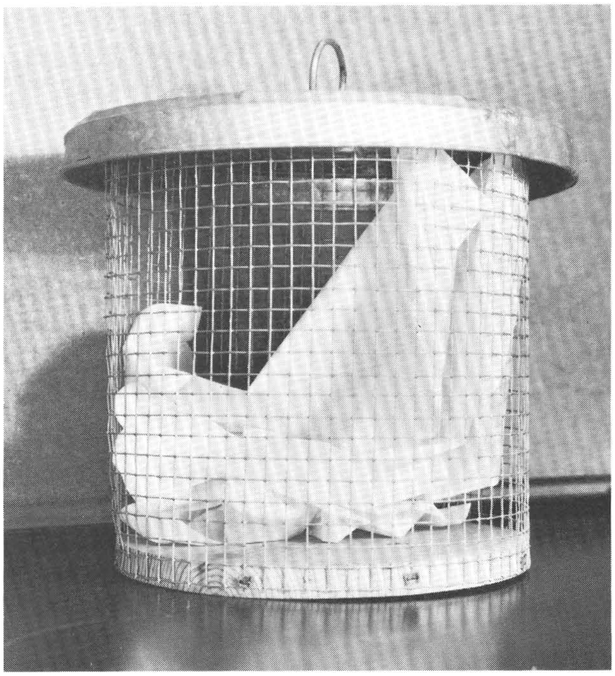

Figure 3.—A type of wire mesh basket used to collect saturated heavy-liquid papers. Photograph by Louise Hedricks.

Figure 4.—Recommended storage bottles for pure heavy liquids and wash, appropriately labeled. Photograph by Louise Hedricks.

removed from the laboratory and not left in open waste containers outside the hood. A wire mesh basket (fig. 3), suspended or set in the hood, can be used during the day, while the hood is operating, to evaporate heavy liquid and solvent from saturated filter paper and tissues. It should be emptied into a large and, if possible, ventilated or outside disposal container at the end of the job or the day.

In the transfer of bromoform, care should be taken with any plastics exposed to the liquid. Most laboratory plastics will deteriorate and eventually fail with prolonged exposure to bromoform. Glass and teflon should be used whenever possible (Cole-Parmer, 1977). Figure 4 shows recommended storage bottles. Any area where heavy liquids or their products are used must be adequately ventilated and care must be taken that fumes are not recirculated into adjacent ventilation systems.

Remember to prominently label any storage area or bottles containing a heavy liquid.

Spills

In the event of a large spill, several precautions should be taken. Make certain all potential ignition sources are neutralized. Toxic vapors from the spill create the greatest problem, especially if the spill occurs outside the hood area. If possible, turn off central air-conditioning to keep fumes from nonlaboratory areas. Proper equipment must be worn to avoid vapor inhalation. A self-contained breathing unit (preferable) or a respirator with detachable, renewable filters should be worn by each member of the cleanup party. Protective clothing is essential. Rubber aprons are recommended as are rubber gloves and goggles because cloth laboratory coats may absorb the heavy liquid. A cleanup kit of absorbent material (fuller's earth or kaolin powder) should be kept close to the area where heavy liquids are used. A typical spill station containing the appropriate items for spill cleanup is shown in figure 5. The fuller's earth is

Figure 5.--Spill station in the laboratory showing basic equipment and neutralizing materials. Photograph by Louise Hedricks.

sprinkled over the spill and allowed to absorb the heavy liquid. Then, using a broom and dustpan, sweep up the now-saturated absorbent. Transfer it to filter paper in a funnel set into a filter flask attached to a vacuum. By filtering under a high vacuum there is a chance of recovery of some of the heavy liquid. Because fuller's earth is highly absorbent, recovery is not complete, but worth the effort. After the mess is removed from the floor or countertop, pour acetone on the area and wipe clean to remove final traces of heavy liquid.

A small spill can be cleaned by squirting the spill with acetone and wiping it up with absorbent paper. The papers are disposed of as described in the section on handling.

Great care should be taken to avoid skin contact with chemicals and cleanup material and inhalation of toxic vapors.

METHYLENE IODIDE—Storage, handling, and spills

Storage

A main consideration in the storage of methylene iodide is that it is highly sensitive to light and decomposes quickly upon exposure.

When MEI decomposes, it darkens to an opaque, blood-red liquid due to the free iodine that is liberated with oxidation. To retard decomposition, store MEI in a dark, cool place, tightly sealed in amber glass bottles. Good ventilation in the storage area is also recommended. A small piece of copper or silver wire mesh in the bottle will theoretically maintain the stability of the liquid and thus the lighter color for extended periods of time when stored in tightly closed containers away from heat, light, air, and moisture (National Biochemical Co., 1975).

Handling

Transfer of MEI should take place only in areas of adequate ventilation and should use nonreactant tubing. In the various references available, no data was found on the resistance of various plastics to MEI. Any plastic to be used in prolonged contact with methylene iodide should be tested before use. A wise assumption, for safety reasons, is that MEI reacts to plastics in a fashion similar to bromoform.

The same precautions listed in the bromoform section relative to protective clothing, handling, and air contamination also apply to methylene iodide.

Spills

Methylene iodide spills are handled in the same manner as those involving bromoform.

ACETONE—Storage, handling, and fires

Storage

Acetone will dissolve some types of plastic and, therefore, care must be taken in its storage. Acetone is safely kept in containers of teflon, polypropylene, polymethylpentene, and glass (Cole-Parmer, 1977). It cannot be stored or transferred in polycarbonate or polyvinyl chloride containers. The plastic laboratory gloves and aprons used when handling acetone must be constantly checked for signs of deterioration. Acetone must also be stored in a well-ventilated area.

Handling

When transferring acetone from a larger drum, use a self-closing faucet and be certain that the drum is effectively grounded; static electricity could generate a spark that may lead to a fire or explosion. Remember that the flash point of acetone is $-17.8^{\circ}C$. Never transfer acetone by means of compressed air as acetone is too volatile a substance. For storage in an active laboratory for daily access, use an approved safety-storage can with appropriate dispenser. Avoid storing large volumes in the laboratory. Figure 6 shows a grounded storage can with an automatic closing dispenser and a safety can.

Any time there is a chance of external exposure, protective clothing should be worn, including apron, goggles, and gloves.

Fires

Because acetone is so highly inflammable, the chance of a fire is great in any laboratory using acetone. In the event of a fire, a CO_2, foam or dry chemical extinguisher will smother the blaze. Water will only be effective when there is less than 10 percent acetone remaining (National Safety Council, 1963).

Also remember that any heavy liquid diluted with greater than 10 percent acetone will burn just like acetone itself.

TETRABROMOETHANE—Storage, handling, and spills

Storage

This liquid, like bromoform, is a halogenated hydrocarbon, and is similarly unstable. Tetrabromoethane should be stored in a cool, dark, ventilated area in glass or nonreactive plastic.

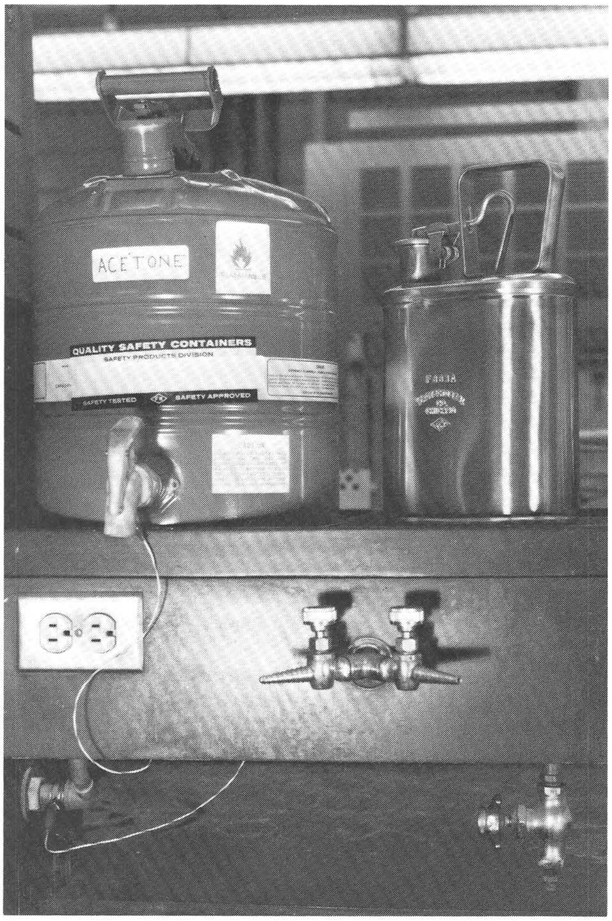

Figure 6.--Acetone storage safety can. Acetone should be stored in an approved safety can that is grounded for sparkless transfer. Photograph by Louise Hedricks.

Handling

When transferring tetrabromoethane, bromoform, or methylene iodide, keep in mind that plastics and rubbers swell and soften with exposure. Fluorinated polymers (teflon, Kel-F) show the greatest resistance. A recommended procedure is to test a proposed plastic or rubber before using it in a transfer system. Prior testing will eliminate the problems of cleaning up after a messy spill caused by a deteriorated bottle. Transfer should be carried out only in a well-ventilated area, and protective clothing, such as that described for bromoform, should be worn (O'Connell, 1963; Sax, 1968).

Spills

Tetrabromoethane spills are handled in the same fashion as bromoform spills.

CLERICI SOLUTION—Storage, handling, and spills

Storage

As a safety precaution, thallium formate and thallium malonate should be stored in a solid form, especially if storage is for any appreciable length of time. These are highly poisonous compounds and the liquid phase is more mobile than the solid crystals. There is a greater chance of a spill and subsequent spread of this highly toxic substance through the laboratory.

Whether clerici is stored as a solid or in the less-desirable liquid phase it must be placed in a secluded area, segregated from high-use chemicals and prominently labeled as to extreme toxicity and hazard and tightly sealed. Clerici compounds are not light-sensitive. They should be stored in glass containers as they will corrode metal and become discolored. (Engineered Materials, 1965).

The lethal aspects of clerici cannot be overemphasized.

Clerici compounds are dissolved in distilled water for use in mineral separations, but after use the water is evaporated off until dry. Care must be taken not to evaporate the water at a temperature higher than 130°C, as the thallium compounds decompose at this temperature (Browning, 1961).

Handling

The greatest consideration in handling these compounds is the extreme toxicity of thallium. Thallium compounds are readily absorbed through the skin (the damage caused was previously discussed under toxicity). Extreme caution must be used in handling these compounds and includes wearing a respirator, protective clothing, gloves, and safety goggles (O'Connell, 1963). Gloves should be rubber, neoprene, or latex (0.014 in. thick) and aprons should also be rubber (Engineered Materials, 1965). Any protective gear must be thoroughly cleaned inside and out after exposure to clerici compounds. Use of these solutions should be restricted to a posted area of the laboratory so that an unaware worker will not be exposed to them. As an added precaution, all operations involving thallium should be performed under a ventilated hood with a face velocity of 170-200 fpm.

Smoking, drinking, and eating are to be

prohibited in any area where thallium or its compounds are handled.

Instructions for handling should be reviewed in detail before using clerici solutions in mineral separation procedures (see Rosenblum, 1974; Vassar, 1925; Jahns, 1939).

Spills

Use water-saturated paper towels to clean up spills. Dispose of waste material with all precautions. Wash down spill area thoroughly with soap and water. (Engineered Materials, 1965).

SECTION III: RECLAMATION OF SOLVENTS AND HEAVY LIQUIDS

Due to the high cost of heavy liquids, the increasing costs of solvents, and the large volumes of these materials used in mineral-separation processes, it has become economically expedient to recover as much as possible for reuse. Because these compounds are relatively unstable, chemical deterioration may occur, thus decreasing their usefulness. In this section, several methods are described for the recovery of solvents and the rejuvenation of diluted or decomposed heavy liquids.

Reclamation of Diluted Solutions

Water Cascade Method of Heavy Liquid Recovery

The principle of the water-washing method is based on the relative insolubility of the heavy liquid in water as compared to the high solubility of the dilutant. Table 1 compares these solubilities at 20°C.

Table 1.—Solubilities of acetone and heavy liquids in water

Data from Muller, 1977; and Benjamin, 1971

Substance	Solubility
Acetone	Infinite
Bromoform	0.319 g/100 ml H_2O
Tetrabromoethane	0.065 g/100 ml H_2O
Methylene iodide	1.420 g/100 ml H_2O

A typical water-washing system is described in Hutchinson (1974, p. 122). The heavy liquid-solvent mixture is placed in an apparatus such as that shown in figures 7 and 8 and washed with a constant flow of water which carries away the dilutants or solvents. This washing process is fairly time-consuming as it takes about 2 hours per 1,000 milliliters of mixture.

The diluted heavy liquid is poured into a large separatory funnel (funnel 1 in fig. 8). Water is introduced through a glass tube that runs to the bottom of the funnel. Water is bubbled through the heavy liquid, dissolving the solvent and floating it above the heavy liquid, due to the lesser specific gravity of the solvent. The solvent then mixes with the water. The water-solvent mixture then flows into funnels 2 and 3 (fig. 8) where any heavy liquid that may have been carried along is trapped. The water leaves the system by a drainage tube and is disposed of, usually down a waste drain.

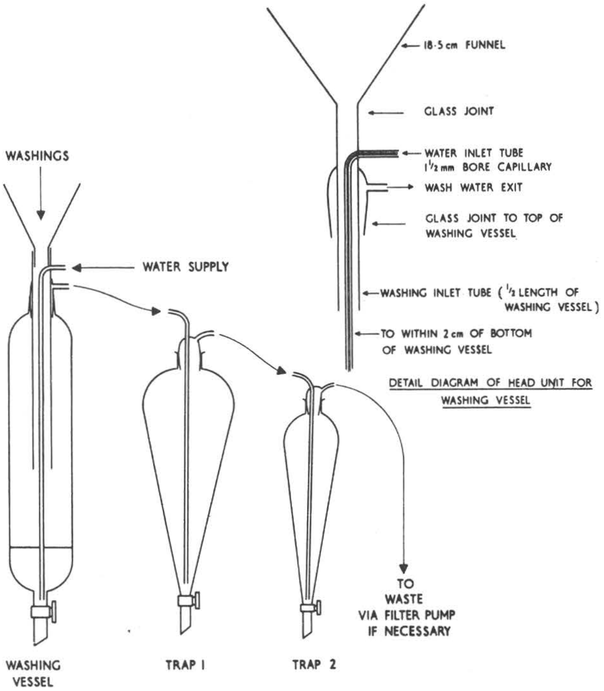

Figure 7.—Water cascade method for heavy-liquid recovery. Design from Benjamin (1971, p. 616). Published with permission.

The advantage of this method is the relatively low cost of the apparatus used. However, all of the solvent is lost, as is the heavy liquid at the water-heavy liquid interface, and a percentage of the heavy liquid that is water-soluble.

A variation on this system would be to bubble air through the solvent-heavy liquid mixture following the general principles just described. (Donald Lee, oral commun., 1979).

Refluxing-Distillation-Condensation Method for Heavy-Liquid Recovery

The refluxing-distillation-condensation method utilizes a direct heat source with variable temperature controls to evaporate acetone (or similar solvent) from a diluted heavy-liquid solution. Water is circulated throughout the condensing tube where the evaporated acetone is recondensed. (See figure 9.) The acetone is then drained off, with the heavy liquid remaining in the evaporating flask. The method is slow and must be constantly monitored. The temperature of evaporation needed for acetone is 56°C. If the heavy liquid is left exposed to this temperature for too long, it will decompose, forming toxic byproducts and darkening to a point of opacity. Once the heavy liquid is thus oxidized, rejuvenation is extremely difficult. With proper precautions, however, including the

Figure 8.—Water cascade apparatus, U.S. Geological Survey laboratory, Denver, Colorado. 1, Separatory funnel; 2, 3, additional funnels to trap heavy liquid. Photograph by Louise Hedricks.

stabilization of the distilled heavy liquid, this method has proved to be effective.

Flash-Evaporation Method of Heavy-Liquid and Solvent Recovery

The flash evaporator provides a closed, contained environment for the separation of mixtures of organic solvents and organic heavy liquids. The system operates under decreased pressure which lowers the boiling point of the solvent and allows for differential evaporation at reduced temperatures. This method reclaims both solvent and heavy liquid and operates at a lower temperature which does not oxidize the heavy liquid as does the reflux-distillation method.

The system, illustrated in figure 10, is a Buchler flash evaporator, model PTFE-1gn. Generalized, the following procedure is applicable to other types of commercially available flash-evaporation units.

The apparatus has six major parts: (1) evaporating pan and water bath; (2) evaporating flask; (3) thermoregulator and heater; (4) rotatable motor; (5) spiral condenser coil; and (6) collecting flask.

A heavy liquid-solvent mixture is introduced into the evaporating flask, which is immersed in a temperature-controlled water bath. The pressure in the system is then reduced to a 10-15 mm vacuum (the value recommended in the Buchler operating instructions) to facilitate evaporation of the solvent. A lesser vacuum will slow the process somewhat but will still give efficient results. If the available vacuum source cannot reach this value, the temperature can be raised to improve evaporation. However, care must be taken not to raise the temperature too high or oxidation of the heavy liquid will occur.

There are several advantages to a flash evaporation system that justify its initial cost. First, because the solvent is reclaimed, the total amount consumed in mineral separation procedures is reduced substantially. Also, the recovered solvent is relatively uncontaminated and can be recycled in separations as "new" acetone.

A second advantage is the elimination of large volumes of water in the recovery process and the accompanying loss of heavy liquids due to their solubility. Although the solubilities of these liquids are rather low (table 1), there will still be some loss. Water accelerates decomposition of heavy liquids. Lessening the exposure of a heavy liquid to water will increase the overall period of usefulness of the heavy liquid and will decrease the chance of creation of and exposure to harmful, toxic byproducts of decomposition.

Bromoform that has been run through the flash evaporator will have a final specific gravity of 2.75. Five to ten minutes in the water cascade system will return the density of the bromoform to its original value of 2.85. Even though there is some water exposure, this is still a greater than 92 percent decrease in the total amount of water that the heavy liquid is exposed to relative to the water cascade method when that is the sole recovery method.

Because the evaporation process takes place in a closed system, there is less chance of atmospheric contamination. The safety hazards of heavy-liquid exposure have been previously documented.

Detailed operating instructions are included in Appendix B of this report.

Modifications to the standard unit

Some modifications were made to the flash-evaporation unit to increase the efficiency of the solvent recovery. The condensing coil was wrapped on the outside with tygon tubing and covered with insulating tape. Tapwater was then run through the outside hose as well as through the inside coils of the condenser. This procedure kept the spiral condenser cooler by insulating it from the warmer room air and thus speeded up the solvent recovery. A second modification (R. Tripp, oral commun., 1978) for enhancing the recovery is to run refrigerated water

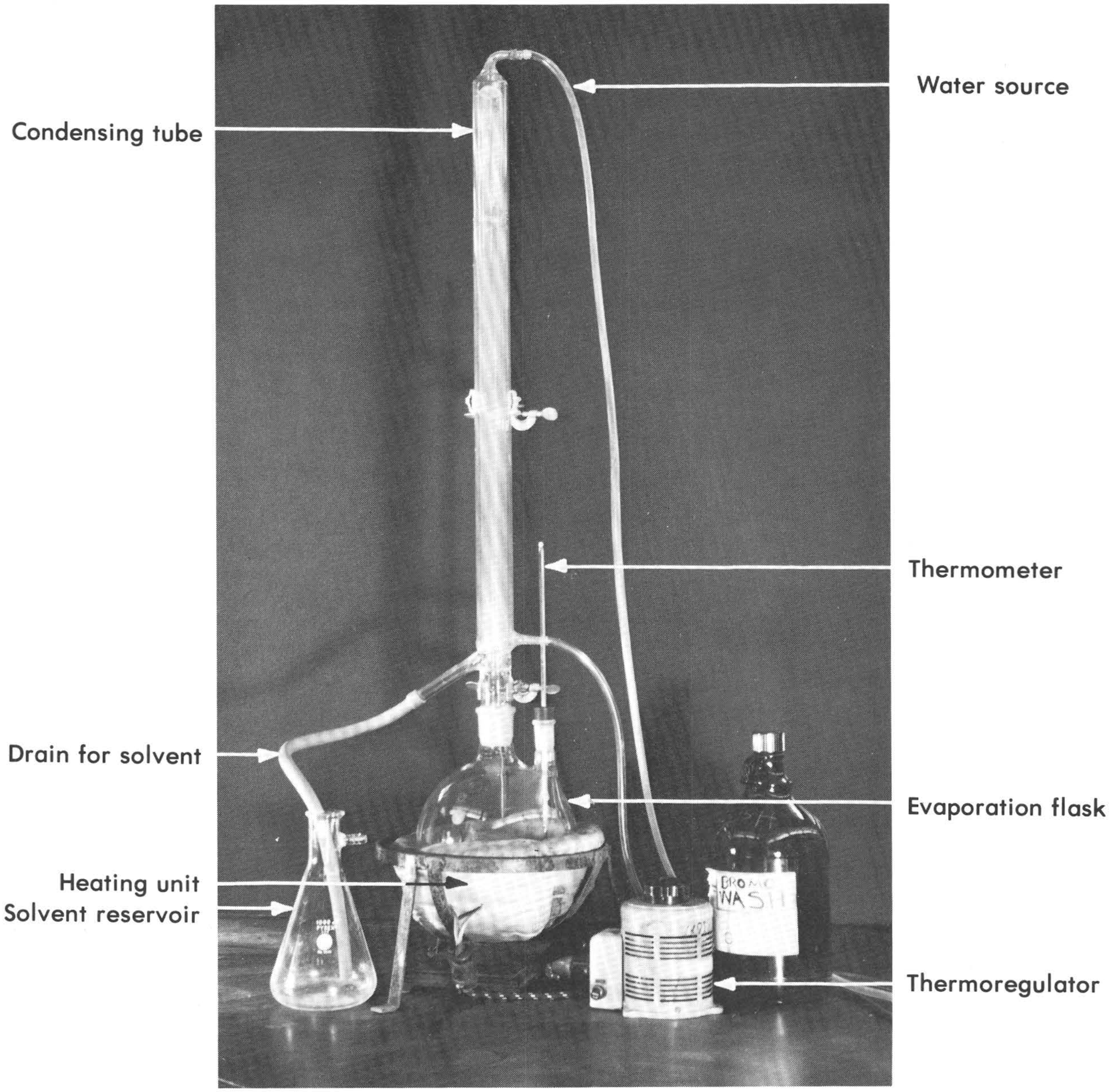

Figure 9.--Refluxing-distillation-condensing apparatus formerly used in the U.S. Geological Survey laboratory, Denver, Colorado. Photograph by Louise Hedricks.

through the condenser. A pump circulating water from an ice bath not only conserves water, but greatly reduces the temperature of the condensing environment.

Recovery and Stabilization of Heavy Liquids

The halogenated hydrocarbons are, by nature, unstable and will break down and darken in color with normal use. This decomposition is accelerated by exposure to heat, water, and ultraviolet radiation. The darkening is caused by hydrolysis and the release of free halogens into the solution (Sax, 1968; Browning, 1961). The following are recommended procedures for the reclamation of several common heavy liquids. These procedures have been empirically verified by the authors.

BROMOFORM—Recovery and stabilization

As bromform breaks down it releases free bromine (Br_2) and hydrobromic acid (HBr) which can

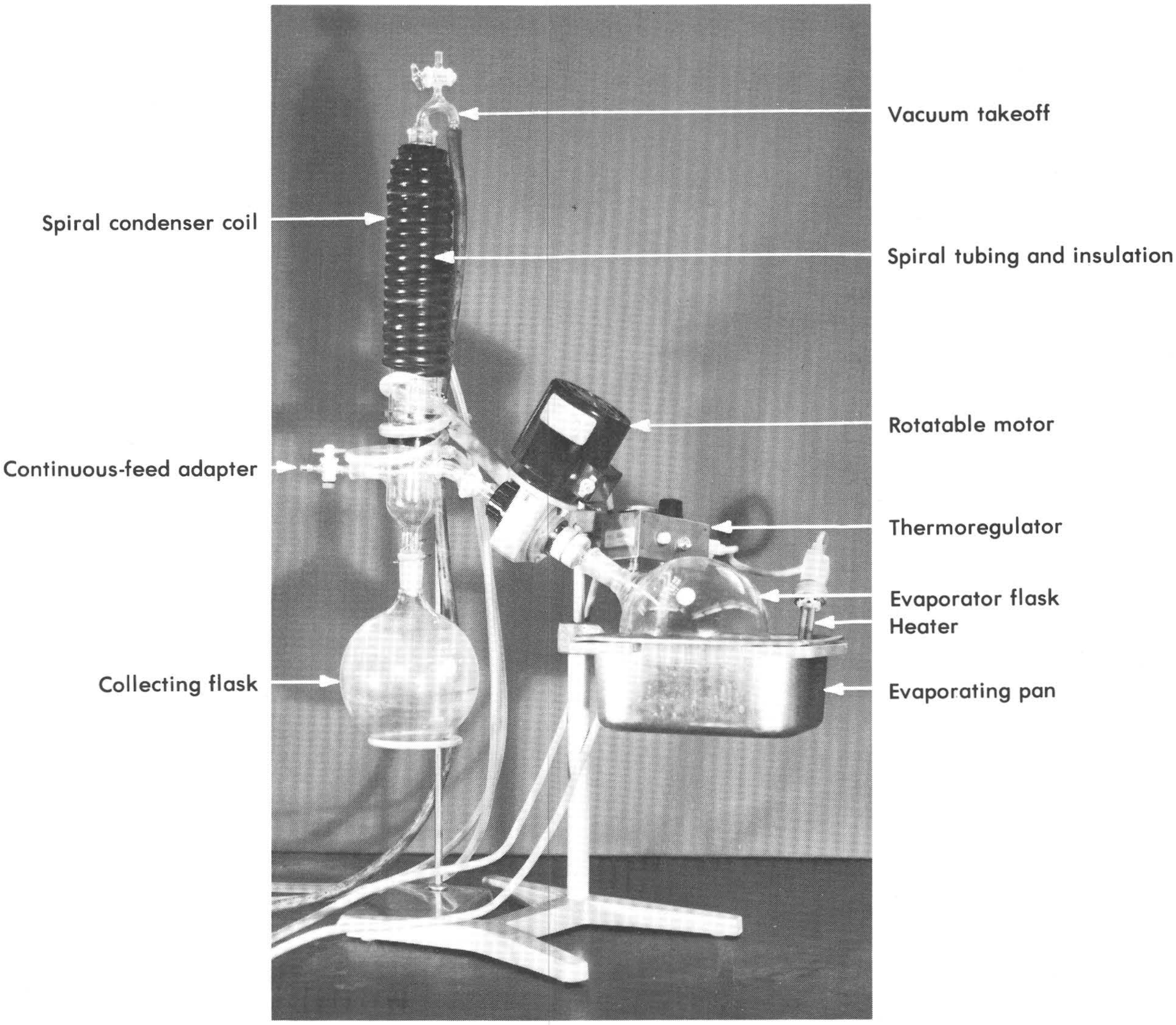

Figure 10.--Flash-evaporator apparatus used in the U.S. Geological Survey laboratory, Denver, Colorado. Photograph by Louise Hedricks.

darken the solution to the point of opacity (Sax, 1968). The reclamation procedures are as follows:

1. Shake the darkened heavy liquid with a 5-10 percent solution (approximately 20 percent by volume) of sodium hydroxide (NaOH) and let it stand for 24 hours. Sodium thiosulfate and sodium bicarbonate (5-10 percent solution) have also been suggested as possible substitutes for NaOH. These chemicals do an adequate job, are not as strong as the hydroxide base, and increase the longevity of the heavy liquid (R. Doll, oral commun., 1978). Be aware that sodium hydroxide is itself a hazard and should be treated with caution.
2. Put the mixture into a separatory funnel. Allow the bromoform and NaOH (or whatever solution is used) mixture to separate. Draw off the heavy liquid.
3. Repeat the first two steps until the bromoform is transparent.
4. Wash the heavy liquid in the water cascade system for 5 minutes. This will remove any solvent.
5. Dry the bromoform by introducing several grams of calcium chloride ($CaCl_2$) per 100 milliliters of heavy liquid; shake the mixture and allow it to set for several hours. This "dewatering" process will increase the density (Browning, 1961; Benjamin, 1971).

6. Filter out the $CaCl_2$ and add 1-1.5 percent ethyl alcohol (Ijlst, 1973), or 0.05 percent propylene oxide (R. Doll, oral commun., 1978) as a preservative. This will prevent the heavy liquid from darkening again prematurely. The preservative can be removed by washing with water and drying with $CaCl_2$. The slight loss in specific gravity (commonly from 2.89-2.85) is a small sacrifice relative to the increased longevity of the bromoform. A copper wire mesh can also be used as a stabilizing agent. However, this mesh is less effective and introduces metal contamination which can affect elemental analysis results. Bromoform that is removed directly from the evaporator, washed in the water cascade system, and which is not darkened by decomposition products can be directly stabilized without going through steps 1-5.

METHYLENE IODIDE—Recovery and stabilization

Methylene iodide is darkened by the presence of free iodine (Sax, 1968) giving a characteristic blood-red color. Due to hydrolysis (O'Connell, 1963), HI is formed in the solution.

The reclamation process for MEI is similar to that of bromoform (Browning, 1961) with one exception. In stabilizing, instead of introducing chloroform or one of the other additives, a small amount of metallic copper or silver, in the form of wire mesh, is added to each bottle. Again, beware of contamination from the metals. (Ijlst, 1973; Cuttitta and others, 1960).

An additional reclamation procedure is suggested by James Svoboda (written commun., 1979). Wash the discolored MEI with a 5- to 10-percent solution of sodium sulfide, dry with calcium chloride, and stabilize with a small piece of copper or silver mesh.

TETRABROMOETHANE—Recovery and stabilization

The decomposition of this organic compound is similar to that of bromoform and the same reclamation methods can be utilized.

An additional reclamation procedure, discussed in Patty (1962), is as follows:

1. Wash the darkened solution three times with 12 percent, by volume, concentrated H_2SO_4 and three times with water.
2. Dry with potassium carbonate and calcium sulfate and filter. Specific amounts were not cited in the source references; however, several grams per 100 ml should be sufficient. (Potassium carbonate is considered a strong caustic with a moderate rating for external exposure and ingestion (Sax, 1975). The appropriate precautions should be taken when using it.)

CLERICI COMPOUNDS—Recovery and stabilization

Clerici solution, as previously described, is a mixture of thallium salts dissolved in distilled water. These salts can be recovered by evaporation in a sand or water bath. Either of these baths is recommended over evaporation on a hot plate as the temperature is more controllable in a bath. The evaporation process requires a controlled temperature because the salts will decompose if subjected to temperatures in excess of 130°C (Browning, 1961). The process of evaporation requires scrutiny as the salts can be decomposed if heated past the point of complete dryness (Benjamin, 1971).

Clerici solutions, however, are much more stable than the halogenated hydrocarbons but, if left standing for a long period of time, a dark residue of decomposed thallium salts may form. This is reclaimed by the following procedure:

1. Add a small amount of formic acid to the solution and evaporate just to dryness. This will volatilize any remaining acid.
2. Add distilled water to the dried compound and again evaporate. Do not overdry. (Browning, 1961.)

FULLER'S EARTH RECLAMATION

A method that is sometimes useful for organic heavy-liquid reclamation is to filter the liquid through fuller's earth or kaolin powder (Griffitts and Marranzino, 1960). The method is as follows:

1. Shake a few grams of fuller's earth with 100 ml of discolored heavy liquid. Pour the mixture into a separatory funnel and allow the residue to rise.
2. Draw off the clear liquid and filter. A minimal amount of fuller's earth must be used in order to avoid excessive loss of the heavy liquid by adsorption into the fuller's earth. A bonus is that the fuller's earth will preferentially absorb any water that happens to be present in the solution. The saturated powder should be discarded in a closed container. It cannot be reused.

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APPENDIX A

Properties of Some Less-Common Dilutants

The following solvents are suggested by various sources as possible dilutants for specialized heavy liquids. Upon closer examination of their properties, however, it becomes apparent that, in general, they are more toxic than acetone and less easy to handle. Great caution is advised in their use.

BENZENE (benzol, phenyl hydride)

Chemistry:	C_6H_6
Physical properties:	
Description:	Colorless liquid.
Molecular weight:	78-1
Flash point:	6°C
Density:	0.879 at 20°C
Source references:	Sax (1978); McElwee, (1979).

Toxicity

TLV 10 ppm, considered highly toxic.

Exposure

Benzene can easily penetrate the skin, inducing a high amount of irritation.

Heavy exposure can cause respiratory paralysis, narcosis, and failure of the central nervous system. Benzene is a carcinogen of the blood-forming tissues, and repeated blood examination is necessary for those who may have been exposed.

Hazards

Benzene is highly flammable, and reacts vigorously with oxidizing agents. Adequate ventilation is imperative.

CARBON TETRACHLORIDE (tetrachloromethane, perchloromethane)

Chemistry:	CCl_4
Physical properties:	
Description:	Colorless liquid.
Molecular weight:	153.8
Boiling point:	76.8°C
Flash point:	None.
Density:	1.597 at 20°C
Source reference:	Sax (1978).

Toxicity

TLV 10 ppm, considered highly toxic.

Exposure

The initial sign of exposure is gastronomic upset. CCl_4 has a narcotic reaction, having markedly varying individual susceptibility. Repeated exposure can cause severe liver, kidney, and lung damage. Heavy exposure may result in death due to respiratory failure.

Hazards

Carbon tetrachloride is nonflammable; however, if it is heated to decomposition, it emits flammable and toxic fumes of phosgene.

DIMETHYL SULFOXIDE (DMSO)

Chemistry:	$(CH_3)_2SO$
Physical properties:	
Description:	Clear, water-white, hygroscopic liquid.
Molecular weight:	78.1
Melting point:	18.5°C
Flash point:	189°C
Density:	1.100 at 20°C
Source references:	Sax (1978); Zussman (1977).

Toxicity

TLV not assigned.

Exposure

Dimethyl sulfoxide freely penetrates the skin, causing redness, burning, and general irritation. Systematic exposure can cause chills, nausea, and cramps. In some cases, an anaphylactic or allergic reaction may take place. In laboratory experimentation it has caused corneal opacity.

Hazards

DMSO is moderately flammable, and may react vigorously with oxidizing materials.

ISOAMYLACETATE (amyl acetate, banana oil)

Chemistry:	$CH_3COO(C_5H_{11})$
Physical properties:	
Molecular weight:	130.2
Boiling point:	142.0°C
Flash point:	10.7°C
Density:	0.876
Source reference:	Sax (1978).

Toxicity

TLV 50 ppm or 700 mg/m^3 air.

Exposure

Exposure to more than 1,000 ppm in an hour results in the following symptoms: headache, fatigue, pulmonary irritation.

Hazards

Isoamylacetate is highly flammable, and may react vigorously with reducing materials.

N,N-DIMETHYL FORMAMIDE (DMF, N,N dimethyl formic acid)

Chemistry:	$(CH_3)_2NHCO$
Physical properties:	
Description:	Colorless, mobile liquid, (fishy odor).
Molecular weight:	73.1
Boiling point:	152.8°C
Density:	0.9445
Source references:	Sax (1978); American Mutual Insurance Alliance (1972); McElwee (1979).

Toxicity

Assigned TLV of 10 ppm.

Exposure

DMF is extremely irritating, and can be absorbed through the skin. A fishy odor may warn of overexposure, with symptoms being: itching, nausea, and loss of equilibrium. Prolonged inhalation may cause liver and kidney damage.

Hazards

DMF is moderately flammable, and can be extinguished by dry chemical or CO_2 fire extinguishers. According to Sax (1978), contact with DMF and halogenated hydrocarbons and inorganic nitrates should be avoided.

ORTHODICHLOROBENZENE (o-dichlorobenzene)

Chemistry:	$C_6H_4Cl_2$
Physical properties:	
Description:	Clear liquid.
Molecular weight:	140.0
Melting point:	-17.5°C
Boiling point:	180-183°C
Flash point:	52°C
Density:	1.307 at 20°C
Source references:	Sax (1978); Zussman (1977); American Mutual Insurance Alliance (1972).

Toxicity

The TLV set by the ACGIH is 50 ppm.

Exposure

Orthodichlorobenzene is highly narcotic, with overexposure causing cyanosis, rapid breathing and, in more extreme cases, kidney and liver failure. It is also an irritant to the skin and mucous membranes, with slight exposure causing nasal and eye irritation.

Hazards

Orthodichlorobenzene is moderately flammable, and may react vigorously with oxidizing materials.

TRIETHYLORTHOPHOSPHATE (triethyl phosphate, TEP)

Chemistry:	$(C_2H_5)_3PO_4$
Physical properties:	
Description:	Colorless liquid, completely miscible in water.
Molecular weight:	182.2
Melting point:	-56.4°C
Boiling point:	216°C
Flash point:	101.2°C
Weight per gal:	8.9 lbs
Density:	1.067
Source reference:	Sax (1978).

Toxicity

The toxicity is unknown, but this substance should be considered hazardous until proved otherwise.

Exposure

Exposure may cause nerve injury, and TEP is known to be a cholinestrase inhibitor.

Hazards

TEP is labeled a poison. It is slightly flammable, and may react vigorously with oxidizing materials.

APPENDIX B

Procedure for the use of a Buchler Flash Evaporator Model PTFE-1gn

1. Check the stability of the apparatus. Make sure all holding clamps are tight. The glassware components are extremely expensive!
2. Seal all the glass joints with stopcock grease. This will greatly improve the efficiency of the vacuum.
3. Fill the evaporating pan approximately half full of water and submerge the heating coil. Never apply voltage to the coil unless it is immersed in water.
4. Fill the evaporating flask with approximately 1,000 ml of solvent-heavy liquid mixture and assemble it on the apparatus. Be sure to check all glassware for flaws, stars, or cracks. Applying a vacuum to a damaged flask could result in an implosion.
5. Put the condensing flask on the spiral condenser. Support it with a Thomas clamp and a ring stand. As evaporation takes place the balance of the unit will be shifted to the condenser side, necessitating this additional support.
6. Close the vacuum release valve and the continuous-feed stopcocks and apply a vacuum. A closed vacuum pump seems to give the best results. A water aspirator system was experimented with but a loss of solvent and accidental, but consistent, introduction of water into the system occurred.
7. Turn on and adjust the thermoregulator to the desired temperature. A temperature between 35° and 40°C is recommended, but most efficient results may be obtained by experimenting with different temperatures. The optimum temperature may vary with instrument and environment.
8. Turn on the water to the spiral condenser. Adjusting the water pressure slowly will allow time to check all connections.
9. Start the rotatable motor. Make a second examination to be sure that all the glass joints are sealed and secure.
10. Continue evaporation until all of the solvent has been removed; this is indicated by no further condensation in the spiral condenser. The time required will depend on the heavy liquid to solvent ratio, but should be no greater than 30 minutes per 1,000 ml.
11. To shut off the evaporator:
    a. turn off the thermoregulator,
    b. turn off the rotatable motor,
    c. shut off the water,
    d. turn off the vacuum pump, and
    e. release the vacuum.
12. Remove the reclaimed solvent and store it. The density should be checked. If it is too high, the solution may need to be re-evaporated at room temperature to complete purification. A density greater than 0.88 for the acetone is an indication of the presence of heavy liquid as a contaminate.
13. Remove the heavy liquid and store it in a dark, glass container. See Section III for reclamation and stabilization procedures.
14. CLEAN UP THE WORK AREA!!!

Although this system is relatively safe, some simple precautions will reduce the chance of accident and injury.

Be sure the system is under a properly ventilating hood. The chance of exposure to fumes will be greatly reduced and, in the event of an implosion, the debris will be contained, as will any solvent or heavy liquid released.

Wear proper, protective laboratory gear. The use of safety goggles, rubber apron, and rubber gloves, will greatly reduce the chance of personal injury or exposure. Cloth and plastic aprons or coats are not recommended. Cloth is permeable to heavy liquids and many plastics deteriorate with exposure.

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