Kirk-Othmer Encyclopedia of Chemical Technology.

TRICHLOROETHYLENE

Trichloroethylene [79-01-6], trichloroethene, $CHCL{=}CCL_2$, commonly called "tri," is a colorless, sweet smelling (chloroformlike odor), volatile liquid and a powerful solvent for a large number of natural and synthetic substances. It is nonflammable under conditions of recommended use. In the absence of stabilizers, it is slowly decomposed (autoxidized) by air. The oxidation products are acidic and corrosive. Stabilizers are added to all commercial grades. Trichloroethylene is moderately toxic and has narcotic properties.

Trichloroethylene was first prepared by Fischer in 1864. In the early 1900s, processes were developed in Austria for the manufacture of tetrachloroethane and trichloroethylene from acetylene. Trichloroethylene manufacture began in Germany in 1920 and in the United States in 1925. Early uses of trichloroethylene were as an extraction solvent for natural fats and oils, such as palm, coconut, and soybean oils. It was later used for decaffeination of coffee, but this use has essentially been replaced by steam processes today. The demand for trichloroethylene was stimulated by the development of the vapor-degreasing process during the 1920s and by the growth of the dry-cleaning industry during the 1930s. By the mid-1950s, perchloroethylene had replaced trichloroethylene in dry-cleaning, and metal cleaning became the principal use for trichloroethylene.

The demand for trichloroethylene grew steadily until 1970. Since that time trichloroethylene has been a less desirable solvent because of restrictions on emissions under air pollution legislation and the passage of the Occupational Safety and Health Act. Whereas previously the principal use of trichloroethylene was for vapor degreasing, currently 1,1,1-trichloroethane is the most used solvent for vapor degreasing. The restrictions on production of 1,1,1-trichloroethane [71-55-6] from the 1990 Amendments to the Montreal Protocol on substances that deplete the stratospheric ozone and the U.S. Clean Air Act 1990 Amendments will lead to a phase out of 1,1,1-trichloroethane by the year 2005, which in turn will likely result in a slight resurgence of trichloroethylene in vapor-degreasing applications. The total production, however, will probably stay relatively low because regulations will require equipment designed to assure minimum emissions.

1. Physical and Chemical Properties

The physical properties of trichloroethylene are listed in Table 1. Trichloroethylene is immiscible with water but miscible with many organic liquids; it is a versatile solvent. It does not have a flash or fire point. However, it does exhibit a flammable range when high concentrations of vapor are mixed with air and exposed to high energy ignition sources.

The most important reactions of trichloroethylene are atmospheric oxidation and degradation by aluminum chloride. Atmospheric oxidation is catalyzed by free radicals and accelerated with heat and with light, especially ultraviolet. The addition of oxygen leads to intermediates (**1**) and (**2**).

Table 1. Properties of Trichloroethylene

Property	Value
molecular weight	131.39
melting point, °C	−86.5
boiling point, °C	87.3
specific gravity, liquid	
20/4°C	1.464
100/4°C	1.322
vapor density at bp, kg/m^3	4.61
n_D	
liquid, 20°C	1.4782
vapor, 0°C	1.001784
viscosity, mPa·s(= cP) liquid	
20°C	0.57
60°C	0.42
vapor at 100°C	0.01246
surface tension at 20°C, mN/m(=dyn/cm)	29.3
heat capacity, J/(kg·K)[a]	
liquid at 20°C	938
vapor at 100°C	693
critical temperature, °C	300.2
critical pressure, MPa[b]	4.986
thermal conductivity, W/(m·K)	
liquid at 20°C	0.115
vapor at bp	0.00851
coefficient of cubical expansion, liquid at 0–40°C	0.00119
dielectric constant, liquid at 16°C	3.42
dipole moment, C·m[c]	3.0×10^{-30}
heat of combustion, kJ/g[a]	−6.56
heat of formation, kJ/mol[a]	
liquid	−42.3
vapor	−7.78
latent heat of evaporation at bp, kJ/kg[a]	238
explosive limits, vol % in air	
25°C	8.0-saturation
100°C	8.0–44.8
vapor pressure[d], kPa[e]	
Antoine constants	
A	5.75373
B	1076.67
C	199.991
solubility, g	
H_2O in 100 g trichloroethylene	
0°C	0.010
20°C	0.0225
60°C	0.080
trichloroethylene in 100 g H_2O	
20°C	0.107
60°C	0.124

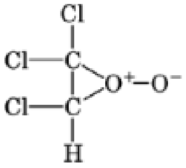
[a] To convert J to cal, divide by 4.184.

[b] To convert MPa to atm, divide by 0.101.

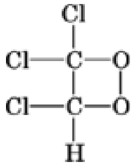
[c] To convert C·m to debye, divide by 3.336×10^{-30}.

[d] $\log_{10} P = A - B/(T + C)$, T in °C.

[e] To convert kPa to mm Hg, multiply by 7.5.



(**1**)



(**2**)

Compound (**1**) decomposes to form dichloroacetyl chloride, which in the presence of water decomposes to dichloroacetic acid and hydrochloric acid (HCl) with consequent increases in the corrosive action of the solvent on metal surfaces. Compound (**2**) decomposes to yield phosgene, carbon monoxide, and hydrogen chloride with an increase in the corrosive action on metal surfaces.

In the presence of aluminum, oxidative degradation or dimerization supply HCl for the formation of aluminum chloride, which catalyzes further dimerization to hexachlorobutene. The latter is decomposed by heat to give more HCl. The result is a self-sustaining pathway to solvent decomposition. Sufficient quantities of aluminum can cause violent decomposition, which can lead to runaway reactions (1, 2). Commercial grades of trichloroethylene are stabilized to prevent these reactions in normal storage and use conditions.

Amine-stabilized products, once the predominant grade, are sold today only in limited amounts. Most vapor-degreasing grades contain neutral inhibitor mixtures (3–5) including a free-radical scavenger, such as an amine or pyrrole, to prevent the initial oxidation reaction. Epoxides, such as butylene oxide and epichlorohydrin, are added to scavenge any free HCl and $AlCl_3$. Concern over the toxicity of these epoxides has eliminated the use of epichlorohydrin in the United States during the 1980s and may restrict butylene oxide in the future.

Trichloroethylene is not readily hydrolyzed by water. Under pressure at 150°C, it gives glycolic acid, $CH_2OHCOOH$, with alkaline hydroxides. Strong alkalies dehydrochlorinate trichloroethylene with production of spontaneously explosive and flammable dichloroacetylene. Reaction with sulfuric acid (90%) yields monochloroacetic acid, $CH_2ClCOOH$. Hot nitric acid reacts with trichloroethylene violently, producing complete oxidative decomposition. Under carefully controlled conditions, nitric acid gives trichloronitromethane (chloropicrin) and dinitrochloromethane (6). Dichloroacetylene, C_2Cl_2, can also be formed from trichloroethylene in the presence of epoxides and ionic halides (7).

In the presence of catalysts, trichloroethylene is readily chlorinated to pentachloro- and hexachloroethane. Bromination yields 1,2-dibromo-1,1,2-trichloroethane [13749-38-7]. The analogous iodine derivative has not been reported. Fluorination with hydrogen fluoride in the presence of antimony trifluoride produces 2-chloro-1,1,1-trifluoroethane [75-88-7] (8). Elemental fluorine gives a mixture of chlorofluoro derivatives of ethane, ethylene, and butane.

Liquid trichloroethylene has been polymerized by irradiation with ^{60}Co γ-rays or 20-keV x-rays (9). Trichloroethylene has a chain-transfer constant of <1 when copolymerized with vinyl chloride (10) and is used extensively to control the molecular weight of poly(vinyl chloride) polymer.

A variety of trichloroethylene copolymers have been reported, none with apparent commercial significance. The alternating copolymer with vinyl acetate has been patented as an adhesive (11) and as a flame retardant

(12, 13). Copolymerization with 1,3-butadiene and its homologues has been reported (14–16). Other comonomers include acrylonitrile (17), isobutyl vinyl ether (18), maleic anhydride (19), and styrene (20).

Terpolymers have been made with vinyl chloride–vinylidene chloride (21) and vinyl acetate–vinyl alcohol (22).

2. Manufacture

As late as 1968, 85% of the production capacity in the United States was based on acetylene, but rising acetylene [74-86-2] costs reduced this figure to 8% by 1976 (23), and now most trichloroethylene is made from ethylene [74-85-1] or 1,2-dichloroethane [107-06-2].

2.1. From Acetylene

The acetylene-based process consists of two steps. First acetylene is chlorinated to 1,1,2,2-tetrachloroethane [79-34-5]. The reaction is exothermic (402 kJ/mol = 96 kcal/mol but is maintained at 80–90°C by the vaporization of solvent and product. Catalysts include ferric chloride and sometimes phosphorus chloride and antimony chloride (24).

The product is then dehydrohalogenated to trichloroethylene at 96–100°C in aqueous bases such as $Ca(OH)_2$ (25) or by thermal cracking, usually over a catalyst (24) such as barium chloride on activated carbon or silica or aluminum gels at 300–500°C. The yield of trichloroethylene (23) is about 94% based on acetylene. A significant disadvantage of the alkaline process is the loss of chlorine as calcium chloride. In thermal cracking the chlorine can be recovered as hydrochloric acid, an important feedstock in many chemical processes. Since it poisons the catalysts during thermal cracking, all ferric chloride must be removed from the tetrachloroethane feed (24). Tetrachloroethane can also be cracked to trichloroethylene without catalysts at 330–770°C, but considerable amounts of tarry by-products are formed.

2.2. Chlorination of Ethylene

Dichloroethane, produced by chlorination of ethylene, can be further chlorinated to trichloroethylene and tetrachloroethylene. The exothermic reaction is carried out at 280–450°C. Temperature is controlled by a fluidized bed, a molten salt bath, or the addition of an inert material such as perchloroethylene. The residence time in the reactor varies from 2 to 30 seconds, depending on conditions (24). Catalysts include potassium chloride and aluminum chloride (26), Fuller's earth (27), graphite (28), activated carbon (29), and activated charcoal (27).

Maximum conversion to trichloroethylene (75% of dichloroethane feed) is achieved at a chlorine to dichloroethane ratio of 1.7:1. Tetrachloroethylene conversion reaches a maximum (86% conversion of dichloroethane) at a feed ratio of 3.0:1 (24).

2.3. Oxychlorination of Ethylene or Dichloroethane

Ethylene or dichloroethane can be chlorinated to a mixture of tetrachoroethylene and trichloroethylene in the presence of oxygen and catalysts. The reaction is carried out in a fluidized-bed reactor at 425°C and 138–207 kPa (20–30 psi). The most common catalysts are mixtures of potassium and cupric chlorides. Conversion to chlorocarbons ranges from 85–90%, with 10–15% lost as carbon monoxide and carbon dioxide (24). Temperature control is critical. Below 425°C, tetrachloroethane becomes the dominant product, 57.3 wt % of crude product at 330°C (30). Above 480°C, excessive burning and decomposition reactions occur. Product ratios can be controlled but less readily than in the chlorination process. Reaction vessels must be constructed of corrosion-resistant alloys.

2.4. Other Routes

A unique process that produces vinyl chloride, trichloroethylene, dichloroethane, and trichloroethane simultaneously has been developed by Produits Chemiques Pechiney-Saint-Gobain in France (31). Dichloroethylene is chlorinated directly at low temperature to tetrachloroethane, which is then thermally cracked to give trichloroethylene and hydrochloric acid. The dichloroethylene feed is coproduced with vinyl chloride in a hot chlorination reactor, using chlorine and ethylene as feedstocks.

A Japanese process developed by Taogosei Chemical Co. chlorinates ethylene directly in the absence of oxygen at 811 kPa (8 atm) and 100–130°C (32). The products are tetrachlorethanes and pentachloroethane [76-01-7], which are then thermally cracked at 912 kPa (9 atm) and 429–451°C to produce a mixture of trichloroethylene, perchloroethylene [127-18-4], and hydrochloric acid.

3. Shipping and Storage

Shipment of trichloroethylene is usually by truck or rail car and also in 208-liter (55-gallon) steel drums. Mild steel tanks, if appropriately equipped with vents and vent driers to prevent the accumulation of water, are adequate for storage. Precautions, such as diking, should be taken to provide for adequate spill containment at the storage tank. Seamless black iron pipes are suitable for transfer lines, gasketing should be of Teflon, Viton, or other solvent impermeable material. Centrifugal or positive-displacement pumps made from cast iron, steel, or stainless steel are suitable for use. Aluminum should never be used as a construction material for any halogenated hydrocarbon. Glass containers, amber or green, are suitable for small quantities, such as in a laboratory, but care should be taken for spill containment in the event of breakage.

Containers should bear warning labels against breathing vapors, ingesting the liquid, splashing solvent in eyes or on skin and clothing, and using it near an open flame, or where vapors will come in contact with hot metal surfaces ($>176°C$). Precautions in handling any waste products in conformance with federal, state, and local regulations should be included.

Although the flammability hazard is very low, ignition sources should not be present when trichloroethylene is used in highly confined or unventilated areas. Tanks in which flammable concentrations could develop should be grounded to prevent build-up of static electric charges. Under no circumstances should welding or cutting with a torch take place on any storage container or process equipment containing trichloroethylene.

4. Economic Aspects

In 1990, worldwide capacities and production figures in metric tons were Western Europe capacity 266,000, production 180,000; Japan capacity 90,000, production 64,000; and United States capacity 145,000, production 88,600 (23).

United States production and price statistics are presented in Table 2. The demand for trichloroethylene in the United States has been shrinking sharply since 1970 because of pressures from environmental and safety legislation. Similar pressures have weakened the demand in Europe and Japan.

In 1966, the Los Angeles Air Pollution Control Board designated trichloroethylene as a photochemically reactive solvent that decomposes in the lower atmosphere, contributing to air pollution. In 1970 all states were required to submit pollution control plans to EPA to meet national air quality standards. These plans, known as State Implementation Plans (SIPS), controlled trichloroethylene as a volatile organic compound (VOC). They

Table 2. United States Trichloroethylene Production and Prices[a]

Year	Production, 10^3 t	Price, ¢/kg
1960	160.4	28.05
1965	197.5	22.55
1970	277.6	23.1
1975	133.0	39.05
1980	121.1	59.95
1985	79.5	84.7
1986	77.3	84.7
1987	88.6	84.7

[a] Ref. 23.

were designed to have each state achieve the National Ambient Air Quality Standard (NAAQS) for ozone. The regulations were established to control the emission of precursors for ozone, of which trichloroethylene is one.

For worker exposure to trichloroethylene vapor, OSHA set a maximum eight-hour time-weighted average (TWA) concentration of 100 ppm. This severely restricted certain applications, and many organizations converted to other chlorinated solvents. As a result, U.S. production of trichloroethylene declined about 70% from a peak in 1970 (Table 2). In 1989, OSHA lowered the permissible exposure limit (PEL) from 100 ppm eight-hour TWA to 50 ppm eight-hour TWA (33). This added further pressure for some users to consider changing to alternative solvents.

In addition to environmental and safety factors, some of the early decline in manufacture was hastened by a series of plant shutdowns between 1971 and 1973 resulting primarily from the high costs of the acetylene-based process. No new production capacity is planned in the United States for the foreseeable future.

Shortages, together with rapidly escalating fuel and feedstock prices, have led to a dramatic increase in the price of trichloroethylene, which more than doubled between 1972 and 1976 and doubled again between 1975 and 1985. The price stayed flat during the late 1980s. During the 1990s, the price will likely depend on energy demands and the availability of trichloroethylene.

5. Specifications and Standards

Commercial grades of trichloroethylene, formulated to meet use requirements, differ in the amount and type of added inhibitor. The grades sold in the United States include a neutrally inhibited vapor-degreasing grade and a technical grade for use in formulations. U.S. Federal Specification O-T-634b lists specifications for a regular and a vapor-degreasing grade.

Apart from added stabilizers, commercial grades of trichloroethylene should not contain more than the following amounts of impurities: water 100 ppm; acidity, ie, HCl, 5 ppm; insoluble residue, 10 ppm. Free chlorine should not be detectable. Test methods have been established by ASTM to determine the following characteristics of trichloroethylene: acid acceptance, acidity or alkalinity, color, corrosivity on metals, nonvolatile-matter content, pH of water extractions, relative evaporation rate, specific gravity, water content, water-soluble halide ion content, and halogen content (34).

The passage of the Resource Conservation and Recovery Act in 1978 and its implementation in 1980 generated an increase in the recycling of trichloroethylene, which, in turn, defined the need for specifications for recycled solvent. The ASTM is currently working on a set of consensus specifications for recycled solvent.

6. Health and Safety Factors (Toxicity)

Trichloroethylene is acutely toxic, primarily because of its anesthetic effect on the central nervous system. Exposure to high vapor concentrations is likely to cause headache, vertigo, tremors, nausea and vomiting, fatigue, intoxication, unconsciousness, and even death. Because it is widely used, its physiological effects have been extensively studied.

Exposure occurs almost exclusively by vapor inhalation, which is followed by rapid absorption into the bloodstream. At concentrations of 150–186 ppm, 51–70% of the trichloroethylene inhaled is absorbed. Metabolic breakdown occurs by oxidation to chloral hydrate [302-17-0], followed by reduction to trichloroethanol [115-20-8], part of which is further oxidized to trichloroacetic acid [76-03-9] (35–37). Absorbed trichloroethylene that is not metabolized is eventually eliminated through the lungs (38). The OSHA permissible exposure limit (PEL) eight-hour TWA concentration has been set at 50 ppm for eight-hour exposure (33).

It is estimated that concentrations of 3000 ppm cause unconsciousness in less than 10 minutes (39). Anesthetic effects have been reported at concentrations of 400 ppm after 20-min exposure. Decrease in psychomotor performance at a trichloroethylene concentration of 110 ppm has been reported in one study (33), whereas other studies find no neurotoxic effects at concentrations of 200 ppm (40–43).

Victims of overexposure to trichloroethylene should be removed to fresh air, and medical attention should be obtained immediately. A self-contained positive pressure breathing device should be used wherever high vapor concentrations are expected, eg, when cleaning up spills or when accidental releases occur.

The distinctive odor of trichloroethylene may not necessarily provide adequate warning of exposure, because it quickly desensitizes olfactory responses. Fatalities have occurred when unprotected workers have entered unventilated areas with high vapor concentrations of trichloroethylene or other chlorinated solvents. For a complete description of proper entry to vessels containing any chlorinated solvent, see ASTM D4276-84, Standard Practice for Confined Area Entry (34).

Ingestion of large amounts of trichloroethylene may cause liver damage, kidney malfunction, cardiac arrhythmia, and coma (38); vomiting should not be induced, but medical attention should be obtained immediately.

Protective gloves and aprons should be used to prevent skin contact, which may cause dermatitis (44–46). Eyes should be washed immediately after contact or splashing with trichloroethylene.

The National Cancer Institute reported in 1975 that massive oral doses of trichloroethylene caused liver tumors in mice but not in rats (47). Trichloroethylene was tested again in the 1980s by the National Toxicology Program (NTP) with similar results (48, 49). The EPA has classified trichloroethylene as B2, a probable human carcinogen. The International Agency for Research on Cancer (IARC) classifies it as group 3, ie, unclassifiable as to human carcinogenicity. Teratogenicity studies conducted with trichloroethylene by The Dow Chemical Company showed no significant effects on fetal development (50). During the 1980s several epidemiology studies were conducted on worker populations exposed to trichloroethylene (51–53). Each of these studies failed to show a positive link between human exposure in the work place and cancer.

During the 1980s a significant amount of work was done on developing methods for treatment of contaminated groundwater and also on setting standards for trichloroethylene under the Safe Drinking Water Act. The EPA has set a maximum contaminant level goal (MCLG) at 0 based on the animal carcinogenic effects (54). The maximum contaminant level (MCL) is currently set at five micrograms per liter.

7. Uses

Approximately 85% of the trichloroethylene produced in the United States is consumed in the vapor degreasing of fabricated metal parts (see Metal surface treatments); the remaining 15% is divided equally between exports and miscellaneous applications (23). The Western European consumption was 95% in vapor degreasing and 5% for other uses. Japanese consumption is similar to the United States at 83% and 17% (23). A variety of miscellaneous applications include use of trichloroethylene as a component in adhesive and paint-stripping formulations, a low temperature heat-transfer medium, a nonflammable solvent carrier in industrial paint systems, and a solvent base for metal phosphatizing systems. Trichloroethylene is used in the textile industry as a carrier solvent for spotting fluids and as a solvent in waterless preparation dying and finishing operations. Many of these uses have gradually switched over to alternatives, such as 1,1,1-trichloroethane, because of the environmental regulations enforced on trichloroethylene.

Trichloroethylene was approved for use for many years as an extraction solvent for foods. In late 1977, the Food and Drug Administration (FDA) banned its use as a food additive, directly or indirectly, prohibiting the use in hop extraction, decaffeination of coffee, isolation of spice oleoresins, and other applications. The FDA also banned the use of trichloroethylene in cosmetic and drug products (23).

Trichloroethylene is widely used as a chain-transfer agent in the production of poly(vinyl chloride). An estimated 5500 metric tons are consumed annually in this application.

Trichloroethylene is being evaluated by the industry as a precursor in the production of hydrochlorofluorocarbons (HCFC), the replacement products for the chlorofluorocarbons implicated in the depletion of the stratospheric ozone. At this time it is too early to project any estimates or probabilities for potential volume changes as a result of this opportunity (23).

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