Although chlorosulfuric acid [7790-94-5],  $ClSO_3H$ , is the *Chemical Abstracts* name, chlorosulfonic acid is the commercial designation by which this compound is more widely known. Other synonyms include sulfuric chlorohydrin, sulfuric acid chlorohydrin, monochlorosulfuric acid, chlorohydrated sulfuric acid, monochlorosulfonic acid, and chlorohydrosulfurous acid.

Chlorosulfuric acid is a clear to straw-colored liquid with a pungent odor. It is a highly reactive compound that reacts violently with water to produce heat and dense white fumes of hydrochloric acid and sulfuric acid. Chlorosulfuric acid reacts with most organic materials, in some cases with charring. It is used principally in organic synthesis as a sulfating, sulfonating, or chlorosulfonating agent. The main application for chlorosulfuric acid is as an intermediate in the production of synthetic detergents, drugs, and dyestuffs (see Detergency; Dyes and dye intermediates; and Pharmaceuticals). This acid is preferred in many applications because it yields the desired isomers. It has also been used as a smoke-forming agent in warfare (see Chemicals in war).

Chlorosulfuric acid preparation and properties were described in 1854 (1), but the structure was debated for many years until it was shown in 1941 by magnetic susceptibility measurements that the chlorine was bonded directly to the sulfur atom (2). The chlorosulfuric acid structure (Cl–SO<sub>2</sub>–OH) is analogous to the structure of sulfuric acid, the chlorine replacing one of the hydroxyl groups (see Sulfuric acid and sulfur trioxide). This structure has been substantiated using Raman spectra (3, 4).

### 1. Physical Properties

Chlorosulfuric acid is actually an equilibrium mixture of chlorosulfuric acid and minor amounts of hydrogen chloride, sulfur trioxide, and some related compounds. Heating chlorosulfuric acid results in formation of sulfuryl chloride [7791-25-5],  $Cl_2O_2S$ , sulfuric acid [7664-93-9], pyrosulfuryl dichloride [7791-27-7],  $Cl_2O_5S_2$ , and pyrosulfuric acid [7783-05-3],  $H_2O_7S_2$ . There is also evidence of the formation of higher polyacids such as  $H(SO_3)_4Cl$  (see Sulfur compounds; Sulfuric and sulfurous esters). Heating beyond the boiling point results in decomposition into sulfur dioxide [7446-09-5], chlorine, and water. Distillation tends to degrade the acid rather than purify it; therefore, the physical constants reported reflect the influence of varying amounts of these impurities. The physical property values given in Table 1 are considered to be the most reliable available. In addition, values have been reported for heats of formation (5), vapor pressure data (6), infrared spectra (7), and thermal constants of mixtures with sulfur trioxide [7446-11-9] (8).

Chlorosulfuric acid is miscible with sulfur trioxide, sulfuric acid, and pyrosulfuryl chloride in all proportions. Mixtures with sulfur trioxide are used as smoke-forming agents. The properties of such mixtures have been described (3, 15, 16). Mixtures of chlorosulfuric acid and pyrosulfuryl chloride form an azeotrope when distilled (17).

Chlorosulfuric acid is soluble in *sym*-tetrachloroethane,  $C_2H_2Cl_4$ , chloroform, and dichloromethane, ie, halocarbons that also contain hydrogen, but is only slightly soluble in carbon disulfide and carbon tetrachloride, a halocarbon not containing hydrogen. It is soluble in liquid sulfur dioxide, nitrobenzene, acetic acid and acetic

Property	Value
mol wt	116.531
mp, °C	-81 to -80
bp, °C	151–152
vapor pressure, in Pa <sup>a</sup> , T in K	$\log P = 11.496 - 2752/T$
vapor density	
at 216°C, kg/m <sup><math>3b</math></sup>	2.4
specific gravity at 15.6°C	1.752
density, kg/m <sup>3</sup>	
from $0-100^{\circ}C^{c}$	$1784.7 - 1.616T + 1.21T^2  imes 10^{-3} - 4.1T^3  imes 10^{-6}$
at $-10^{\circ}$ C	1800
at $-70^{\circ}$ C <sup>d</sup>	1900
viscosity, $mPa \cdot s(= cP)$	
at $-31.6^{\circ}$ C	10.0
$ m at - 17.8^{\circ}C$	6.4
at $15.6^{\circ}\mathrm{C}$	3.0
at $49^{\circ}C$	1.7
specific heat, $J/(kg\cdot K)^e$	$1.18 imes 10^3$
heat of formation, $\Delta H_{\rm f}$ , 298, J/mol <sup>e</sup>	$-597.1 imes10^3$
heat of vaporization, J/g <sup>e</sup>	452-460
heat of solution in water, J/mol <sup>e</sup>	$168.6 imes 10^3$
index of refraction, $n_{\rm D}$ , at $14^{\circ}{ m C}$	1.437
dielectric constant at $15^{\circ}C^{f}$	$60\pm10$
electrical conductivity, $(ohm \cdot cm)^{-1}$ at $25^{\circ}C^{g}$	$0.2-0.3 imes 10^{-3}$

Table 1. Physical Properties of Chlorosulfuric acid

<sup>*a*</sup> To convert Pa to mm Hg, multiply by 0.0075.

 $^b$  The calculated value is 4.04 kg/m  $^3.$ 

<sup>c</sup> Ref. 9.

 $^d$  Ref. 10.

<sup>e</sup> To convert J to cal, divide by 4.184.

<sup>f</sup> Ref. 11.

<sup>g</sup> Ref. 12. For the value in sulfuric acid or in liquid HCl, see Refs. 13 and 14, respectively.

anhydride, and trifluoroacetic acid and its anhydride, as well as sulfuryl chloride. Chlorosulfuric acid reacts with alcohols, ketones (qv), diethyl ether, and dimethyl sulfoxide, although some literature references report the use of the latter two as solvents. Caution should be used when working with any solvent because a reaction may occur when the temperature is increased or in the presence of catalysts. The solubility of hydrogen chloride in chlorosulfuric acid is indicated to be 0.51 wt % at 20°C and 101.3 kPa (760 mm Hg) (18), but this decreases rapidly with increasing temperature.

### 2. Chemical Properties

Chlorosulfuric acid is a strong acid containing a relatively weak sulfur-chlorine bond. Many salts and esters of chlorosulfuric acid are known, most of them are relatively unstable or hydrolyze readily in moist air.

Strong dehydrating agents such as phosphorous pentoxide or sulfur trioxide convert chlorosulfuric acid to its anhydride, pyrosulfuryl chloride [7791-27-7],  $S_2O_5Cl_2$ . Analogous trisulfuryl compounds have been identified in mixtures with sulfur trioxide (3, 19). When boiled in the presence of mercury salts or other catalysts, chlorosulfuric acid decomposes quantitatively to sulfuryl chloride and sulfuric acid. The reverse reaction has been claimed as a preparative method (20), but it appears to proceed only under special conditions. Noncatalytic

decomposition at temperatures at and above the boiling point also generates sulfuryl chloride, chlorine, sulfur dioxide, and other compounds.

In organic reactions, chlorosulfuric acid is a powerful sulfating and sulfonating agent, a fairly strong dehydrating agent, and a specialized chloridating agent. In most of its applications it is used to form sulfates, sulfonates, sulfonyl chlorides, and occasionally other chlorine derivatives with organic compounds such as hydrocarbons (qv), alcohols, phenols, and amines (qv). Reactions of chlorosulfuric acid are the result of attachment of a  $-SO_3H$  or a  $-SO_2Cl$  group to give a sulfonate, sulfate, or a sulfonyl chloride. The general reactions are

 $RH + ClSO_3H \longrightarrow RSO_3H + HCl$ 

 $ROH + ClSO_3H \longrightarrow ROSO_3H + HCl$ 

$$RH + 2 ClSO_3H \longrightarrow RSO_2Cl + H_2SO_4 + HCl$$

The acid is rather slow to react with aliphatic hydrocarbons unless a double bond or other reactive group is present. This permits straight-chain fatty alcohols such as lauryl alcohol [112-53-8],  $C_{12}H_{26}O$ , to be converted to the corresponding sulfate without the degradation or discoloration experienced with the more vigorous reagent sulfur trioxide. This is important in shampoo base manufacture (see Hair preparations).

In the presence of excess acid, a sulfonyl chloride group  $(-SO_2Cl)$  can be attached to an aromatic group, ie, chlorosulfonation can occur,

$$C_6H_6 + 2 ClSO_3H \longrightarrow C_6H_5SO_2Cl + HCl + H_2SO_4$$

or attachment can be to an alkoxy group in a process called chlorosulfation.

$$(CH_3)_2$$
 CHOH + 2 ClSO<sub>3</sub>H  $\longrightarrow$   $(CH_3)_2$  CHOSO<sub>2</sub>Cl + HCl + H<sub>2</sub>SO<sub>4</sub>

In the presence of a large excess of acid, sulfones such as diphenyl sulfone [127-63-9],  $(C_6H_5)_2SO_2$ , can be formed (see Sulfolanes and sulfones). Sulfamation forms a -C-N-S- bond as in sodium cyclohexylsulfamate [139-05-9],  $C_6H_{11}NHSO_3Na$ , (see Sulfamic acid and sulfamates). Reviews of chlorosulfuric acid reactions are available (21, 22).

The fluorine analogue of chlorosulfuric acid, fluorosulfuric acid [7789-21-1], FSO<sub>3</sub>H, is considerably more stable than chlorosulfuric acid because of the stronger fluorine–sulfur bond (see Fluorine compounds, inorganic-sulfur, fluorosulfuric acid). Bromosulfuric acid [25275-22-3], BrSO<sub>3</sub>H, decomposes in air at  $-30^{\circ}$ C, and the iodine equivalent has not been synthesized (23).

### 3. Manufacture

Modern plants manufacture chlorosulfuric acid by direct union of equimolar quantities of sulfur trioxide and dry hydrogen chloride gas. The reaction takes place spontaneously with evolution of a large quantity of heat. Heat removal is necessary to maintain the temperature at 50–80°C and thus minimize unwanted side reactions. The sulfur trioxide may be in the form of 100% liquid or gas, as obtained from boiling oleum, ie, fuming sulfuric

acid, or may be present as a dilute gaseous mixture as obtained directly from a contact sulfuric acid plant (24). The hydrogen chloride gas can be in the form of 100% gas or in a diluted form.

Processes for the manufacture of high quality acid are described in a number of patents (24–34). The most common features of these processes are continuous-flow operation, two or more vessels in series for gas–liquid contacting, heat exchangers for controlling temperatures, and the use of excess chlorosulfuric acid as a solvent during at least part of the reaction. If the product is to be used only as a smoke-forming agent and not as a chemical intermediate, or wherever else quality is not critical, then an adiabatic or high temperature operation can be used. The design and operation of this type of plant is described in several reports (35, 36). In some older, discontinued processes, hydrogen chloride reacts directly with oleum, or an alkali or alkaline-earth chloride reacts with oleum, followed by distillation.

Chlorosulfuric acid attacks brass, bronze, lead, and most other nonferrous metals. From a corrosion standpoint, carbon steel and cast iron are acceptable below 35°C provided color and iron content is not a concern. Stainless steels (300-series) and certain aluminum alloys are acceptable materials of construction, as is Hastelloy. Glass, glass-lined steel, or Teflon-lined piping and equipment are the preferred materials at elevated temperatures and/or high velocities or where trace iron contamination is a problem, such as in the synthetic detergent industry.

### 4. Economic Aspects

There are 20 manufacturers of chlorosulfuric acid in Europe, Asia, and Australia, plus manufacturers in Brazil and Mexico (37). The two United States manufacturers are E. I. du Pont de Nemours & Co., Inc. having a capacity in excess of 30,000 t/yr (38), and Gabriel Chemical Co. having a capacity of 13,600 t/yr (39). The United States and Canadian consumption is about 27,000 t/yr. Pricing from 1988 through the early 1990s has held constant at \$386/t, down 6% from previous years (38).

Detergent and other surfactants (qv) manufacturing is the leading consumer of chlorosulfuric acid at approximately 40%; pharmaceuticals (qv) is next at 20%.

### 5. Specifications and Standards

No formal industrywide specifications for chlorosulfuric acid exist. Each producer or user establishes individual specifications as needed. However, typical commercial chlorosulfuric acid meets the specifications given in Table 2. The U.S. military specification MILC 379A applies to a mixture of chlorosulfuric acid and sulfur trioxide.

The acid may be shipped in tank cars, tank trucks, iso-tainers or drums via common carrier (40). The shipping hazard class is Corrosive Material, the DOT labels and placards required are Corrosive and Poison. The UN number is 1754. When iron content and color are not of concern, the acid may be stored and shipped in steel equipment. The iron content is generally 25–50 ppm and the color is pale yellow to amber.

#### 6. Analytical and Test Methods

Total acidity and total chlorides can be determined by conventional techniques after hydrolyzing a sample. Satisfactory procedures for determining hydrogen chloride and free-sulfur trioxide are described in the literature (18, 41). Small amounts of both hydrogen chloride and sulfur trioxide can be found in the same sample because of the equilibrium nature of the liquid. Procedures for the direct determination of pyrosulfuryl chloride

Property	Value	Typical analysis
appearance	clear, mobile liquid, colorless to slightly straw colored	
color, APHA <sup>a</sup>	100	<10
turbidity, APHA <sup>a</sup>	16	<5
assay, ClSO $_3$ H, wt % <sup>b</sup>	98.5	99.4
total Cl as HCl, wt $\%^b$	30.8	31.2
iron as Fe, $ppm^a$	5	$1^c$
free SO <sub>3</sub> , wt $\%^a$	0.7	0.4
sulfuric acid, wt $\%^a$	1.5	0.4
aluminum as Al		$<1^{c}$

Table 2. Specifications for Chlorosulfuric Ac
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<sup>a</sup> Value given is maximum value.

 $^{b}$  Value given is minimum value.

<sup>c</sup> Value is given in ppm.

have also been described (42, 43), but are not generally required for routine analysis. Small concentrations of sulfuric acid can be determined by electrical conductivity.

Spot tests for determining chlorosulfuric acid are based on the use of powdered tellurium, which gives a cherry-red color, and powdered selenium, which gives a moss-green color in the presence of the acid.

#### 7. Health and Safety Factors

#### 7.1. Safety

Chlorosulfuric acid is a strong acid and the principal hazard is severe chemical burns when the acid comes into contact with body tissue. The vapor is also hazardous and extremely irritating to the skin, eyes, nose, and respiratory tract. Exposure limits for chlorosulfuric acid have not been established by OSHA or ACGIH. However, chlorosulfuric acid fumes react readily with moisture in the air to form hydrochloric and sulfuric acid mists, which do have established limits. The OSHA 8-h TWA limits and ACGIH TLV–TWA limits are sulfuric acid = 1 mg/m<sup>3</sup>; hydrochloric acid = 5 ppm or 7 mg/m<sup>3</sup> (ceiling limit).

Personal protective equipment should be used whenever contact with the acid could be encountered. Chemical safety goggles, hard hat with brim, safety shoes, and acid-resistant clothing, ie, wool or polyester, should be the minimum requirement when working near chlorosulfuric acid. Where the potential for exposure is higher as in loading, sampling, valve operation, etc, the additional use of an acid-proof jacket, pants, and gauntlet gloves should be a required minimum. For emergencies or where there is a high potential for exposure, the protective equipment should include a complete acid suit with hood, gloves, and boots. In some instances, respiratory protective equipment may be needed.

#### 7.2. Fire Hazard

Although chlorosulfuric acid itself is not flammable, it may cause ignition by contact with combustible materials because of the heat of reaction. Open fires, open lights, and matches should not be used in or around tanks or containers where hydrogen gas may be collected because of the action of chlorosulfuric acid on metals. Water, carbon dioxide, and dry-chemical fire extinguishers should be kept readily available.

#### 7.3. Storage and Handling

The acid should never be allowed to stand in a line completely sealed between two closed valves or check valves. Excessive pressure caused by thermal expansion of the liquid can cause leaks or pipe ruptures. All lubricants and packing materials in contact with chlorosulfuric acid must be chemically resistant to the acid. Flanged connections are recommended over screwed fittings and flange guards should be used.

#### 7.4. Spills and Waste Disposal

Chlorosulfuric acid spills generally have a liquid and vapor hazard associated with them. The liquid is an extremely strong acid; fumes react with moisture in the air to form dense clouds of hydrochloric acid and sulfuric acid mists. When the fumes pose the greater danger, the use of a fog nozzle to dilute the acid to nonfuming strengths or the use of a foam to blanket the acid is recommended.

Depending on the magnitude of the spill, control can be achieved by absorption (qv) into absorbents such as expanded clay, diatomaceous earth, or sand (see Clays; Diatomite). The materials can be removed from the area for controlled dilution with water and/or neutralization with alkali. The use of limestone, ashes, or sand-soda ash mixtures have the advantage of both absorption and partial neutralization. SPILL-X-A (Ansul Fire Protection Co., Marinette, Wisconsin), a magnesium oxide-based absorbent and neutralizer, reduces vapor emissions and neutralizes and solidifies spills of fuming acids such as sulfur trioxide, 65% oleum, and chlorosulfuric acid (44).

Spills can also be diluted with large volumes of water. Care should be taken, however, because chlorosulfuric acid reacts violently with water liberating heat, hydrochloric acid, and sulfuric acid mists and steam. The water should be applied from a safe distance upwind of the spill using a fog nozzle. Remaining traces of acid should be neutralized with soda ash, caustic soda, or lime before disposal.

### 8. Uses

Surfactant manufacturing from chlorosulfuric acid includes sulfates of olefins or unsaturated oils, sulfates of polyoxypropylene glycol, sulfonates of long-chain alcohols, particularly lauryl sulfonate, and alkylated diphenyl ether. Pharmaceuticals such as sulfa drugs (see Antibacterial agents, sulfonamides), synthetic sweeteners (qv), anticoagulants (see Blood coagulants and anticoagulants), phenolphthalein, substituted sulfuric acids and salts, diuretics, and active chlorine agents for disinfection, are made from chlorosulfuric acid. Dyes and pigments (qv) from chlorosulfuric acid include acid dyes, vat dyes, monoazo dyes (see Azo dyes), phthalocyanine dyes, and surface treatment of polyethylene or polyester fibers and films.

Chlorosulfuric acid is used as a sulfonating agent in the preparation of resin-based ion exchange (qv) materials. Sulfonation of glycol phthalate or benzyl chloride–naphthalene resins with chlorosulfuric acid produces water-soluble resins (qv). Thermosetting resins are produced by reaction of indoles and formaldehyde (qv) using chlorosulfuric acid as a catalyst.

Chlorosulfuric acid exhibits catalytic properties in the following reactions: esterification of aliphatic acids in both liquid and vapor phase, alkylation of olefinic hydrocarbons, preparation of alkyl halides from olefinic halides and isoparaffins with tertiary hydrogen, and preparation of unsaturated ketones from olefins and anhydrides of fatty acids. Miscellaneous uses include preparation of pesticides, plasticizers (qv), tanning agents, textile and paper specialties, fluorocarbons, rubber and plastic release agents (qv), as a vulcanization aid for isoolefin copolymers, condensing agents, source of anhydrous hydrogen chloride, and as a separating agent for mixtures of sulfur dioxide and chlorine.

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C. E. MCDONALD E. I. du Pont de Nemours & Co., Inc.

## **Related Articles**

Sulfuric acid and sulfur trioxide; Detergency; Pharmaceuticals; Dyes and dye intermediates; Chemicals in war