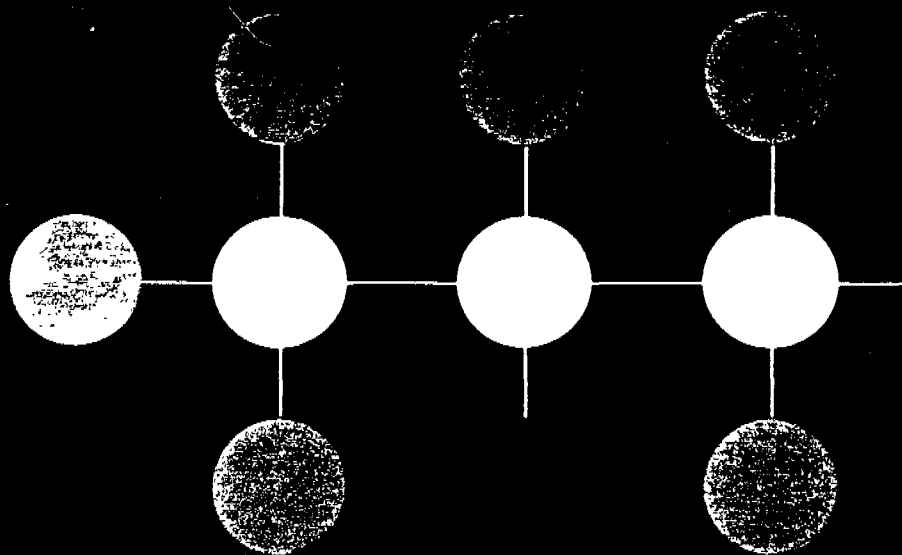
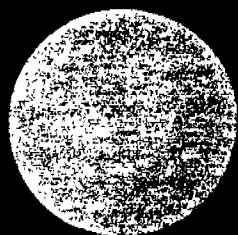


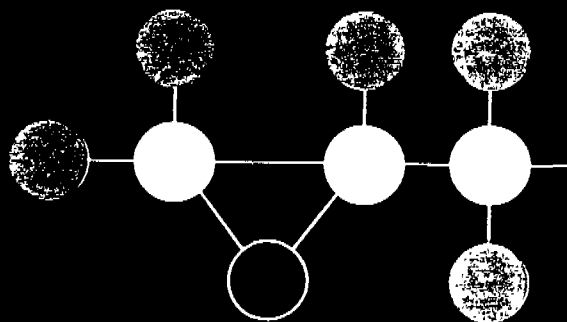


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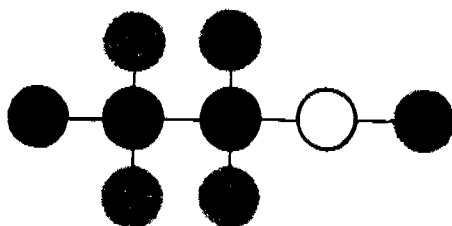
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CHLORINE

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Organic Chlorine Compounds.....	
Tables: Physical Properties.....	
Solubility Data.....	
Ethylene Dichloride.....	
CHLORASOL Fumigant and Solvent.....	
Propylene Dichloride.....	
Trichlorethane.....	
Butyl Chloride.....	
2-Ethylhexyl Chloride.....	
Dichlorethyl Ether.....	
Dichlorisopropyl Ether.....	
Triglycol Dichloride.....	
Ethylene Chlorhydrin.....	
Epichlorhydrin.....	
Advantages of Chlorinated Solvents.....	
Handling and Storage of Chlorinated Compounds.....	
Physiological Properties.....	
Shipping Data.....	
Specifications and Test Methods.....	
Constant Boiling Mixtures.....	
Physical Property Charts.....	
Bibliography.....	
Other Chemicals Produced by CARBIDE.....	
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Division of

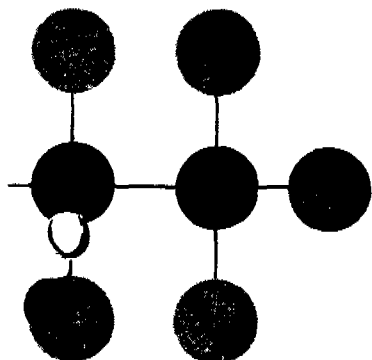


Corporation

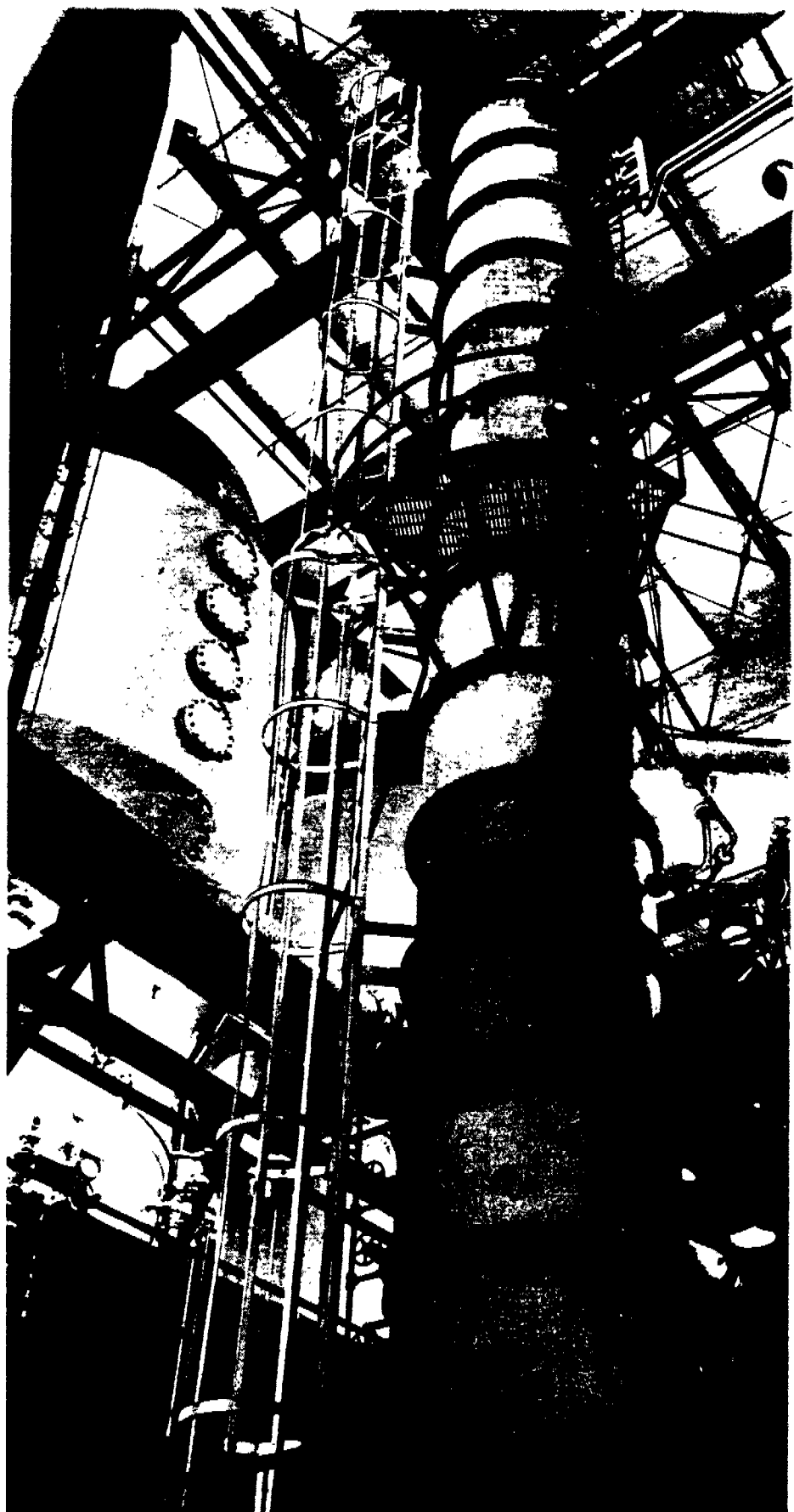
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**Organic
CHLORINE
COMPOUNDS**



This equipment is used to process organic chlorine compounds and their derivatives at CARBIDE's South Charleston, West Virginia plant.

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Most chlorine compounds are stable, non-flammable liquids. They find many applications as solvents, extractants, intermediates, and fumigants.

Organic chlorine compounds generally have a powerful solvent action for oils, fats, waxes, greases, gums, and resins but are insoluble in water. They are good non-polar solvents for many hydrocarbons, alkaloids, and synthetic rubber and only partially dissolve highly polar derivatives such as sugars, proteins, inorganic acids, and glycerol. The addition of a small amount of ethanol to many chlorinated hydrocarbons produces an active solvent mixture for cellulose nitrate.

The lower boiling chlorinated solvents are useful as extractants. Their narrow boiling range and low boiling points permit their easy recovery from the extracted materials.

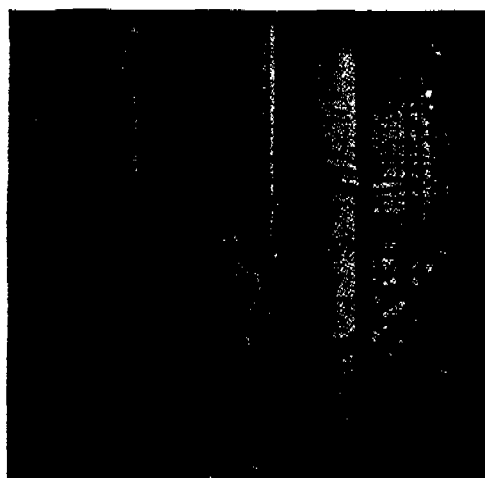
Organic chlorine compounds are used in the synthesis of pharmaceuticals, insecticides, synthetic rubbers, and resins. Some chlorine compounds are effective fumigants.

CARBIDE offers 11 organic compounds in commercial quantities. This book contains information on their uses, physical and physiological properties, shipping regulations, handling and storage, and specifications and test methods. This information will be of value to purchasing agents, management personnel, and research chemists. Research groups will particularly find this book of interest in improving standard products and developing better ones to expand present markets or open new ones. Samples are available for laboratory investigations.

It is not possible to mention in this book all the applications of these organic chlorine compounds. Technical Representatives located in 28 principal industrial cities throughout the country have the necessary knowledge and experience to assist you with the development of these chemicals. They can also aid purchasing agents and production engineers in day-to-day problems of shipping and storing these and other organic chemicals.

Routine check, as shown here at an organic chlorine production still, helps maintain consistent standards of high quality.

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PHYSICAL PROPERTIES

	Molecular Weight	Specific Gravity, 20°/20°C.	$\frac{\Delta \text{ sp. gr.}}{\Delta t.}$	Boiling Point, °C.		
				760 mm.	50 mm.	10 mm.
Ethylene Dichloride	98.97	1.2554	0.00146	83.5	15	-13
Propylene Dichloride	112.99	1.1583	0.00131	96.3	25	- 5
1,1,2-Trichlorethane	133.42	1.4432	0.00154	113.7	41	11
Butyl Chloride	92.57	0.8875	0.00111	78.6	10	-18
2-Ethylhexyl Chloride	148.67	0.8833	0.00086	173.0	89	55
Dichlorethyl Ether	143.02	1.2220	0.00118	179.2	96	64
Dichlorisopropyl Ether	171.07	1.1135	0.00107	187.0	104	68
Triglycol Dichloride	187.07	1.1974	0.00110	240.9	151	114
Ethylene Chlorhydrin	80.52	1.2045	0.00107	128.7	60	32
Epichlorhydrin	92.53	1.1761	0.00120	115.2	45	16

S LUBILITY DATA

	BAKELITE Vinyl Resins		Cellulose Acetate	Nitro-cellulose
	AYAF	VYHH		
Ethylene Dichloride	S	S	SA	SA
Propylene Dichloride	S	S	I	SA
1,1,2-Trichlorethane	S	S	E	E
Butyl Chloride	S	SS	I	I
2-Ethylhexyl Chloride	I	I	I	I
Dichlorethyl Ether	S	S	SA	SA
Dichlorisopropyl Ether	S	SW	I	I
Triglycol Dichloride	S	SW	PSA	SA

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ETHYLENE DICHLORIDE

(Ethylene Chloride; 1,2-Dichlorethane)



Ethylene dichloride's excellent solvent power and resistance to hydrolysis make it useful as an extractant for oils and fats. Vegetable oils are effectively extracted from expeller cakes through the use of ethylene dichloride. Ethylene dichloride is used to concentrate vitamins A and D from cod and shark liver oils. In this way, odorless and tasteless products are prepared. Its boiling point is high enough to permit the use of low-pressure equipment. Ethylene dichloride is also useful in the vapor-phase degreasing of metal articles.

An excellent solvent for all standard low-viscosity types of cellulose acetate (up to 61 per cent combined acetic acid) is a 9 to 1 mixture of ethylene dichloride and ethanol. Ethylene dichloride is also a valuable solvent in compounding polysulfide rubbers and Buna N synthetic rubbers for cements and coatings.

Ethylene dichloride is a useful scouring and spotting agent for the removal of tar, paint, and brand marks from wool. When incorporated into soaps and scouring compounds, it assists in the removal of grease. Wetting-out and penetrating compounds can also incorporate ethylene dichloride. Its solvent action on the natural waxes assists in the degumming of silk and cotton.

Ethylene dichloride lends itself readily to a number of valuable syntheses, as both chlorine atoms can be replaced by other groups. Such an example is its condensation with sodium cyanide to form succinonitrile, followed by hydrolysis with aqueous hydrochloric acid to produce succinic acid. With polysulfides, oil and gasoline-resistant rubber-like materials are formed. Ethylene polysulfide, a useful polymer lubricant, is prepared from ethylene dichloride and sodium polysulfide.¹ Vinyl chloride is produced from ethylene dichloride and aqueous caustic at an elevated temperature and pressure.²

The condensation products of dioleyl- and dilauryldiethylene triamine with ethylene dichloride are useful antifoaming agents for steam boilers and generators.³

Literature Cited

- ¹ B. A. Perkins (to Rockwell Manufacturing Co.) U.S. Patent 2,474,859 (July 5, 1949)
- ² R. J. Koll (to Diamond Alkali Co.), U.S. Patent 2,539,307 (January 23, 1951)
- ³ P. G. Bird, A. L. Jacoby (to National Aluminate Corp.), U.S. Patent 2,580,880 (January 1, 1952)

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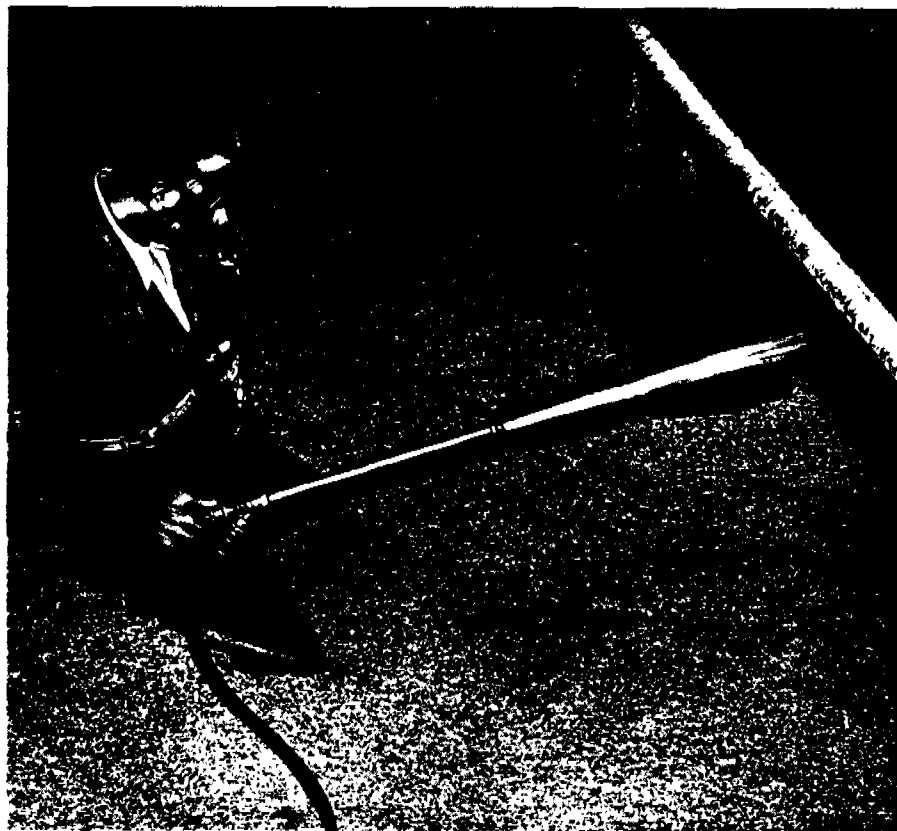
"CHLORASOL" FUMIGANT AND SOLVENT

(Solvent EDM)

CHLORASOL fumigant and solvent is a mixture containing 75 per cent by volume ethylene dichloride and 25 per cent by volume carbon tetrachloride. It is not flammable under conditions of ordinary use.

CHLORASOL solvent readily dissolves oils, waxes, gums, and tars, and is an active solvent component in industrial cleaning compounds, type cleaners, spotting solvents, and similar specialty products. It is also used in cleaning textiles and in removing oils and greases from machinery.

CHLORASOL fumigant is an effective fumigant for grain, and clothes moths; grain weevils; grain, flour, and carpet beetles; the rice weevil; and book lice — as well as their larvae and eggs. Its vapors penetrate stored grain, rolled rugs, upholstered furniture, cartons, sacks, and stacked material. It is non-flammable under ordinary conditions of use and evaporates quickly at a room temperature of 70°F. or higher. The rate of evaporation



Fumigating grain stored in the hold of a grain transport. CHLORASOL fumigant is effective in preventing grain infestation during storage in grain transports, bins, and silos.

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and the depth of penetration increase as the temperature increases. CHLORASOL fumigant is fast acting and is quickly and easily removed by adequate ventilation.

CHLORASOL fumigant can be applied either directly to grain in the storage bin, grain car, or silo or by mixing it with the stream of grain coming into the terminal elevator. When fumigating grain, the storage bins and elevators must be reasonably airtight for the fumigant to be completely effective. Individual sections of mill machinery can be fumigated by spraying or pouring the liquid onto the grain as it is fed into the machine. In this way, the sections likely to be infested can be individually treated. When used in accordance with directions, residues of CHLORASOL fumigant on treated grain are exempt from the tolerance requirements of the Federal Food, Drug, and Cosmetic Act.

In buildings and warehouses, CHLORASOL fumigant is particularly suited for the fumigation of various commodities that have become infested while in storage. It may be used simply yet effectively by pouring the liquid into shallow pans or trays near the ceiling of the room to be fumigated. In more permanent installations, the liquid can be piped into the trays from storage tanks.

Furniture can be satisfactorily fumigated with CHLORASOL fumigant in an atmospheric vault. Fabrics and furs are fumigated in airtight vaults and when properly used, CHLORASOL fumigant has no effect on the hair, pelts, dyes, buttons, or other parts of the garment. CHLORASOL fumigant is one of the few effective fumigants that can be safely used on seeds. Exposures up to 72 hours have no effect upon seed germination.

PROPYLENE DICHLORIDE

(1,2-Dichloropropane)



Propylene dichloride is used as an extraction solvent where its high boiling point offers special processing advantages. Its stability, ease of recovery, and solvent characteristics make it valuable for the preparation of highly concentrated lactic acid. Propylene dichloride is used in the preparation of cleaning and scouring compounds, spot-removing agents, and in the pyrolytic production of chlorhydrocarbons.¹ It is also an intermediate in the manufacture of alcohols, amines, nitriles, and acids.

Literature Cited

¹ E. K. Morris (to Dow Chemical Co.), U.S. Patent 2,588,867 (March 11, 1952)

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Many of the organic chloride compounds such as propylene dichloride are used in specialty dry cleaning formulations.

1,1,2-TRICHLORETHANE

(1,1,2-Trichloroethane)



Trichlorethane's excellent solvent properties make it a valuable component of spotting or cleaning fluids. It is also a suitable freezing-point depressant in fire extinguisher fluids based on carbon tetrachloride.

Vinylidene chloride ($\text{CH}_2 = \text{CCl}_2$), which can be polymerized to form a valuable group of resins, is prepared by splitting hydrogen chloride from trichlorethane.¹

Literature Cited

¹ J. L. Amos (to Dow Chemical Co.), U.S. Patent 2,610,214 (September 9, 1952)

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Butyl chloride is an effective deworming agent in veterinary medicine.

BUTYL CHLORIDE

(1-Chlorobutane)



2-ETHYLHEXYL CHLORIDE



Both butyl chloride and 2-ethylhexyl chloride are used as alkylating agents for the modification of phenols, amines, cellulose, starch, and other materials. Such modification usually leads to derivatives with greater oil and organic solvent solubility and lower water solubility than the parent unalkylated compound. These derivatives are of value in extraction processes and as petroleum additives and plasticizers.

Butyl chloride is an effective anthelmintic (deworming agent) in veterinary medicine. It is used in the manufacture of tin stabilizers for vinyl resins.

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2-Ethylhexyl chloride is used as an instrument fluid because of its clarity, low viscosity, high boiling point, and low vapor pressure at normal temperatures. It is particularly useful in aircraft instruments because of its extremely low freezing point ($-135^{\circ}\text{C}.$) and its stability over a wide temperature range. 2-Ethylhexyl chloride is also an intermediate for various surface active agents of the alkyl-aryl sulfonic acid type.

DICHLORETHYL ETHER

[2,2'-Dichloroethyl Ether, Di(2-Chloroethyl) Ether]



Dichlorethyl ether is a colorless liquid with a chloroform-like odor. It is extremely resistant to hydrolysis. This chlorinated ether is a particularly good solvent for fats, waxes, or greases.

The solvent action of dichlorethyl ether makes it a useful scouring agent for textiles. When used either in the scouring bath or as a pre-spotter, dichlorethyl ether aids in removing paint and tar brand marks from raw wool, and oil and grease spots from cloth. Since the solvent is retained by the goods throughout the process, hand spotting in many cases is eliminated.

Dichlorethyl ether is an intermediate in the manufacture of alkyl aryl ether sulfate or sulfonate-type ($\text{R} \langle \bigcirc \rangle \text{OC}_2\text{H}_4\text{OC}_2\text{H}_4\text{SO}_3$) anionic surface active agents.^{1,2} The advantage of the use of this chlorinated ether is its ability to introduce only two ethylene oxide linkages into the molecule and thereby controlling the hydrophobic-hydrophilic balance of the resulting detergent. These surface active agents are good emulsifiers for oils, fats, and greases and have excellent detergent, lathering, and rinsing qualities. They are also good emulsifiers for cosmetic preparations and their excellent detergent and lathering properties make them well suited for shampoo and cleansing cream bases. Metal cleaning operations are another important application for surface active agents of this type.

Dichlorethyl ether is a solvent for the separation of butadiene from butylene by extractive distillation. It is also used for the recovery of ethylene

Literature Cited

¹ H. A. Bruson (to Rohm and Haas Co.), U.S. Patent 2,098,203 (November 2, 1937)

² H. A. Bruson (to Rohm and Haas Co.), U.S. Patent 2,115,192 (April 26, 1938)

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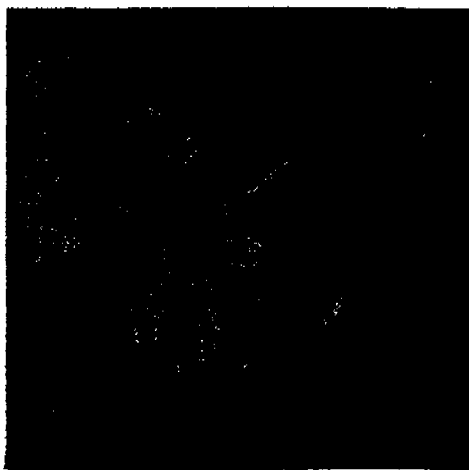
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Lotions and cleansing creams are formulated from alkyl aryl ether sulfonates derived from dichlorethyl ether.



CHLOREX solvent is used in a process for the purification of naphthenic crude oils.

oxide after the oxidation of ethylene.³ A mixture of nitrobenzene and dichlorethyl ether makes an effective solvent in the fractionation of wax-containing mixtures.⁴

Dichlorethyl ether, sold as CHLOREX solvent, is used as a selective solvent in the production of high-grade lubricants from Mid-Continent and other naphthenic crude oils.

The ability of dichlorethyl ether to scavenge lead deposits in engines makes it an important component of anti-knock compounds.⁵

This chlorinated ether is used in the syntheses of morpholine and N-substituted morpholine derivatives. It is an intermediate in the production of divinyl ether, a basic anesthetic. Dichlorethyl ether is also a raw material in the syntheses of resins and plasticizers, textile chemicals, pharmaceuticals, insecticides, rubber chemicals, and lubricating-oil additives.

³ C. J. Thomas (to Phillips Petroleum Co.), U.S. Patent 2,622,088 (December 16, 1952)

⁴ E. W. Clarke (to Atlantic Refining Co.), U.S. Patent 2,604,432 (July 22, 1952)

⁵ G. Calingaert (to Ethyl Corp.), U.S. Patent 2,496,983 (February 7, 1950)

DICHLORISOPROPYL ETHER

(2,2'-Dichlorodiisopropyl Ether)



The properties of dichlorisopropyl ether are very similar to those of dichlorethyl ether. However, this chlorinated isopropyl ether is less sol-

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Literature Cited

- ¹ P. P. Regna, I. A. Solomons (to Chas. Pfizer & Co. Inc.), U.S. Patent 2,556,375 (June 12, 1951)

uble in water, and has a higher boiling point and a lower volatility than dichlorethyl ether. It is miscible with petroleum, vegetable or animal oils, and many organic liquids. It is an excellent extractant for butadiene, fats, vitamins, waxes, and greases.

In textile processes where high temperatures are encountered, dichlorisopropyl ether assists the action of soap solutions without causing excessive loss by vaporization from the hot solutions. The excellent solvent action of dichlorisopropyl ether is advantageous in paint and varnish removers, spotting agents, and cleaning solutions. Dichlorisopropyl ether is employed as a solubility depressant in the extraction of the antibiotic bacitracin from fermentation broths.¹ It offers numerous possibilities as an intermediate in the manufacture of dyes, resins, and pharmaceuticals.

Products made from dichlorisopropyl ether are generally less soluble in water and more soluble in oil than those made from dichlorethyl ether, a difference caused by the two additional methyl groups in its molecule.

TRIGLYCOL DICHLORIDE

[2(2-Chloroethoxy) Ethyl 2'-Chloroethyl Ether,
Triethylene Glycol Dichloride]



Literature Cited

- ¹ L. Coes, Jr. (to Norton Co.), U.S. Patent 2,456,921 (December 21, 1948)

Triglycol dichloride is a water-insoluble liquid with a high boiling point. Its solvent properties are similar to those of dichlorethyl ether. Triglycol dichloride is a useful solvent and extractant because of its high solvent power for oils and hydrocarbons.

In chemical reactions, the chlorine atoms of triglycol dichloride can be replaced by other groups to form a number of compounds such as ethers, esters, thiols, and N-substituted amines and amides. Compounds of this type are useful intermediates for the manufacture of dyes, resins, detergents, and insecticides. Triglycol dichloride is of particular interest in the manufacture of the alkyl aryl ether sulfonate type-detergents ($\text{R} \langle \bigcirc \rangle \text{OC}_2\text{H}_4\text{OC}_2\text{H}_4\text{OC}_2\text{H}_4\text{SO}_3$), similar to those described on page 11.

Triglycol dichloride is used as a cross-linking and hardening agent for aromatic amine-formaldehyde resins used in bonding abrasives to grinding wheels.¹

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ETHYLENE CHLORHYDRIN

(2-Chloroethanol)



The principal use of ethylene chlorhydrin is for the introduction of the hydroxyethyl group ($-\text{CH}_2\text{CH}_2\text{OH}$) into other organic compounds. Resulting derivatives boil about 100°C . higher than the corresponding ethyl compounds and are more soluble in water. Ethylene chlorhydrin reacts with the salts of organic acids to form glycol esters; with sodium cyanide, it yields ethylene cyanohydrin which can be dehydrated to acrylonitrile. Ethylene cyanohydrin can also be hydrolyzed to *beta*-hydroxy propionic acid which in turn can be oxidized to malonic acid or dehydrated to acrylic acid. Ethylene chlorhydrin reacted with brombenzene, by means of the Grignard reaction, produces phenylethanol (synthetic oil of rose).

Ethylene chlorhydrin is also used in the synthesis of choline (2-hydroxyethyl trimethylammonium hydroxide) a member of the Vitamin B group, which is generally considered to be an essential ingredient in chicken feed to aid in the production of eggs. The reaction of ethylene chlorhydrin and thiophenol produces phenyl vinyl sulfone.¹ Sodium terephthalate and ethylene chlorhydrin can be refluxed with sodium iodide and heated in a stream of hydrogen to produce polyesters.² The isolation of styrene may be accomplished by azeotropic distillation with ethylene chlorhydrin.³

Literature Cited

- ¹ E. F. Landau, E. P. Irany (to Celanese Corp. of America), U.S. Patent 2,554,576 (May 29, 1951)
- ² J. G. Napier (to Celanese Corp. of America), U.S. Patent 2,551,732 (May 8, 1951)
- ³ K. H. Engel (to Allied Chemical and Dye Corp.), U.S. Patent 2,465,717 (March 29, 1949)



Ethylene chlorhydrin is an intermediate for choline, a component of the vitamin B complex which is essential to the liver function and good egg production.



CARBIDE's Research and Development Center at South Charleston, West Virginia—the birthplace of many new chemicals—was first completed in 1949. A new Development and Engineering Center is being built adjacent to present extensive research facilities.

ADVANTAGES OF CHLORINATED SOLVENTS

Non-Flammability One of the chief values of chlorinated solvents is their low order of flammability. Their fire hazard is small as shown by these comparative ratings of the Underwriters' Laboratories Standard of Classification:

Trichlorethane.....	3	Non-Flammable at Ordinary Temperatures
Kerosene (100°F. Flash).....	30 to 40	Moderately Flammable
Ethylene Dichloride.....	60 to 70	Moderately Flammable
Ethanol.....	60 to 70	Moderately Flammable
Gasoline.....	90 to 100	Flammable
Ethyl Ether.....	100	Flammable

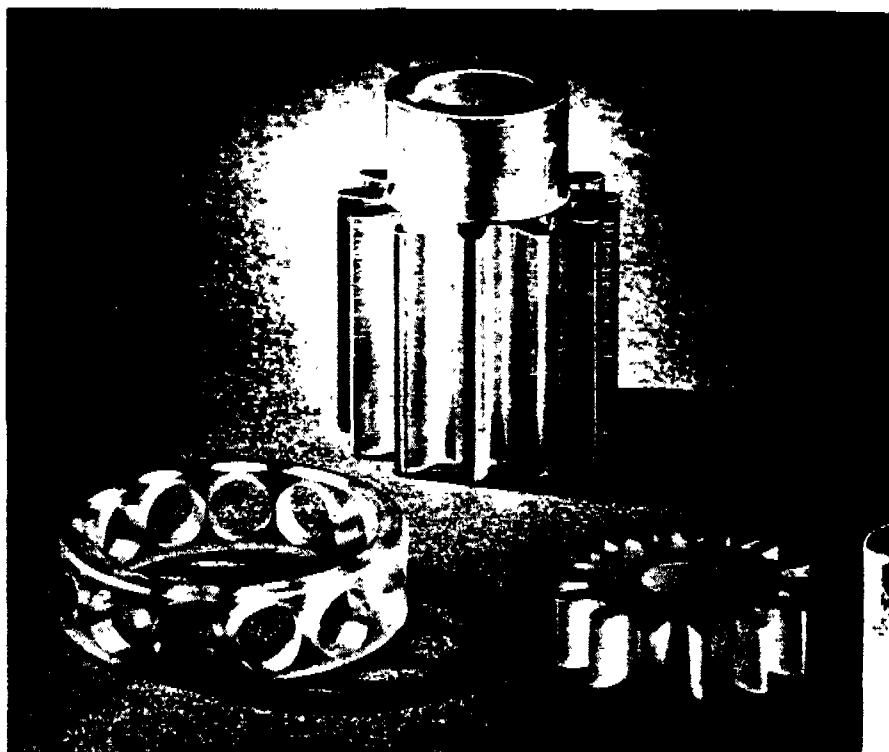
Ethylene dichloride and propylene dichloride will burn, but only with difficulty. A much greater concentration is required in the air to form an explosive mixture with them than with other solvents:

	<u>Explosive Limit</u>
Benzene.....	1.41% in air, by volume
Gasoline.....	1.5% in air, by volume
Ethyl Ether.....	1.71% in air, by volume
Ethylene Dichloride.....	6.2% in air, by volume

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Epichlorhydrin is an intermediate in the manufacture of tough, durable epoxy resin castings and embedding materials.



▲ Epoxy resin gear moldings.

Radio tube embedded in an epoxy resin. ►



EPICHLORHYDRIN

(3-Chloro-1,2-Propylene Oxide, 1-Chloro-2,3-Epoxypropane)



Epichlorhydrin's reactive chlorine and epoxide groups and its complete miscibility with ethers, alcohols, acetone, benzene, acetates, and carbon-tetrachloride make it a useful raw material for a wide variety of syntheses. The epoxide group combines exothermically with the active hydrogen atoms in many alcohols, phenols, carboxylic acids, amines, and mercaptans. The chlorine atom reacts with amines, amides, acid salts, and alkali metal phenolates and alcoholates.

Epichlorhydrin is a primary raw material in the manufacture of epoxy resins — important materials for adhesives, surface coatings, resinous castings for tools and dies and for "potting" electrical components. In the

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manufacture of these epoxy resins, epichlorhydrin is condensed with dihydric phenols^{1,2} or phenolic resins³ to yield materials ranging from liquids to solids depending on the ratios of epichlorhydrin to the phenol component. The resins may then be converted by amine* or acid curing agents to strong, tough, durable, and solvent and moisture resistant solids.

Condensates of epichlorhydrin with polyethylene amines such as diethylene triamine and tetraethylene pentamine also yield resinous materials. These are useful as ion exchange resins,⁴ resins for improving the wet strength of paper,⁵ and resins for promoting the adhesion of lacquers to cellulose films.⁶

Epichlorhydrin is an intermediate in the manufacture of an anti-spasmodic⁷ and other pharmaceuticals, detergents and wetting agents,^{8,9,10} and textile softeners. It is used in the preparation of dyestuff intermediates, plasticizers, and stabilizers. It is used directly as a stabilizer for chlorinated solvents and insecticides and chlorine containing polymers. Epichlorhydrin is a raw material for the manufacture of a host of other chemicals.

Literature Cited

- ¹ P. Castan: U.S. Patent 2,324,483 (July 20, 1943)
- ² M. DeGroote (to Petrolite Corp., Ltd.) U.S. Patent 2,076,624 (April 13, 1937)
- ³ M. Steindorff, B. Balle, G. Horst, R. Michel (to General Aniline and Film Corp.), U.S. Patent 2,213,477 (September 3, 1940)
- ⁴ J. R. Dudley and L. A. Lundberg (to American Cyanamid Co.) U.S. Patent 2,469,683 (May 10, 1949)
- M. W. Michael (to American Cyanamid Co.) U.S. Patent 2,543,666 (February 27, 1951)
- ⁵ J. H. Daniel, Jr. and C. G. Landes (to American Cyanamid Co.) U.S. Patent 2,595,935 (May 6, 1952)
- ⁶ J. H. Daniel, Jr. and C. G. Landes (to American Cyanamid Co.) U.S. Patent 2,573,956 (November 6, 1951)
- ⁷ H. L. Yale et. al., J. AM. CHEM. SOC. 72, 3710-16 (1950)
- ⁸ N. B. Tucker (to the Procter and Gamble Co.) U.S. Patent 2,289,391 (July 14, 1942)
- ⁹ A. S. Richardson (to the Procter and Gamble Co.) U.S. Patent 2,383,737 (August 28, 1945)
- ¹⁰ N. B. Tucker (to the Procter and Gamble Co.) U.S. Patent 2,334,517 (November 16, 1943)

*Available from Union Carbide Chemicals Company.

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The presence of ethylene dichloride in the air can be detected by its characteristic sweet, ethereal odor long before its concentration approaches 6.2 per cent. The addition of carbon tetrachloride renders ethylene and propylene dichlorides safe, with regard to fire hazard, under ordinary conditions of storage or use. A mixture of 75 per cent ethylene dichloride and 25 per cent carbon tetrachloride by volume is classed as "Non-Flammable" by Underwriters' Laboratories and by the U. S. Bureau of Explosives. This mixture is not approved for use as a fire-extinguishing liquid.

Stability Ethylene dichloride has the lowest acidity specification and is the most stable of the chlorinated hydrocarbons. It is *resistant to hydrolysis and oxidation* in the presence of air, water, or metals at temperatures up to 100°C. In the absence of air and water, it is stable up to 160°C. Trichlorethane is stable in the presence of air at ordinary temperatures and in the absence of air and water up to 110°C.

The following table summarizes the resistance of various chlorinated solvents to loss of chlorine in aqueous solutions of acids, bases, and reducing and oxidizing agents. In comparing the figures given, only large differences should be considered significant. Inhibitors can be added to chlorinated solvents to minimize the development of acidity. These inhibitors are usually weak nitrogen bases such as the pyridines.

Comparative Stability

Chlorinated Compound	8.4% Sodium Bicarbonate	6% Acetic Acid	Iron Powder	2% Hydrogen Peroxide
Mg. of Chloride ion per 100 ml. of Compound Refluxed for 48 Hours:				
Ethylene Dichloride	1732	55	30	342
Propylene Dichloride	225	12	64	188
Trichlorethane	2160	23	1982	106.4
Dichlorethyl Ether	348	694	78	632
Dichlorisopropyl Ether	135	284	71	320
Triglycol Dichloride	263	557	99	895
Butyl Chloride	293	145	64	167
Ethylene Chlorhydrin	3080	2160	650	1188
Carbon Tetrachloride	14	10	1860	9

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Ease of Recovery Narrow boiling range and complete volatility simplify the removal of chlorhydrocarbons from extracted material and facilitate economical recovery of the solvent. No "heavy-ends" are left in the extract or in the residue. Boiling range and heat of vaporization of chlorhydrocarbons are low enough to permit easy distillation without using excessive temperatures and to give minimum loss on handling.

HANDLING AND STORAGE OF CHLORINATED COMPOUNDS

In using organic chlorine compounds, particular attention must be given to adequate ventilation. Prolonged or repeated breathing of vapors or contact with skin and eyes must be avoided. Additional precautions may be necessary with particular compounds. Physiological properties and toxicity information are given on pages 20 and 21. From the data given on page 17 in the section on "Advantages of Chlorinated Solvents", it is evident that chlorinated solvents have a low order of flammability and their fire hazard is small.

These compounds react in varying degrees with different metals. For safety reasons and to avoid contamination, care must be taken in the selection of storage containers and piping, pumps, and other processing equipment.

Steel drums or tanks coated with a baked phenolic resin have been found to be the most satisfactory type of storage container for chlorinated compounds. Butyl chloride and ethylene chlorhydrin should be stored *only* in these resin coated containers. Aluminum should never be used for the storage of chlorinated compounds.

When freshly distilled and of a low acid content, most of the chlorinated compounds can also be stored in containers constructed of mild steel, 18-8 stainless steel, nickel, galvanized iron, or tinned iron. If the acidity increases, however, due to prolonged storage, the rate of attack on unprotected metals may be appreciable with subsequent contamination of the solvent.

Steel piping is generally satisfactory for the transfer of chlorinated compounds. If corrosion of the lines or discoloration of the material are to be avoided during prolonged storage or recycling, then the lines should be drained after use. If this procedure proves impractical, baked phenolic resin coated steel pipes are recommended to minimize contamination.

Additional information on the storage and handling of chlorinated compounds may be found in the following Manufacturing Chemists' Association pamphlets — Ethylene Dichloride SD-18 and Carbon Tetrachloride SD-3.

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PHYSI LOGICAL PROPERTIES

These organic chlorine compounds differ greatly from one another in their toxicity as indicated by the table on the right. As a general rule, the major hazard in their industrial applications and use is from vapor inhalation. Penetration through the skin by those compounds with a single skin absorption dose for rabbits of 1 milliliter per kilogram or less is hazardous.

None of these chlorine compounds is highly irritating to the skin, but like many solvents, they would have a solvent action on the skin. Additional information on physiological properties may be obtained from the literature referred to on page 42. Handling and storage of chlorine compounds are discussed on page 19.

Toxicity is only one indication of the existence of hazard in handling a chemical. Other factors, such as physical properties, and extent of exposure are equally important in determining the hazard.

These tabular data on the next page indicate relative toxicity to animals. The results of animal experiments may be indicative of the effects to be expected on human subjects, but they cannot be freely applied to humans.

Definitions:

- The National Research Council defines toxicity as the capacity of a substance to produce injury. Hazard is the probability that injury will result from the use of the substance in the quantity and manner proposed.

- The term, LD₅₀ refers to that quantity of chemical which kills 50 per cent of exposed animals. For uniformity, dosage is expressed in grams or milliliters per kilogram of animal body weight.

- Single skin absorption refers to a 24-hour skin contact with the liquid chemical.

- Single inhalation refers to a single continuous breathing of a certain concentration of chemical for a given period of time.

- Primary irritation refers to the skin response following a 4-hour uncovered skin exposure.

- Eye injury refers to surface damage produced by the liquid chemical.

- The word poison is used as defined by the Manufacturing Chemists' Association and the Interstate Commerce Commission.

Legal responsibility is assumed only for the fact that all studies reported here and all opinions are those of qualified experts.

Ethylene Dichloride

CHLORASOL Fumigant and Solvent (Solvent EDM)

Propylene Dichloride

**** 1,1,2-Trichlorethane**

Butyl Chloride

2-Ethylhexyl Chloride

Dichlorethyl Ether

CHLOREX Solvent

Dichlorisopropyl Ether

Triglycol Dichloride

***Ethylene Chlorhydrin**

***Epichlorhydrin**

*** Poison.**

**** Not to be confused with methyl chloroform (1,1,1-trichlorethane).**

Single Oral LD ₅₀ Dose Rats g./kg.	Single Skin Absorption, LD ₅₀ Dose Rabbits ml./kg.	Single Inhalation Rats Saturated Vapors	Primary Irritation, Rabbits' Skin	Eye Injury, Rabbits
0.77	3.89	200 ppm., 1 hr. killed 1 of 10.	None	Minor
1.64	5.99	2 min. killed 1 of 6. 5 min. killed 6 of 6.	None	Minor
2.27	8.75	15 min. killed 1 of 6. 30 min. killed 3 of 6. 1 hr. killed 6 of 6.	None	Minor
1.14	3.73	500 ppm., 4 hrs. killed 1 of 6. 8 hrs. killed 4 of 6.	Minor	Minor
2.67	20 ml./kg. killed 1 of 4.	15 min. killed none of 6. 30 min. killed 6 of 6.	Mild	Minor
7.34	15.8	2 hrs. killed 2 of 6. 8 hrs. killed 2 of 6.	Minor	None
0.105	0.72	1 hr. killed none of 6. 2 hrs. killed 2 of 2.	Minor	None
0.105	0.72	1 hr. killed none of 6. 2 hrs. killed 2 of 2.	Minor	None
0.24	3.00	4 hrs. killed none of 6. 8 hrs. killed 6 of 6.	None	Minor
0.25	1.41	8 hrs. killed none of 4.	Minor	None
0.089	0.105	30 sec. killed none of 6. 10 min. killed 6 of 6.	None	None
0.09	1.3	250 ppm., 4 hrs. killed none of 6. 8 hrs. killed 4 of 6.	Minor	Moderate

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SHIPPING DATA

This table presents the various data that pertain to CARBIDE's chlorinated compounds as they are shipped commercially. The table gives the pounds per gallon for each chlorinated compound, its flash point, and net container contents in pounds; and it indicates the various labels that are required by law to be on each container.

Container
contents
are subject
to change.

Chlorinated Compound	Pounds per gal. at 20°C.	Coefficient of Expansion at 55°C.	Flash Point, °F. Open Cup
Ethylene Dichloride	10.45	0.00121	70
CHLORASOL Fumigant and Solvent	11.12	0.00122	No Flash
Propylene Dichloride	9.64	0.00118	70
1,1,2-Trichlorethane	11.94	0.00082	No Flash
Butyl Chloride	7.38	0.00131	15
2-Ethylhexyl Chloride	7.33	0.00101	140
Dichlorethyl Ether	10.17	0.00100	185
CHLOREX Solvent	10.17	0.00100	185
Dichlorisopropyl Ether	9.29	0.00098	185
Triglycol Dichloride	9.97	0.00095	250
Ethylene Chlorhydrin	10.03	0.00092	140
Epichlorhydrin	9.84	0.00104	105

Net Container Contents and Type of Containers

1-gal. Can	5-gal. Drum	55-gal. Drum	Labels Required	Freight Description	Bureau of Explosives Description on Container
10.0 lb.; tin	50 lb.; ICC 17E iron drum	570 lb.; ICC 17E iron drum with "Garlock" gasket	Red	Ethylene Dichloride	Same
11.0 lb.; tin	55 lb.; ICC 17E double- coated, resin-lined iron drum	600 lb.; ICC 17E double- coated, resin-lined iron drum with "Garlock" gasket	None	Insecticides, Noibn, O/T Agricultural	None
9.5 lb.; tin	45 lb.; ICC 17E double- coated, modified phenolic resin-lined iron drum	520 lb.; ICC 17E double- coated, modified phenolic resin-lined iron drum with "Garlock" gasket	Red	Propylene Dichloride (Flammable Liquid NOS)	Flam. Liq. NOS
12.0 lb.; tin	60 lb.; ICC 17E double- coated, resin-lined iron drum	650 lb.; ICC 17E double- coated, resin-lined iron drum with fiber gasket	None	Trichlorethane	None
7.0 lb.; tin	35 lb.; ICC 17E iron drum	400 lb.; ICC 17C iron drum with fiber gasket	Red	Chemicals, Noibn	Same
7.0 lb.; tin	35 lb.; ICC 17E iron drum	400 lb.; ICC 17E iron drum with "Garlock" gasket	None	Chemicals, Noibn	None
10.0 lb.; tin	50 lb.; ICC 17E iron drum	560 lb.; ICC 17E iron drum with "Garlock" gasket	None	Dichloroethyl Ether	None
10.0 lb.; tin	50 lb.; ICC 17E iron drum	560 lb.; ICC 17E iron drum with "Garlock" gasket	None	Dichloroethyl Ether	None
9.0 lb.; tin	45 lb.; ICC 17E iron drum	510 lb.; ICC 17E iron drum with "Garlock" gasket	None	Paint and Varnish Solvent, Noibn	None
9.5 lb.; tin	50 lb.; ICC 17E double- coated, epoxy-phenolic resin-lined iron drum	550 lb.; ICC 17E double- coated, epoxy-phenolic resin-lined iron drum with polyethylene gasket	None	Paint and Varnish Solvent, Noibn	None
10.0 lb.; tin	50 lb.; ICC 17E double- coated, epoxy-phenolic resin-lined iron drum	550 lb.; ICC 17E double- coated, epoxy-phenolic resin-lined iron drum with polyethylene gasket	Poison B	Ethylene Chlorhydrin	Poison B
9.5 lb.; tin	45 lb.; ICC 17E iron drum	540 lb.; ICC 17E iron drum with fiber gasket	Poison B	Epichlorhydrin	Poisonous Liquid NOS

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SPECIFICATIONS AND TEST METHODS

The following table (pages 24 and 25) gives the specifications for CARBIDE's organic chlorine compounds when this book was printed. Improvements are constantly being made in all products CARBIDE manufactures. Consequently, these specifications are subject to change. For the latest specifications on any of these products, please contact the Sales Office nearest you. Our representatives will be glad to furnish the information. A list of the addresses of these offices is on the outside back cover.

To use this table, look in the left-hand vertical column for the product and in the top horizontal column to find the requirement. At the intersection are the specification limits and a key to the corresponding specification method or quality control test. For example, the water requirement for ethylene dichloride is 0.03 per cent by weight, maximum, using the method G1. This method is found under the "key" column on page 28, which gives the specific sample size for determining water in ethylene dichloride when the general method G is used.

The test methods used in determining the specifications of CARBIDE's products are either A.S.T.M. tests or recognized tests that have been improved or simplified in our laboratories.

Chlorinated Compound	Specific Gravity, 20°/20°C.	
Ethylene Dichloride ^b	Limits	1.2550 to 1.2570
	Method	A
CHLORASOL Fumigant same as Solvent EDM	Limits	1.334 to 1.339
	Method	B
Propylene Dichloride	Limits	1.157 to 1.160
	Method	B
1,1,2-Trichloroethane	Limits	1.425 to 1.445
	Method	B
Butyl Chloride ^b	Limits	0.885 to 0.889
	Method	B
Dichloroethyl Ether	Limits	1.219 to 1.224
	Method	B
CHLOREX Solvent ^b	Limits	1.219 to 1.224
	Method	B
Dichlorisopropyl Ether	Limits	1.113 to 1.119
	Method	B
Triglycol Dichloride	Limits	1.195 to 1.200
	Method	B
Ethylene Chlorhydrin ^b	Limits	1.202 to 1.208
	Method	B
Epichlorhydrin ^b	Limits	1.180 to 1.185
	Method	B
^a No turbidity at temperature indicated		

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Distillation at 760 mm. Hg		Acidity, % by wt. max. as hydro- chloric acid	Cold Test ^a	Water % by wt. max.	Nonvolatiles g./100 ml.	Color Pt-Co units, max.	Odor	Suspended Matter
Shall distill entirely within a 1.5°C. range including 83.5°C.		0.001	—20°C.	0.03	0.001 max.	10		Substantially free
C		E1	F	G1	H	I		K
lbp	75.0°C., min.	0.001		0.05	0.005 max.	15	Non-residual	Substantially free
Dp	85.0°C., max.							
C		E2		G2	H	I	J	K
lbp	94.0°C., min.	0.005	—5°C.		0.005 max.	15		Substantially free
Dp	98.0°C., max.							
C		E3	F		H	I		K
lbp	110.0°C., min.	0.02				15		Substantially free
Dp	115.0°C., max.							
C		E7				I		K
Shall distill entirely within a 1.5°C. range including 78.4°C.		0.005		0.10	0.005 max.	15	Non-residual	Substantially free
C		E6		G3	H	I	J	K
lbp	175°C., min.	0.005	—20°C.	0.10		30		Substantially free
Dp	181°C., max.							
D		E4	F	G3		I		K
lbp	175°C., min.	0.005	—30°C.	0.10		150		Substantially free
Dp	181°C., max.							
D		E4	F	G3		I		K
5 ml.	180°C., min.	0.01		0.10		40		Substantially free
95 ml.	190°C., max.							
C		E5		G2		I		K
lbp	230°C., min.	0.03		0.2		100	Mild	Substantially free
Dp	245°C., max.							
C		E8		G4		I	J	K
lbp	126.0°C., min.	0.02				20 ^c	Mild and non-residual	Substantially free ^c
Dp	132.0°C., max.							
C		E8				I	J	K
lbp	113.0°C., min.			0.10		15		
Dp	118.0°C., max.							
C				G5		I		

^b See also Chart of Miscellaneous Specification Requirements, p. 26.

^c If shipped in glass, otherwise this requirement will not be included.

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MISCELLANE US SPECIFICATION REQUIREMENTS

	Requirement	Ethylene Dichloride	Tendency to Hydrolyze	Ammoniacal Silver Nitrate Test
ETHYLENE DICHLORIDE	Limits	99.0% by wt., min.	1st extraction 1.0 ml., max. 2nd extraction 0.1 ml., max. 3rd extraction 0.1 ml., max.	No Coloration
	Method	L	M	N
BUTYL CHLORIDE	Requirement	Butanol		
	Limits	0.8% by wt. max.		
	Method	O		
"CHLOREX" SOLVENT	Requirement	Ethylene Dichloride		
	Limits	1.0% by wt. max. This requirement is limited by the distillation range.		
	Method	D		
ETHYLENE CHLORHYDRIN	Requirement	Ethylene Chlorhydrin	Water Solubility	
	Limits	98.0% by wt., min.	Miscible in all proportions	
	Method	P 1	Q	
EPICHLORHYDRIN	Requirement	Epichlorhydrin		
	Limits	98.0% by wt., min.		
	Method	R		
TRIGLYCOL DICHLORIDE	Requirement	Total Chlorine		
	Limits	37 to 38% by wt.		
	Method	S		

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Modern analytical equipment helps maintain the high quality of CARBIDE's products.

TEST METHODS

KEY METHOD

A SPECIFIC GRAVITY

Determine at 20°C. by means of a 60-ml. "Pyrex" glass pycnometer calibrated to give the apparent specific gravity at 20/20°C. Maintain the constant temperature bath at $20 \pm 0.05^\circ\text{C}$.

B SPECIFIC GRAVITY

Determine at 20°C. by means of a hydrometer calibrated to give the apparent specific gravity at 20/20°C. and capable of being easily read to the nearest 0.0005 unit. Maintain the constant temperature bath at $20 \pm 0.05^\circ\text{C}$.

C DISTILLATION

Conduct the distillation test in accordance with the American Society for Testing Materials Tentative Methods of Test for Distillation Range of Lacquer Solvents and Diluents (ASTM Designation: D 1078) except to use a 100-ml. standard distillation flask as specified in ASTM D 86, and to use a gas burner as the source of heat. Use the proper ASTM

KEY

METH D

thermometer, or an equivalent thermometer of suitable range, calibrated for 100-mm. immersion, graduated in 0.2°C . units, and capable of being easily read to 0.1°C .

Conduct the distillation at 760 mm. or apply the boiling point correction given in the Physical Properties Table on page 5.

DISTILLATION

Conduct the distillation test in accordance with the American Society for Testing Materials Tentative Methods of Test for Distillation Range of Lacquer Solvents and Diluents (ASTM Designation D 1078) except to use a gas burner as the source of heat. Use the proper ASTM thermometer, or an equivalent thermometer of suitable range, calibrated for 100-mm. immersion, graduated in 0.2°C . units, and capable of being easily read to 0.1°C .

Conduct the distillation at 760 mm. or apply the boiling point correction given in the Physical Properties Table on page 5.

ACIDITY

Measure the specified amount of sample in a graduate and transfer to a 250-ml. Erlenmeyer flask. Add a few drops of 1.0 per cent alcoholic solution of phenolphthalein indicator and titrate with standard alcoholic potassium hydroxide to a pink end-point permanent for at least 15 seconds.

Calculation

ml. KOH x factor = acidity, % by weight

Sample and Reagent

Sample, ml.	Normality of KOH	Factor
E1 116	0.02	0.0005
E2 55	0.02	0.001
E3 63	0.02	0.001
E4 60	0.1	0.005
E5 65	0.1	0.005
E6 82	0.1	0.005
E7 51	0.02	0.001
E8 61	0.1	0.005
E9 25*	0.02	0.002

*Transfer the sample to a 150-ml. separatory funnel; wash with 100 ml. of neutral distilled water and determine the acidity of the aqueous layer.

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F

COLD TEST

Transfer 25 ml. of the sample to a 150-mm. test tube and immerse the tube in a suitable cold bath maintained at approximately 15°C. below the temperature limit given in the specification. Stir the sample continuously with a suitable thermometer until the specified temperature is reached. Examine the contents of the tube for evidence of cloudiness or turbidity.

G

WATER

Reagent — Transfer 487 ml. of freshly distilled anhydrous pyridine to a 4-liter bottle having a 29/42 ground-glass neck. Add 154 g. of reagent-grade iodine, stopper securely, and place on a mechanical roller for 30 min. or until the iodine is completely in solution. Dilute the contents of the bottle to 3500 ml. with anhydrous c.p. methanol. By means of a suitable glass tube extending to the bottom of the bottle add 115 g. of refrigerant-grade sulfur dioxide dried by passing through concentrated sulfuric acid. Allow the contents of the bottle to cool and fit the bottle with a 50-ml. automatic buret having a 29/42 ground-glass joint. Protect all vents open to the air with Ascarite tubes. Much of the accuracy of the method is lost if an ordinary buret is used without special precautions.

Standardization — Place 25 ml. of substantially dry methanol in a 125 ml. glass-stoppered Erlenmeyer flask and titrate with the reagent to the first permanent reddish-brown end-point. Do not confuse the light yellow color formed during the addition of the first few ml. of reagent with the true end-point.

Introduce 1 or 2 drops (0.03 to 0.05 g.) of distilled water weighed to the nearest 0.1 mg. by means of a suitable weighing pipet. Again titrate to the same reddish-brown end-point. Make several check determinations.

Calculation

$$\frac{\text{g. water} \times 100}{\text{ml. titration}} = \text{factor} = \frac{\text{g. water consumed}}{\text{by 100 ml. of reagent}}$$

Procedure — For the sample proceed as directed above to the addition of water. Pipet the prescribed amount of the sample into the flask and immediately titrate with the reagent to the same reddish-brown end-point as that observed in the standardization.

$$\frac{\text{ml. titration} \times \text{factor}}{\text{ml. of sample} \times \text{sp. gr. of sample}} = \text{water, \% by weight}$$

Sample, ml.

G1

100

G2

50

G3

25

G4

15

G5

10

H

NONVOLATILE MATTER

From a graduate introduce 100 ml. of sample into a 125 ml. platinum evaporating dish which has been heated to constant weight at 105 to 110°C., cooled in a desiccator, and weighed to the nearest 0.1 mg.

Evaporate the sample to dryness on a hot water bath and place the dish in an oven maintained at 105 to 110°C. for 30 minutes or until constant weight is attained. Cool the dish in a desiccator and weigh the residue to the nearest 0.1 mg.

Calculation

$$\text{g. residue} = \text{nonvolatile matter, g. per 100 ml.}$$

I

COLOR

Prepare suitable platinum-cobalt color standards according to the method described in the American Public Health Association's "Standard Methods for the Examination of Water and Sewage", 8th edition (1936).

Transfer 100 ml. of the sample to one of two matched tall-form Nessler tubes. Fill the second tube to the mark with the platinum-cobalt standard representing the maximum limit permitted by the specification. Substitute 0.005 per cent potassium dichromate solution for the platinum-cobalt standard, if specified.

Compare the colors of the sample and the standard by viewing vertically down through

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METHOD

the tubes against a white background. If the exact color of the sample is desired, replace the standard in the second tube with other standards until a satisfactory match is obtained.

J

ODOR

Pour a few ml. of the sample on a clean filter paper and observe the odor at once. If specified, allow the sample to evaporate in air at room temperature and notice whether any residual odor remains on the paper.

K

SUSPENDED MATTER

Invert a bottle of the sample and examine by transmitted light.

L

ETHYLENE DICHLORIDE

Prepare a sodium methylate solution by adding short pieces of sodium wire to 500 ml. of anhydrous methanol until effervescence ceases. **Handle the sodium wire with caution!** Transfer 75 ml. of this solution to each of two 250-ml. Erlenmeyer flasks fitted with a 24/40 standard-taper joint. Reserve one of the flasks as a blank and into the other flask introduce 1.2 to 1.4 g. of the sample weighed to the nearest 0.1 mg. by means of a suitable weighing pipet.

Attach the flasks to 500-mm. brine-cooled "West" condensers fitted with 24/40 standard-taper joints, heat to boiling by means of a 500-watt electric mantle, and allow to reflux for one hour. Remove the mantles and allow the flask to cool to room temperature. Wash down the inside walls of each condenser with 50 ml. of distilled water and catch the rinsings in the flask.

Add 1 ml. of a 1.0 per cent alcoholic solution of phenolphthalein indicator and then add a slight excess of 2 N nitric acid. Neutralize the excess with powdered calcium carbonate. Add one ml. of 10 per cent potassium chromate and titrate with standard 0.15 N silver nitrate to the first permanent reddish tinge.

Calculation

$$\frac{(A-B)N \times 9.896}{\text{g. sample}} = \frac{\text{Ethylene Dichloride, \%}}{\text{by weight}}$$

KEY

METHOD

A = ml. of N normal AgNO_3 required for the sample

B = ml. of N normal AgNO_3 required for the blank

M

TENDENCY TO HYDROLYZE

Transfer 200 ml. of distilled water to a 500-ml. separatory funnel, add 200 ml. of the sample, and shake vigorously for 10 minutes. Allow the mixture to separate into two layers, and transfer 100 ml. of the upper (water) layer to one of two 250-ml. Erlenmeyer flasks. Discard the remainder of the upper layer. To the second Erlenmeyer flask add 100 ml. of distilled water and reserve as the blank.

Add 3 to 4 drops of a 0.04 per cent alcoholic solution of bromothymol blue indicator to each flask and titrate with standard 0.01 N barium hydroxide to the first permanent bluish end-point. Make two subsequent water extractions of the sample layer by adding 200 ml. of distilled water to the contents of the separatory funnel each time and repeating the above procedure.

Calculation

A-B = ml. of reagent per extraction

A = ml. of N normal $\text{Ba}(\text{OH})_2$ required for sample

B = ml. of N normal $\text{Ba}(\text{OH})_2$ required for blank

N

AMMONIACAL SILVER NITRATE TEST

Ammoniacal Silver Nitrate Solution—

Add a 10 per cent aqueous solution of ammonium hydroxide dropwise to 100 ml. of a 5 per cent aqueous solution of silver nitrate until the precipitate which first forms is almost but not entirely dissolved. Filter the solution and store the filtrate in a dark colored bottle.

CAUTION! Ammoniacal silver nitrate reagent readily forms explosive compounds on standing. Do not store this reagent but prepare a fresh quantity for each series of determinations. Neutralize the excess reagent and rinse all glassware used with concentrated hydrochloric acid immediately after completing the test.

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Procedure — Transfer 10 ml. of the reagent to a 150-mm. test tube, add 10 ml. of the sample, and mix thoroughly. Allow the mixture to stand in the dark for 20 minutes at 20°C. and examine visually for any evidence of coloration in both layers. The lower layer may be turbid from the suspension of water in the sample, but no coloration should be present.

O

BUTANOL

Phthalic anhydride-pyridine reagent

— Add 42 g. of c.p. phthalic anhydride to 300 ml. of freshly distilled pyridine contained in a one-quart glass-stoppered brown bottle. Shake the bottle vigorously until complete solution is effected. The reagent preferably should stand overnight before using; however, the solution may be heated under hot tap water until a slight cooling of the reagent occurs, indicating complete reaction.

Procedure — Prepare a sufficient number of *heat-resistant* pressure bottles to make all blank and sample determinations in duplicate. Carefully pipet 25 ml. of the phthalic anhydride-pyridine reagent into each of the bottles, using the same pipet for each transfer. Do not allow the reagent to come in contact with the rubber gasket. Before capping purge the bottles for 2 minutes with a gentle stream of nitrogen by means of a glass tube inserted through the neck of the bottle and clamped so that the opening is just above the surface of the liquid. Reserve two of the bottles as blanks.

Into each of the other bottles, introduce 25 ml. of the sample by means of a suitable transfer pipet. Stopper the bottles and wrap each securely in a canvas bag. Place the samples and blanks as close together as possible in a water bath maintained at $98 \pm 2^\circ\text{C}$. for 60 minutes. Maintain sufficient water in the bath to just cover the liquid in the bottles. Remove the bottles from the bath and *allow them to cool in air to room temperature*. When the bottles have cooled, loosen the wrappers, uncap to release any pressure, and then remove the wrappers.

To each bottle add exactly 50 ml. of standard 0.5 N sodium hydroxide, using the same

pipet for each addition. Be sure to allow the same drainage time for each bottle as this amount is not considered in the final calculations. Add 5 drops of a 1.0 per cent pyridine solution of phenolphthalein indicator and titrate with standard 0.5 N sodium hydroxide to a pink end-point permanent for at least 15 seconds. Agitate the contents of the bottle vigorously during the titration.

Calculation

$$\frac{(B-A)N \times 7.41}{25 \times \text{sp. gr.}} = \text{butanol, } \% \text{ by weight}$$

A = ml. of N normal NaOH required for the sample

B = average ml. of N normal NaOH required for the blank

P

ETHYLENE CHLORHYDRIN

Prepare a sufficient number of heat-resistant pressure bottles to make all blank and sample determinations in duplicate. To each of the bottles, add 50 ml. of 0.5 N sodium hydroxide from a buret and exactly 10 ml. of 0.1 N hydrochloric acid by means of a transfer pipet. Reserve two of the bottles for blanks.

Into each of the other bottles, introduce the prescribed amount of sample weighed to the nearest 0.1 mg. by means of a suitable weighing pipet. Fit the bottles with pressure stoppers, wrap each securely in a canvas bag, and place the samples and blanks as close together as possible in a hot water bath maintained at a temperature of $98 \pm 2^\circ\text{C}$. for 45 minutes. Remove the bottles from the bath *and allow them to cool in air to room temperature*. When the bottles have cooled, loosen the wrappers and uncap carefully to release any pressure. Wash down the inside walls of each bottle with a few ml. of distilled water. Carefully wash any liquid on the stopper into the bottle.

Add 6 to 8 drops of phenolphthalein indicator and neutralize the excess caustic by the dropwise addition of 2 N nitric acid until the pink color just disappears. Add 1 ml. excess acid and slowly add powdered calcium carbonate until effervescence ceases. Add about 40 ml. of fresh 1.0 per cent aqueous starch

KEY

METHOD

solution and titrate with standard 0.1 N silver nitrate until approximately 3 ml. of reagent has been added. Add 25 drops of a 0.05 per cent methanolic solution of dichlorofluorescein indicator and continue the titration to the first permanent pink end-point, swirling the contents of the flask vigorously during the titration.

Calculation

$$\frac{(A-B)N \times F}{\text{g. sample}} = \text{chlorhydrin, \% by weight}$$

A = ml. of N normal AgNO_3 required for sample

B = average ml. of N normal AgNO_3 required for blank

F = factor

Sample and Conditions

Sample, g.	Factor
0.4 to 0.7	8.05

P1

Q

WATER SOLUBILITY

Transfer 25 ml. of sample to a 125-ml. Erlenmeyer flask and add 25 ml. of distilled water in 5-ml. portions, shaking the flask well after each addition. Maintain the temperature of the solution at 25°C. Add 25 ml. of the sample to 25 ml. of distilled water in the same manner. The sample is completely miscible if there is no cloudiness or turbidity at any time.

R

EPICHLORHYDRIN

Add from a buret exactly 75 ml. of 0.5 N alcoholic potassium hydroxide to each of three clean dry *heat-resistant* pressure bottles. Reserve one of the bottles for a blank. Into each of the other bottles introduce 0.5 to 0.8 g. of the sample weighed to the nearest 0.1 mg. by means of a suitable weighing pipet. Stopper the bottles and swirl the contents to effect solution.

Wrap the blank and samples in canvas bags or pieces of cloth and place them on a steam bath at $98 \pm 2^\circ\text{C}$. for 45 minutes. Remove the bottles from the bath and allow

KEY

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them to cool in air to room temperature. When the bottles have cooled, loosen the wrappers, uncap to release any pressure, and then remove the wrappers.

To each bottle add exactly 10 ml. of 0.1 N hydrochloric acid and 2 drops of a 1.0 per cent alcoholic solution of phenolphthalein indicator. Add sufficient 2 N nitric acid to just discharge the red color. Slowly add powdered calcium carbonate until effervescence ceases.

Add 40 ml. of fresh starch solution and 25 drops of a 0.05 per cent methanolic solution of dichlorofluorescein indicator; titrate with standard 0.1 N silver nitrate to a permanent pink end-point, swirling the contents of the bottle vigorously during the titration.

Calculation

$$\frac{(A-B)N \times 9.25}{\text{g. sample}} = \text{epichlorhydrin, \% by wt.}$$

A = ml. of N normal AgNO_3 required for the sample

B = ml. of N normal AgNO_3 required for the blank

S

TOTAL CHLORINE

Sodium methylate, approximately 4 N—Carefully add sodium or sodium wire to anhydrous methanol until hydrogen no longer is evolved. Adjust the concentration with additional methanol if necessary.

Standard 0.5 N sodium methylat in pyridine—Prepare this reagent at least one day before using. Transfer 125 ml. of the 4 N sodium methylate to a 1000-ml. volumetric flask containing 125 ml. of anhydrous methanol. Dilute to the mark with redistilled pyridine.

This reagent readily absorbs carbon dioxide from the air, and is best preserved and used in a 50-ml. automatic buret. All vents open to the air must have protective Ascarite tubes. Standardize this reagent daily as described below. Transfer 50 ml. of freshly distilled pyridine to each of four 250-ml. glass-stoppered Erlenmeyer flasks. Reserve two of the flasks for the blank determination.

Into each of the other flasks introduce 1.4

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to 2.0 g. of Bureau of Standards benzoic acid weighed to the nearest 0.1 mg. by means of a tared watch glass. Stopper the flasks and swirl to effect complete solution. Add 2 or 3 drops of 1.0 per cent solution of thymolphthalein indicator in pyridine to each flask and titrate immediately with the sodium methylate-pyridine reagent to the first blue end-point.

Standard sodium methylate-pyridine

$$\frac{W}{0.1221 \times (C-D)} = N, \text{ normality of the sodium methylate-pyridine}$$

C = ml. of NaOCH_3 required for the benzoic acid

D = average ml. of NaOCH_3 required for the blank

W = g. of certified benzoic acid used

$$\Delta N / \Delta t = 0.0005 \text{ per } ^\circ\text{C. for } 0.5 \text{ N } \text{NaOCH}_3$$

Procedure—Prepare a sufficient number of clean, dry, *heat-resistant* pressure bottles to make all blank and sample determinations in duplicate. Pipet 25 ml. of anhydrous ethylene diamine into each of the bottles using the same pipet for each transfer. Reserve two of the bottles for the blank determination, taking care to uncap and expose the contents to the air for the same length of time as the samples.

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METHOD

Into each of the other bottles introduce 0.9 to 1.9 g. of the sample weighed to the nearest 0.1 mg. by means of a suitable weighing pipet. Fit each bottle with a pressure stopper and wrap securely in a fabric bag. Place the samples and blanks as close together as possible in a steam bath maintained at $98 \pm 2^\circ\text{C.}$ for 30 minutes. Maintain sufficient water in the bath to just cover the liquid in the bottles. Remove the bottles from the bath and *allow them to cool in air to room temperature.*

When the bottles have cooled, loosen the bags, uncap to release any pressure, and then remove the bags. To each bottle add 50 ml. of redistilled pyridine, 2 or 3 drops of 1.0 per cent thymolphthalein indicator in pyridine and titrate with standard 0.5 N sodium methylate-pyridine to the first blue end point.

Calculation

$$\frac{(A-B)N \times 3.55}{\text{g. sample}} = \text{total chlorine, per cent by weight}$$

A = ml. of N normal sodium methylate-pyridine required for the sample

B = average ml. of N normal sodium methylate-pyridine required for the blank

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CONSTANT BOILING MIXTURES

COMPONENTS		AZEOTROPE	
Compound	Boiling Point, °C. at 760 mm. Hg	Boiling Point, °C. at 760 mm. Hg	Composition, % by wt. in azeotrope
Butyl chloride	78.6	68.0	93
Water	100.0		7
Dichlorethyl ether	96 ^a	92.7 ^a	—
Ethylene glycol	123 ^a		
Dichlorethyl ether	96 ^a	96 ^a	90
2-Ethylhexanol	109 ^a		10
Dichlorethyl ether	179.2	141.2	28
3-Heptanol	156.4		72
Dichlorethyl ether	179.2	98	34.5
Water	100.0		65.5
Dichlorisopropyl ether	187.0	98.5	37.4
Water	100.0		62.6
Epichlorhydrin	115.2	96	23
Propanol	97.2		77
Epichlorhydrin	115.2	108.3	26
Toluene	110.6		74
Epichlorhydrin	115.2	88.5	74
Water	100.0		26
Ethylene chlorhydrin	128.7	108.8	3
Vinyl 2-chlorethyl ether	109.1		97
Ethylene chlorhydrin	60 ^a	37.1 ^a	39.8
Water	38 ^a		60.2
Ethylene chlorhydrin	75 ^b	51.1 ^b	40.7
Water	51.6 ^b		59.3
Ethylene chlorhydrin	128.7	97.8	42.3
Water	100.0		57.7
Ethylene dichloride	83.5	75.3	22
Carbon tetrachloride	76.5		78
Ethylene dichloride	83.5	71.0	66.5
Ethanol	78.3		33.5
Ethylene dichloride	83.5	67.8	77.1
Ethanol	78.3		15.7
Water	100.0		7.2
Ethylene dichloride	83.5	72.7	60.8
Isopropanol	82.3		39.2
Ethylene dichloride	83.5	69.7	73.3
Isopropanol	82.3		19.0
Water	100.0		7.7
Ethylene dichloride	83.5	59.5	65
Methanol	64.5		35
Ethylene dichloride	83.5	82.3	59.1
Trichlorethylene	87.1		40.9
Ethylene dichloride	83.5	71.6	91.8
Water	100.0		8.2
2-Ethylhexyl chloride	89 ^a	77 ^a	39
2-Ethylbutanol	77 ^a		61
2-Ethylhexyl chloride	106.9 ^b	92 ^b	32
2-Ethylbutanol	92 ^b		68
2-Ethylhexyl chloride	173.0	97.3	45
Water	100.0		55
Propylene dichloride	96.3	78.4	89.4
Water	100.0		10.6
Trichlorethane	113.7	86.0	83.6
Water	100.0		16.4
Triglycol dichloride	240.9	99.7	6.0
Water	100.0		94.0

^a At 50 mm. Hg

^b At 100 mm. Hg

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PHYSICAL PROPERTY CHARTS

Chart 1 *Vapor Pressures at Various Temperatures*

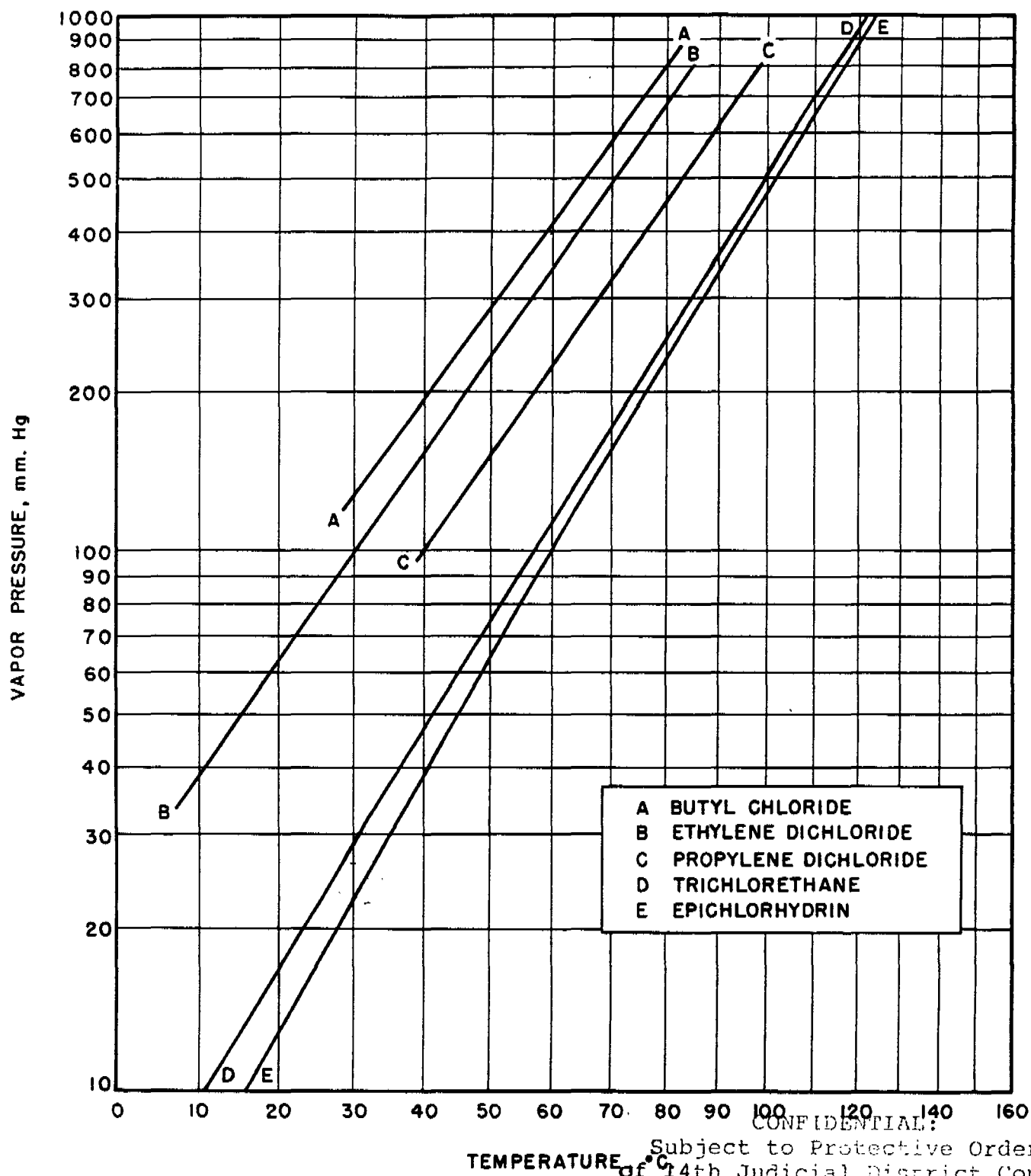
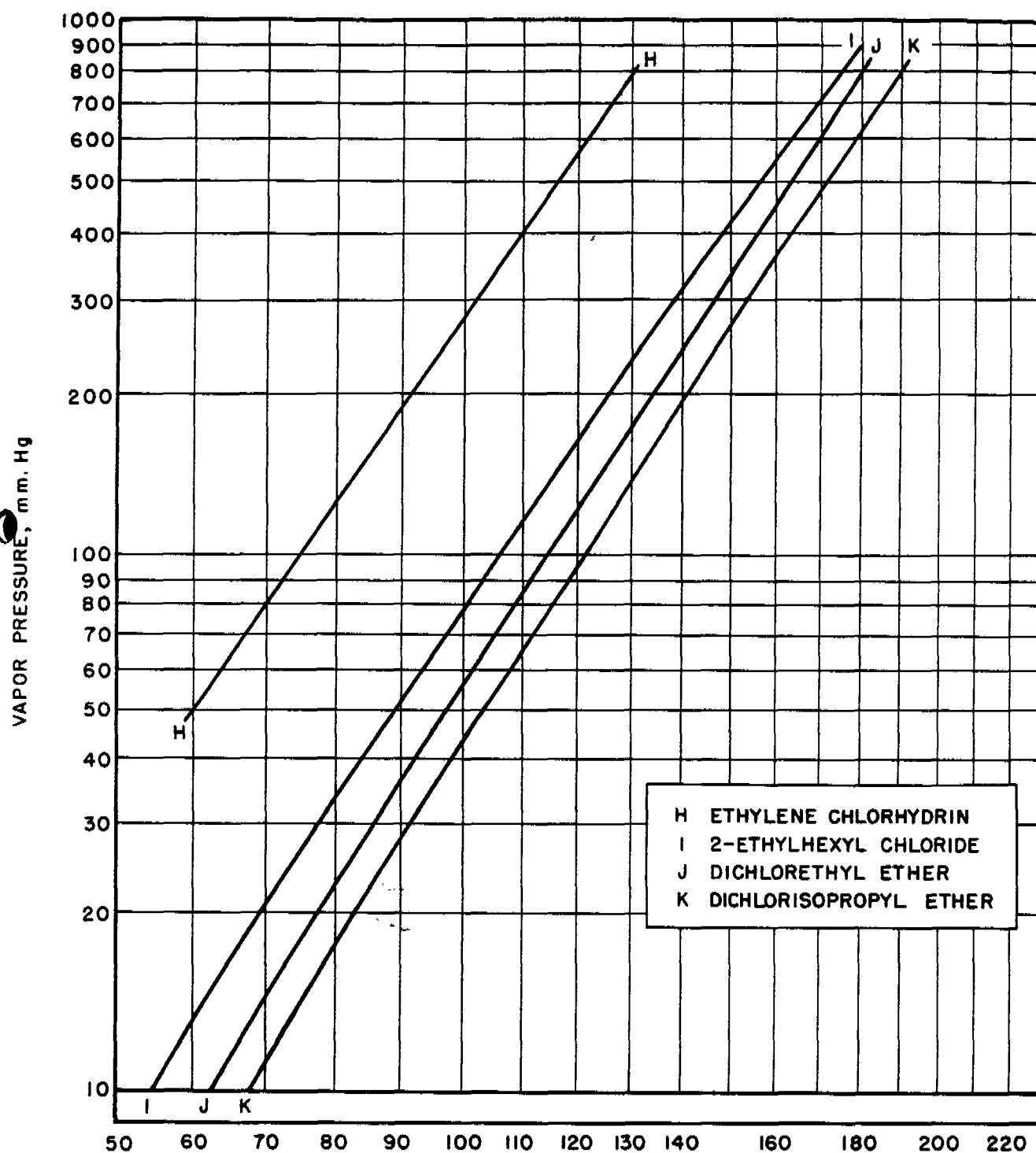


Chart 2 Vapor Pressures at Various Temperatures



H ETHYLENE CHLORHYDRIN
I 2-ETHYLHEXYL CHLORIDE
J DICHLORETHYL ETHER
K DICHLORISOPROPYL ETHER

TEMPERATURE, °C.
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Chart 3 *Specific Gravity at Various Temperatures*

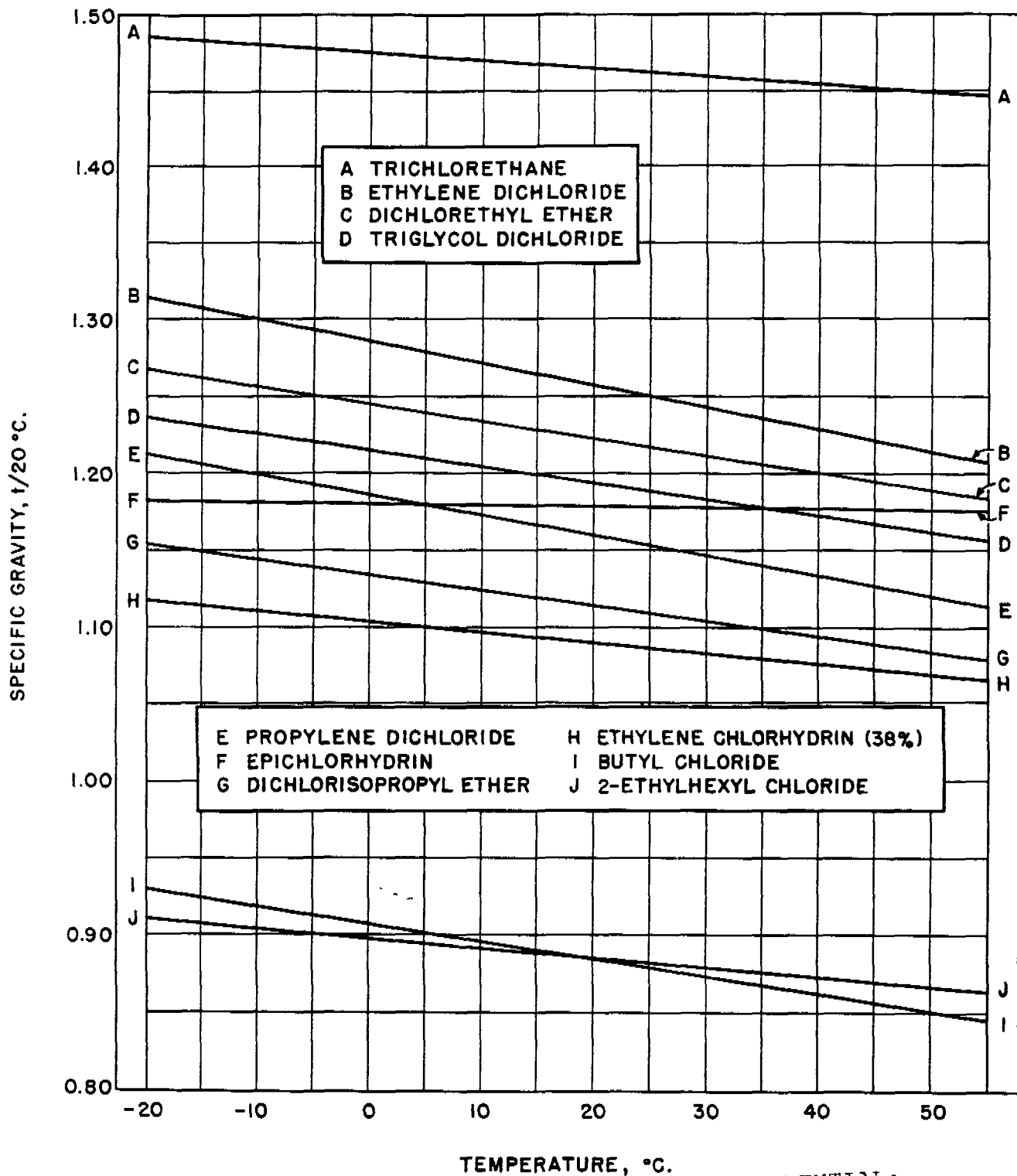


Chart 4 *Specific Gravity of Carbon Tetrachloride Mixtures and Vinyl Chloride Mixtures*

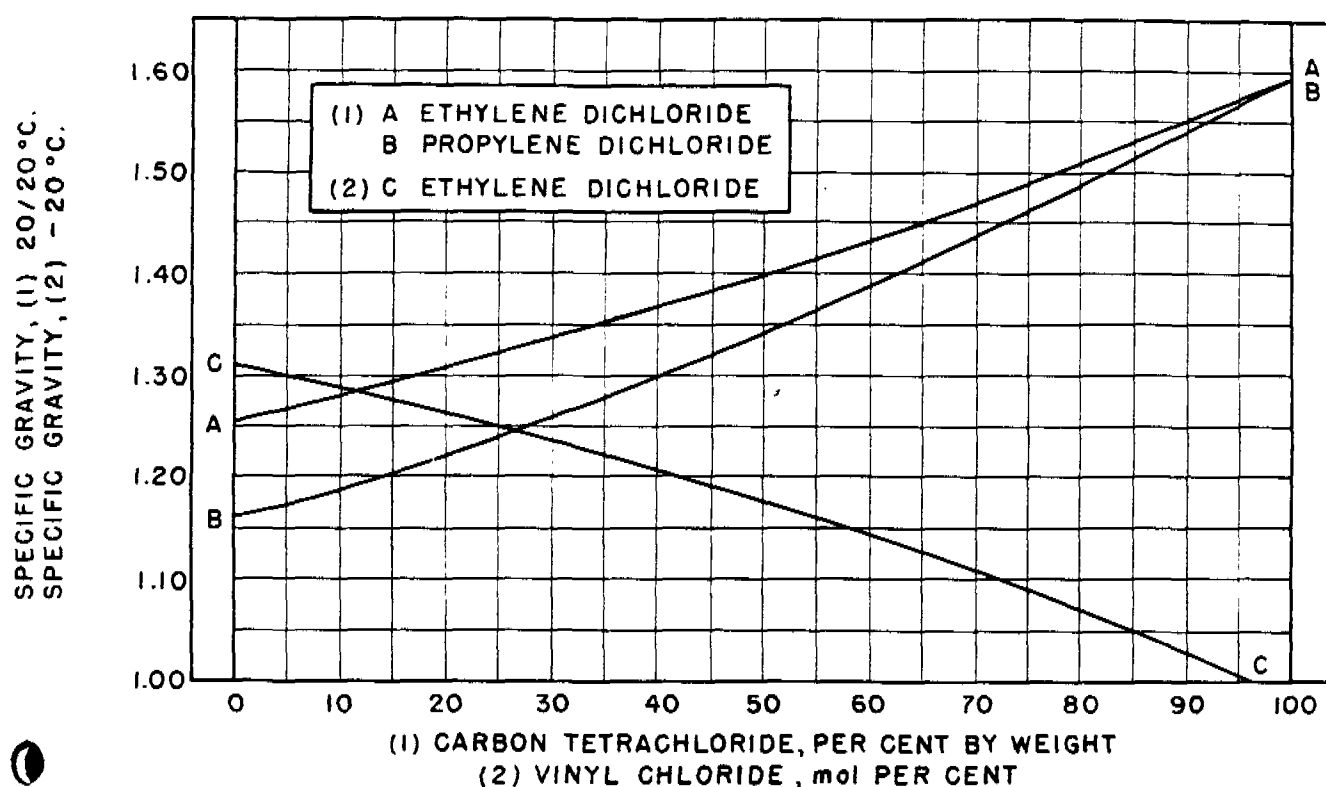
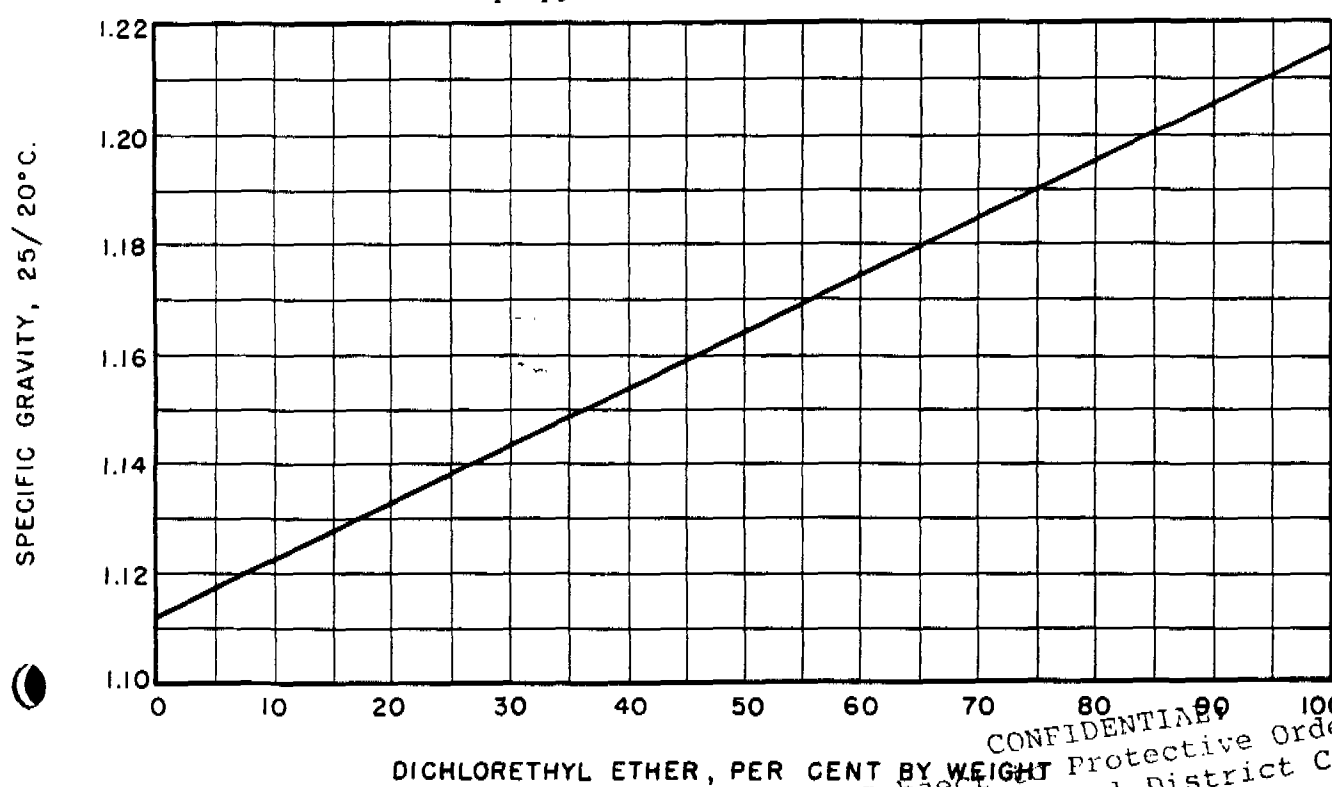


Chart 5 *Specific Gravity of Solutions of Dichlorethyl Ether and Dichlorisopropyl Ether*



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Chart 6 *Solubility of Chlorinated Compounds in Water at Various Temperatures*

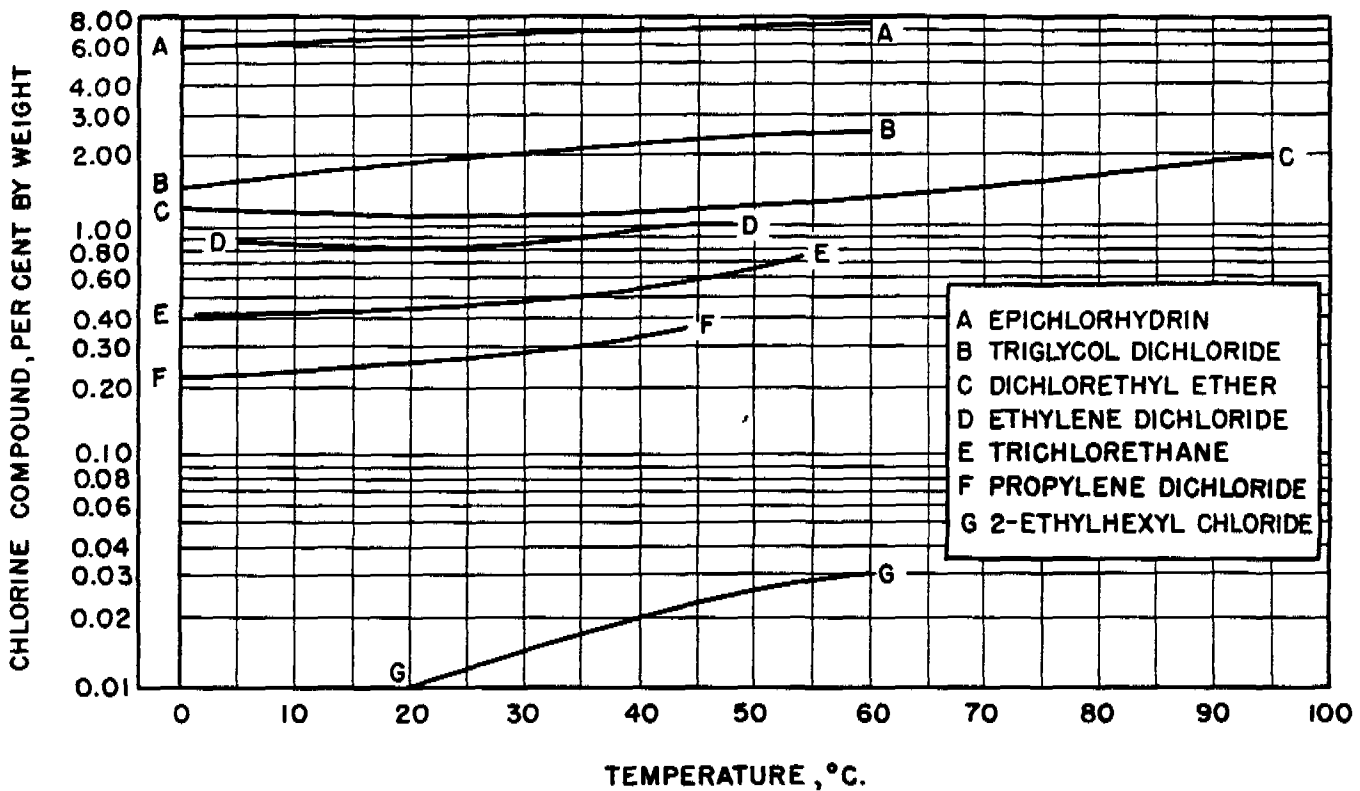
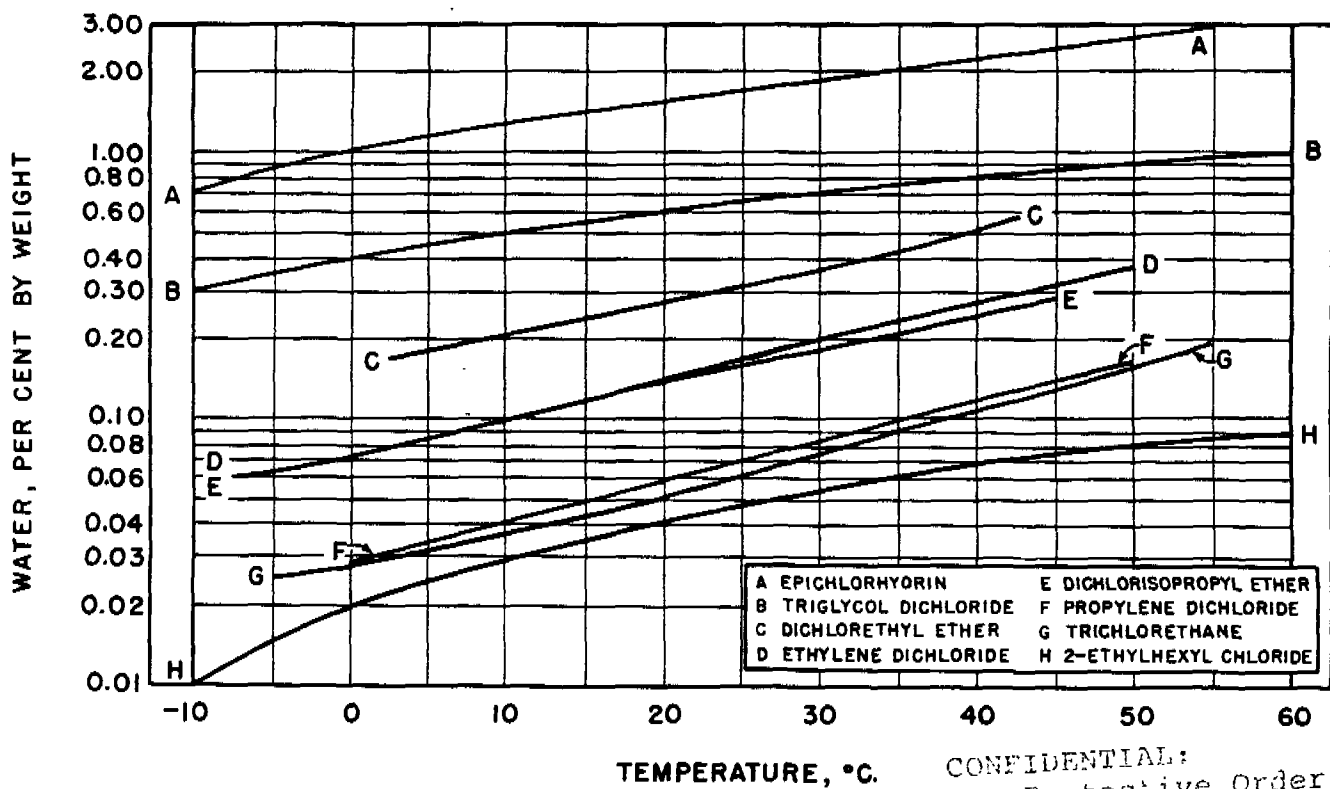


Chart 7 *Solubility of Water in Chlorinated Compounds at Various Temperatures*



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Chart 8 *Viscosities at Various Temperatures*

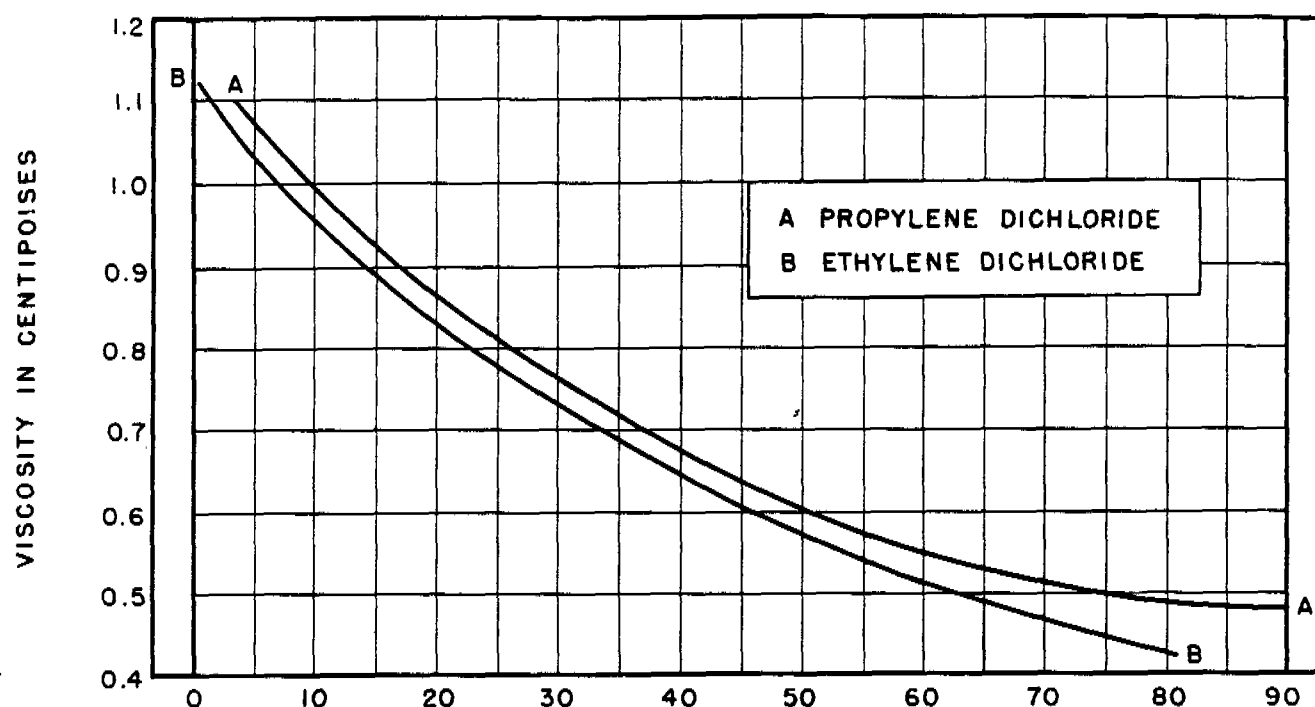
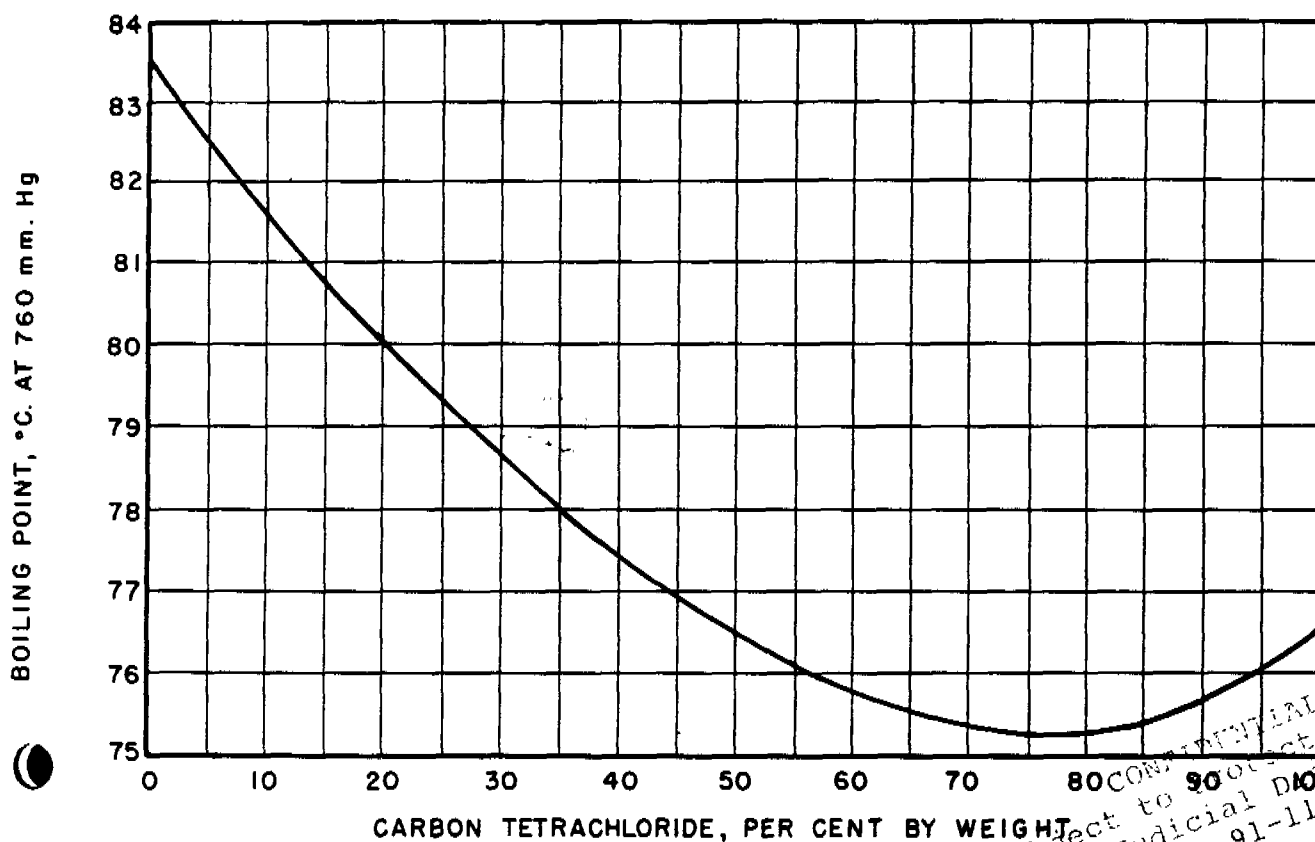


Chart 9 *Boiling Points of Ethylene Dichloride-Carbon Tetrachloride Mixtures*



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Chart 10 *Comparative Evaporation Rates*

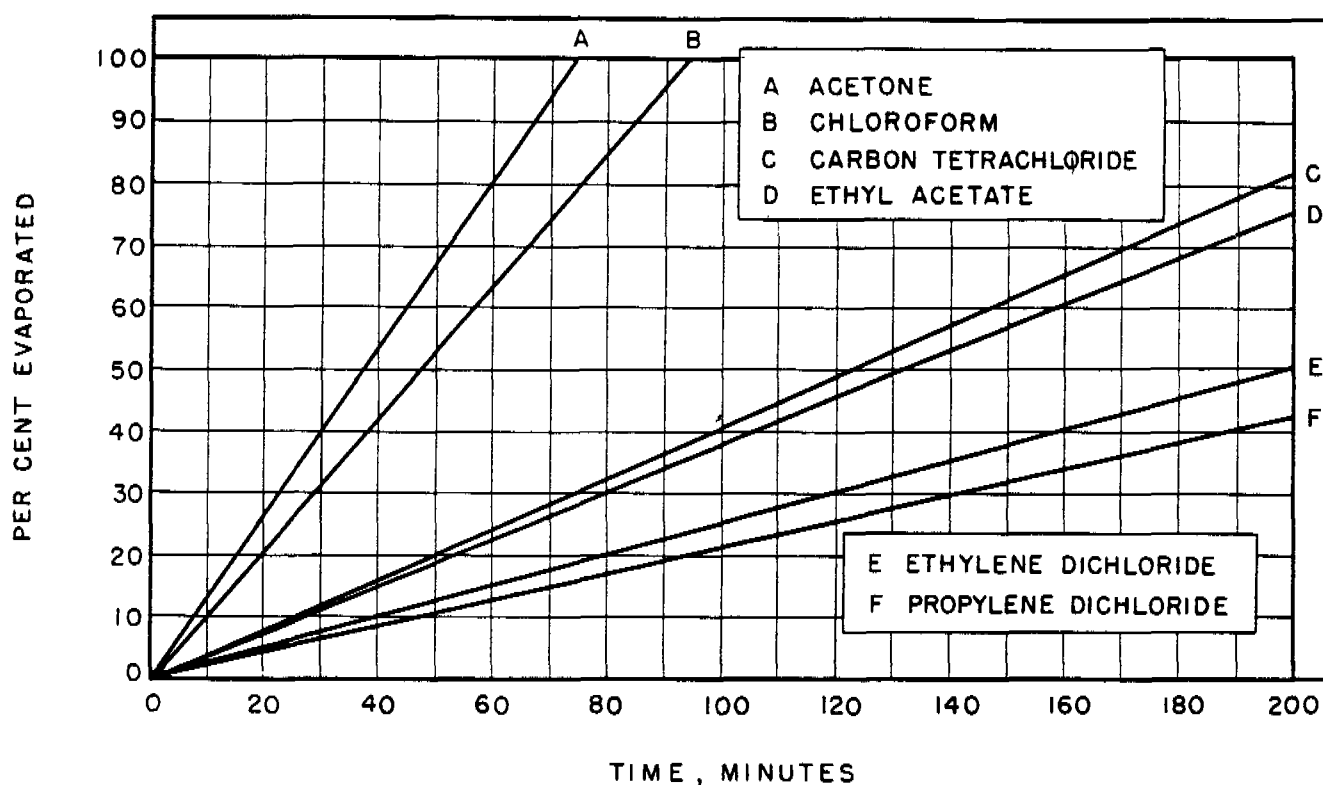
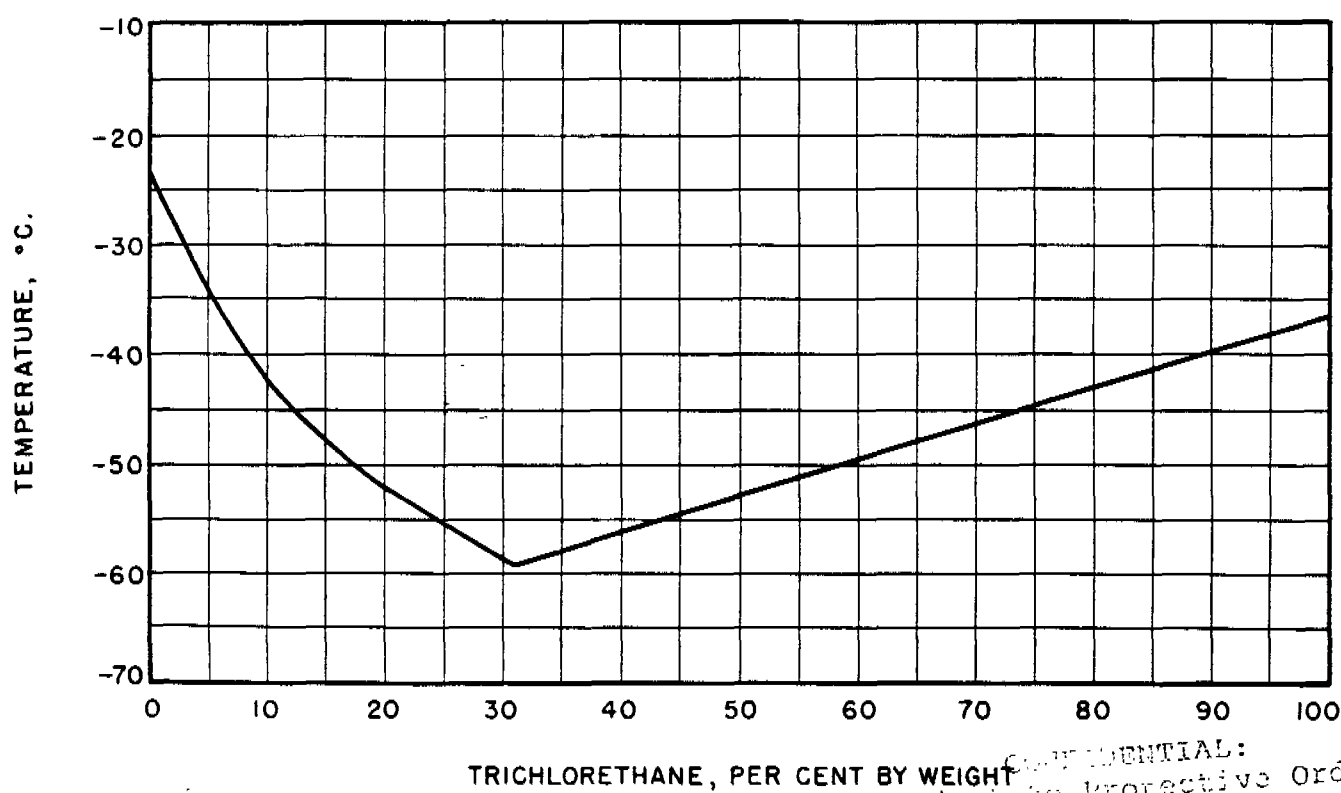


Chart 11 *Freezing Points of Trichlorethane-Carbon Tetrachloride Mixtures*



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Chart 12 *Dichlorethyl Ether: Heat of Vaporization vs. Vapor Pressure*

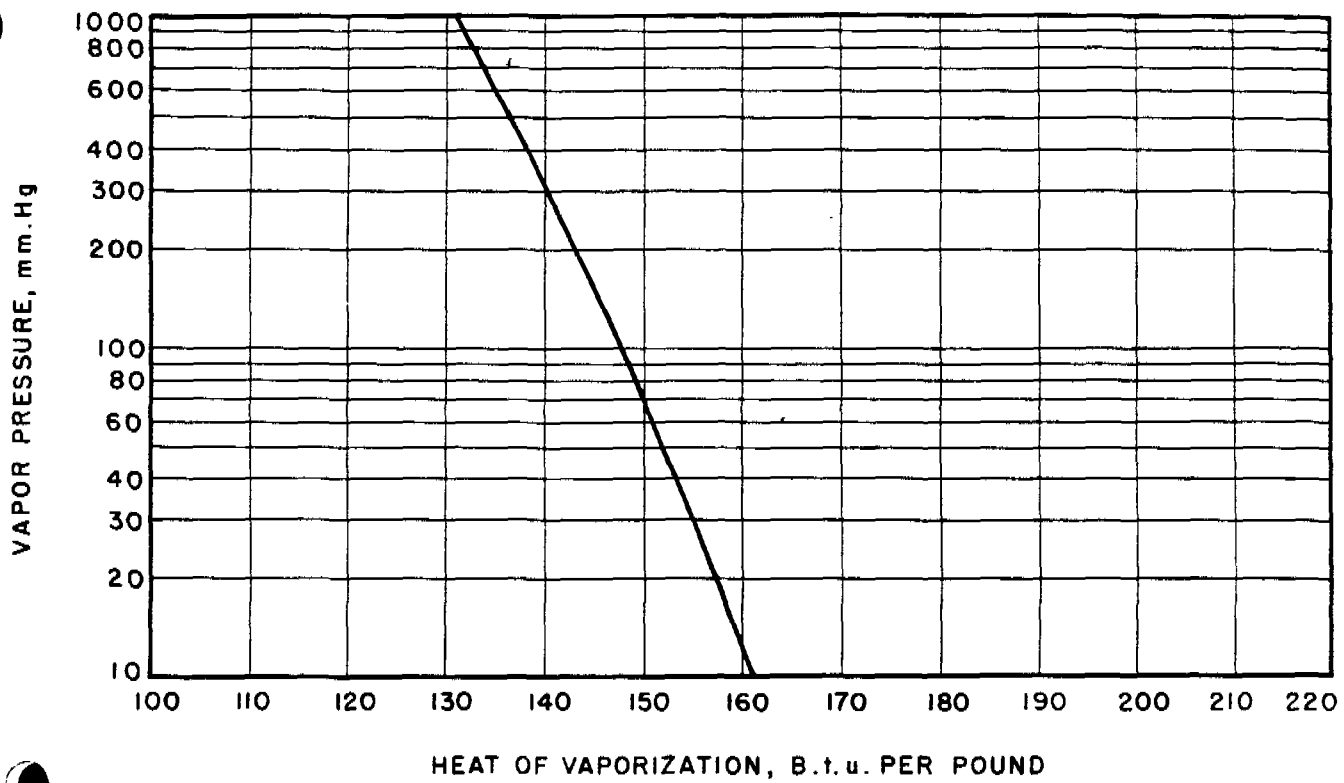
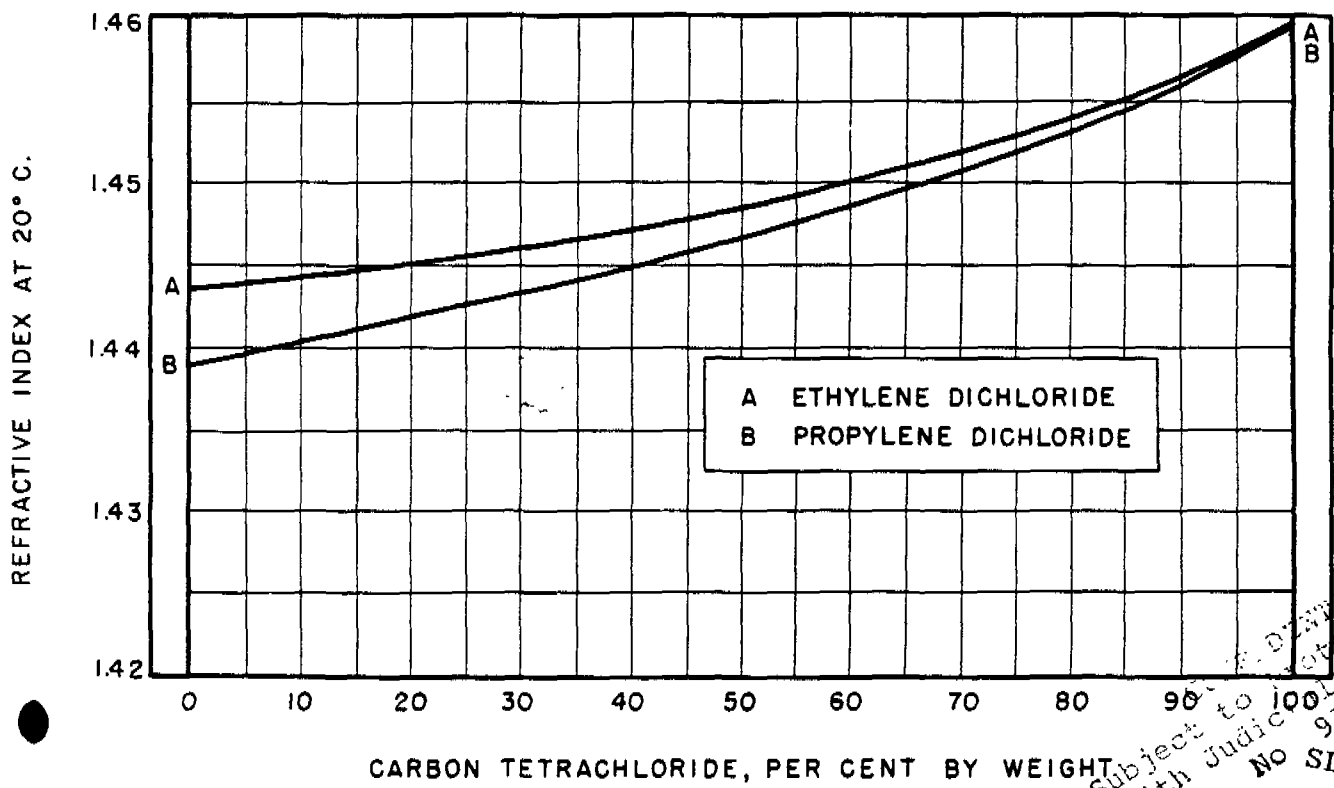


Chart 13 *Refractive Index of Carbon Tetrachloride Mixtures*



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Nothing contained herein shall be construed to constitute a permission or recommendation to practice any invention covered by any patent without a license from the patent owner.

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In addition to this booklet, literature is available on more than 400 synthetic organic chemicals which Union Carbide Chemicals Company produces. Separate booklets and reprints about any specific group of chemicals or individual chemicals may interest you. Your inquiry is invited.

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ANHYDROL Proprietary Solvent
Butanol
iso-Decanol
Diisobutyl Carbinol
Ethanol (All Formulas)
2-Ethylbutanol
2-Ethylhexanol
Heptadecanol
1-Hexanol
Isobutanol
Isopropanol
Methanol
Methyl Amyl Alcohol
2-Methyl-1-Pentanol
SYNASOL Proprietary Solvent
Tetradecanol
2,6,8-Trimethyl-4-Nonanol
Undecanol (3, 9-diethyl-6-tridecanol)

ALDEHYDES

Acetaldehyde
Acetaldol
Acrolein
Acrolein Dimer (2-formyl-3,4-dihydro-2H-pyran)
Butyraldehyde
Crotonaldehyde
Formaldehyde
Glutaraldehyde
Glyoxal
2-Hydroxyadipaldehyde
Isobutyraldehyde
Methacrolein
Paraldehyde
Propionaldehyde
Pyruvic Aldehyde

GLYCOLS AND TRIOLS

CARBOSEAL Anti-Leak
Diethylene Glycol
Dipropylene Glycol
2,2-Diethyl-1,3-Propanediol
2-Ethyl-2-Butyl-1,3-Propanediol
Ethylene Glycol
2-Ethyl-1,3-Hexanediol

1,2,6-Hexanetriol
Hexylene Glycol
1,5-Pentanediol
Propylene Glycol
Triethylene Glycol

POLYALKYLENE GLYCOLS

CARBOWAX Polyethylene Glycols
200, 300, 400, and 600 (liquids)
CARBOWAX Polyethylene Glycols
1000, 1500, 1540, 4000, 6000
and 20M (solids)
CARBOWAX Methoxy Polyethylene
Glycols 350, 550, and 750
Polypropylene Glycols 150,
425, 1025, and 2025
"Niox" Polyols

"UCON" FLUIDS AND LUBRICANTS

"CELLOSIZ" HYDROXYETHYL CELLULOSE

"POLYOX" WATER-SOLUBLE RESINS

SOLVENT RECOVERY PLANTS AND "COLUMBIA" ACTIVATED CARBON

ETHERS AND OXIDES

Butyl Ether
CARBOXIDE Fumigant
CHLOREX Solvent
Dibutyl CARBITOL
Dibutyl CELLOSOLVE
Dichlorethyl Ether
Dichlorisopropyl Ether
Diethyl CARBITOL
1,4-Dioxane
2-Ethoxy-3,4-Dihydro-2H-Pyran
Ethylene Oxide
Ethyl Ether
Hexyl Ether
Isopropyl Ether
Propylene Oxide
Vinyl Ethers

"FLEXOL" PLASTICIZERS

FLEXOL Plasticizer A-26
FLEXOL Plasticizer B-400
FLEXOL Plasticizer CC-55
FLEXOL Plasticizer DOP
FLEXOL Plasticizer 3GH
FLEXOL Plasticizer 3GO
FLEXOL Plasticizer 4GO
FLEXOL Plasticizer 8N8
FLEXOL Plasticizer R-2H
FLEXOL Plasticizer TOF
FLEXOL Plasticizer 426
FLEXOL Plasticizer 810
FLEXOL Plasticizer 10-10
FLEXOL Plasticizer 10-A

ANHYDRIDES

Acetic Anhydride
Butyric Anhydride
Propionic Anhydride

ESTERS

Allylidene Diacetate
Amyl Acetate, Primary
(mixed isomers)
Butyl Acetate
Butyl Acrylate
Butyl CARBITOL Acetate
Butyl CELLOSOLVE Acetate
CARBITOL Acetate
CELLOSOLVE Acetate
Decyl Acrylate
Di(2-Ethylhexyl) Maleate
Diethyl Maleate
Diethyl Succinate
Diethyl Sulfate
Ethyl Acetate
Ethyl Acetoacetate
Ethylene Carbonate
Ethyl Formate
2-Ethylhexyl Acetate
Ethyl Silicates
Glycol Diacetate
Isobutyl Acetate
Isopropyl Acetate
Methoxytriglycol Acetate
Methyl Amyl Acetate
Methyl CELLOSOLVE Acetate
Propylene Carbonate

Synthetic Organic Chemicals

Sucrose Octa Acetate
Tetraethyl Orthosilicate
Vinyl Acetate

METALLIC SALTS

NIAPROOF Aluminum Acetate,
Basic
Aluminum Formo Acetate
Copper Acetate (Cupric)
Potassium Acetate
Sodium Acetate
Sodium Diacetate
Zinc Acetate

KETONES

Acetone
Diacetone Alcohol
Diethyl Ketone
Diisobutyl Ketone
Ethyl Butyl Ketone
Isobutyl Heptyl Ketone (2,6,
8-trimethyl-4-nonanone)
Isophorone
Mesityl Oxide
Methyl Acetone, Synthetic
Methyl n-Amyl Ketone
Methyl Ethyl Ketone
Methyl Isobutyl Ketone
Methyl Propyl Ketone
2,4-Pentanedione

ACETALS

ACIDS

Acetic Acid
Acrylic Acid (glacial and 30%
aqueous solutions)
Butyric Acid
Crotonic Acid
2-Ethylbutyric Acid
2-Ethylhexoic Acid
2,4-Hexadienoic Acid

GLYCOL-ETHERS

1-Butoxyethoxy-2-Propanol
Butyl CARBITOL
Butyl CELLOSOLVE
CARBITOL Solvent

CELLOSOLVE Solvent
Ethoxytriglycol
Hexyl CARBITOL
Hexyl CELLOSOLVE
Methoxytriglycol
Methyl CARBITOL
Methyl CELLOSOLVE

NITROGEN COMPOUNDS

Acetonitrile
N-Acetyl Ethanolamine
Acrylonitrile
Amine 220
N-Aminoethyl Ethanolamine
N-Aminoethyl Piperazine
Butyl Amine
Dibutyl Amine
Diethanolamine
Diethyl Amine
Diethylamino Propylamine
Diethylene Triamine
N,N-Diethyl Ethanolamine
Di(2-Ethylhexyl) Amine
Diisopropanolamine
Diisopropyl Amine
Dimethylamino Propylamine
Dimethyl Ethanolamine
Ethyl Amine
Ethylene Cyanohydrin
Ethylene Diamine
2-Ethylhexyl Amine
N-Ethyl Morpholine
N-Hydroxyethyl Piperazine
Isopropyl Amine
N-Methyl Diethanolamine
N-Methyl Piperazine
Mixed Isopropanolamine
Monoethanolamine
Monoisopropanolamine
Morpholine
Piperazine
Propylene Diamine
Tetraethylene Pentamine
Triethanolamine
Triethyl Amine
Triethylene Tetramine
Triisopropanolamine

ARYL CHEMICALS

Acetoacet-Arylamides
Acetophenone
Aryl Ethanolamines

Butyl Benzoate
Ethylbenzene
alpha-Methylbenzyl Ether
2-Methyl-5-Ethyl Pyridine
Phenol (U.S.P.)
Phenyl Methyl Carbinol
N-Phenyl Morpholine
Picolines
Styrene

SULFUR COMPOUNDS

KROMFAX Solvent
Mercaptoethanol

"TERGITOL" SURFACE ACTIVE AGENTS

HYDROCARBONS

"CRAG" AGRICULTURAL CHEMICALS

DYNEL ACRYLIC FIBERS

"CARBOSEAL" ANTI-LEAK

INDUSTRIAL FUMIGANTS

SORBIC, FUNGISTAT FOR FOODS

"MORLEX" CORROSION INHIBITORS

MONOMERS

Acrylic Acid and Esters
Acrylic Acid (glacial and 30%
aqueous solutions)
Ethyl Acrylate
2-Ethylhexyl Acrylate
Butyl Acrylate
Vinyl Esters
Vinyl Acetate
Vinyl Butyrate
Vinyl 2-Ethylhexoate
Vinyl Propionate
Maleic Esters
Diethylhexyl Maleate
Diethyl Maleate
Vinyl Ethers
Vinyl Butyl Ether
Vinyl 2-Chlorethyl Ether
Vinyl Ethyl Ether

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UNION CARBIDE CHEMICALS COMPANY

DIVISION OF  CORPORATION

30 East 42nd Street, New York 17, New York

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Warehouse Stocks in Principal Cities

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