



# CHLORINATED ORGANICS HANDBOOK



OXYCHEM  
TECHNICAL INFORMATION  
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Dallas-based Occidental Chemical Corporation is a leading North American manufacturer of basic chemicals, vinyls and performance chemicals directly and through various affiliates (collectively, OxyChem). OxyChem is also North America's largest producer of sodium chlorite.

As a Responsible Care® company, OxyChem's global commitment to safety and the environment goes well beyond compliance. OxyChem's Health, Environment and Safety philosophy is a positive motivational force for our employees, and helps create a strong culture for protecting human health and the environment. Our risk management programs and methods have been, and continue to be, recognized as some of the industry's best.

OxyChem offers an effective combination of industry expertise, experience, on line business tools, quality products and exceptional customer service. As a member of the Occidental Petroleum Corporation family, OxyChem represents a rich history of experience, top-notch business acumen, and sound, ethical business practices.

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# Introduction to Chlorinated Organics

## Chlorinated Organics

As a result of their physical and chemical properties, OxyChem's chlorinated organics have a wide range of applications as industrial solvents. Compared to other organic solvents, chlorinated organics exhibit unique chemical properties such as high solvency, non-flammability, low boiling point, and high vapor density.

Chlorinated organics are remarkably stable, noncorrosive to most metals, and have low rates of evaporation and low vapor pressures.

OxyChem's family of chlorinated organics include:

- Carbon Tetrachloride (tetrachloromethane)
- Chloroform (trichloromethane)
- Methylene chloride (dichloromethane)
- Perchloroethylene (tetrachloroethylene)

Because of the difference in the properties of these products, each has some particular application for which it is best suited.

## Manufacturing

The OxyChem Chloromethanes plant produces methylene chloride, chloroform, and carbon tetrachloride from the thermal chlorination of methyl chloride. Hydrogen chloride produced in the thermal chlorination reaction is recycled back to the methyl chloride process.

The OxyChem chlorinated organics technology uses a versatile process to produce perchloroethylene (perc) and carbon tetrachloride (carbon tet), with anhydrous hydrogen chloride (HCl) and crude 1,2-dichloroethane (EDC) by-products. A wide variety of feedstocks including C<sub>1</sub> to C<sub>3</sub> hydrocarbons and partially chlorinated hydrocarbons are utilized in this process. Chlorine (Cl<sub>2</sub>), in excess of stoichiometric requirements, is reacted at high temperature with the organic feed to produce perc and carbon tet.

The organic products are separated from the excess chlorine and the HCl in a series of condensation steps, and then further purified in a series of distillation columns.

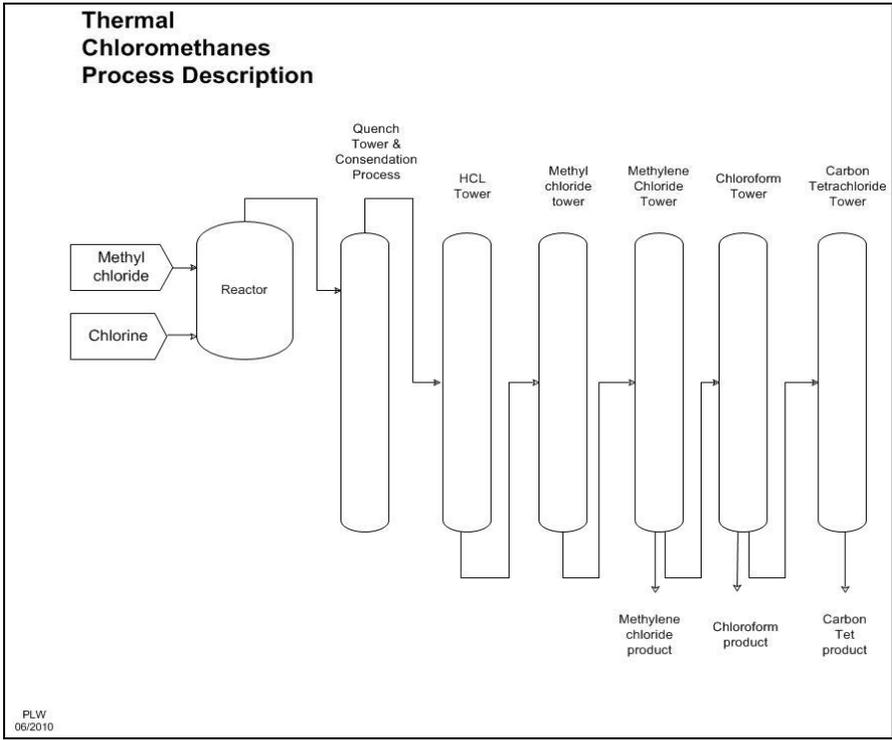


Figure 1: Thermal Chloromethanes Process Diagram

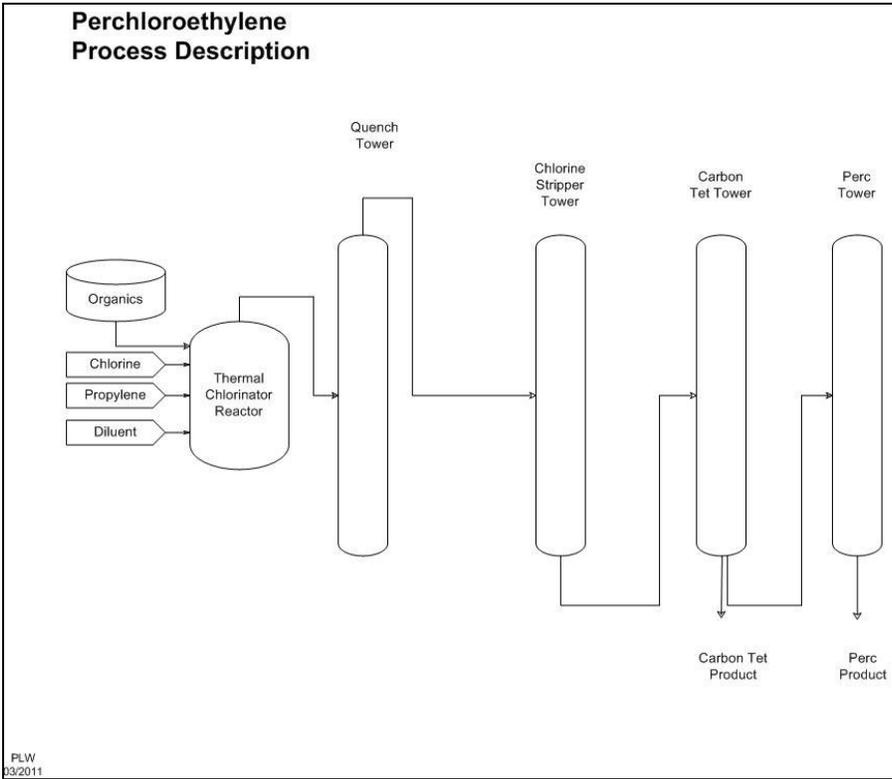


Figure 2: Perc Process Diagram

## Uses and Product Grades

### CARBON TETRACHLORIDE (CTC)

Carbon tetrachloride's use is restricted to non-emissive industrial applications. Although carbon tetrachloride is used for some chemical process reactions today, prior to the Montreal Protocol large quantities of carbon tetrachloride were used to produce the refrigerants R-11 (trichlorofluoromethane) and R-12 (dichlorodifluoromethane). However, these refrigerants are believed to play a role in ozone depletion and have been phased out.

Carbon tetrachloride is tightly controlled under the Montreal Protocol as an ozone-depleting substance (ODS). As a result of the Protocol and the Clean Air Act, most uses of this and other ODSs were banned in the developed economies around the world in 1995.

Use of carbon tetrachloride in consumer products is banned by the Consumer Product Safety Commission (CPSC), under the Federal Hazardous Substance Act (FHSA) 16 CFR 1500.17.

OxyChem produces one grade of carbon tetrachloride as Technical Grade manufactured at both Wichita, KS and Geismar, LA facilities. OxyChem's carbon tetrachloride meets test requirements for General Use according to *ACS Specifications for Reagent Chemicals*, 8<sup>th</sup> Edition, 1993 and the Standard Grade according to *ACS Specifications for Reagent Chemicals* (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)).

### CHLOROFORM

The major use of chloroform today is in the production of the chlorodifluoromethane (R-22), a major precursor to tetrafluoroethylene, which is the main precursor to Teflon®. Before the Montreal Protocol, R-22 was also a popular refrigerant. The U.S. established hydrochlorofluorocarbons (HCFC) production limits in 2002.

**Fluorocarbon Grade:** This grade is the highest purity chloroform manufactured by OxyChem. It is used as a precursor for R-22, which is then transformed into Teflon®. Chloroform Fluorocarbon Grade is manufactured at both the Wichita, KS and Geismar, LA plants.

**Alcohol Stabilized Grade:** This grade is mainly used as a solvent in the extraction and purification of products in the pharmaceutical industry that are not considered to be food related. It is also used in the purification of some antibiotics, alkaloids, and vitamins. A small percentage is also sold as a

laboratory reagent solvent for use as a reaction medium in the preparation of organic nitrogen compounds, acids, aromatic hydrocarbons, ketones, ethers, and other fine chemicals.

The Alcohol Stabilized Grade formerly designated as NF Grade, meets the test requirements for the *National Formulary XVII* (1990) and also the test requirements for General Use Chloroform according to *ACS Specifications for Reagent Chemicals* (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)). The manufacturing process for Chloroform Alcohol Stabilized Grade does not incorporate all of the measures specified in the Food and Drug Administration's current Good Manufacturing Practices (cGMP). It is the responsibility of the user to assess their use of Alcohol Stabilized Chloroform products in food, feed or pharmaceutical related applications and to determine whether appropriate regulatory requirements are being met. The Chloroform Alcohol Stabilized Grade is manufactured only at OxyChem's Wichita, KS facility.

**Technical Grade:** This grade is sold in large part into general distribution where it is used as a chemical intermediate in the preparation of dyes, plastics, resins and pesticides. It can also be used as an industrial solvent in photography. This grade meets the test requirements of Federal Specification O-C-291B (cancelled June 22, 2000 with no replacement) and for General Use Chloroform in *ACS Specifications for Reagent Chemicals* (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)). Chloroform Technical Grade is manufactured and drummed at the OxyChem Wichita, KS facility.

### METHYLENE CHLORIDE

Methylene chloride is a widely used chemical organic with a diverse number of applications. It was introduced as a replacement for more flammable chlorinated organics over 60 years ago. In the mid 1990s methylene chloride replaced 1,1,1-trichloroethane in nonflammable adhesive formulations for industrial applications.

**Aerosol Grade:** Aerosol Grade Methylene Chloride is a higher stabilized product for applications in aerosol packages, adhesives and paint formulations. Methylene chloride is used in aerosols as a strong solvent, a flammability suppressant, vapor pressure depressant, and viscosity thinner. Methylene chloride is an important solvent in adhesive formulations where its strong dissolving power, low flammability and rapid drying time are

essential. It provides adhesive formulations with strong, instant bonding characteristics and efficacy under extremes of temperature and humidity. Current aerosol uses of methylene chloride include specialized spray paints and lubricants.

These same fast-drying, low flammability and high solubility properties make methylene chloride an excellent chlorinated organic in paint formulations. The additional stabilizer helps protect the equipment utilized in non-packaged aerosol applications, as well as additional protection to the application substrate. The EPA suggests methylene chloride as a replacement for 1,1,1-trichloroethane under its Significant New Alternative Policy (SNAP). Aerosol Grade Methylene Chloride is manufactured at the Geismar, LA facility only.

**Decaffeination Grade:** Decaffeination Grade Methylene Chloride is a low stabilized, high purity methylene chloride used as an extractant in the recovery and purification of a wide variety of materials. As a result of methylene chloride's powerful solvent action and stability, it is used in the extraction of naturally occurring heat-sensitive substances such as fats, butter, caffeine, cocoa, hops, and extraction of pharmaceutically active natural products. Methylene chloride is used as an effective reaction and re-crystallization solvent in the extraction of several pharmaceutical compounds and in the production of many antibiotics and vitamins. The chemical also has been used as a carrier for pharmaceutical tablet coating for which the Food and Drug Administration (FDA) has established residue tolerances. This grade meets the test requirements of the *Food Chemical Codex*, the *ACS Specification for Reagent Chemicals*, for General Use, ASTM Specification D 4701-00; and *National Formulary 29* (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)). The manufacturing process for Decaffeination Grade Methylene Chloride does not incorporate all of the measures specified in the Food and Drug Administration's current Good Manufacturing Practices (cGMP). It is the responsibility of the user to assess their use of Decaffeination Grade Methylene Chloride products in food, feed, or pharmaceutical related applications and to determine whether appropriate regulatory requirements are being met. Methylene Chloride Decaffeination Grade is manufactured at the Wichita, KS facility only.

**Degreasing Grade:** It is often necessary to remove grease, oil, or similar substances used as lubricants or temporary protective coatings during metal fabrication. This grade of methylene chloride contains a specifically formulated three-part

stabilizer package for use in vapor degreasing applications. Due to methylene chloride's relatively low boiling point (104.2°F), this chlorinated organic is utilized in the degreasing of temperature sensitive parts, or where immediate handling is required. Methylene chloride is used extensively for this purpose, both for cold (room temperature) cleaning and vapor degreasing of metal parts. This product meets the test requirements of ASTM Specification D 4079-00. Degreasing Grade Methylene Chloride is manufactured at the Geismar, LA facility and drums are available from OxyChem's terminal in Harvey, LA. Degreasing Grade Methylene Chloride can also be sourced from the Lemont, IL terminal facility.

**Special Grade:** OxyChem's Special Grade Methylene Chloride has become widely used to replace fluorocarbons as an auxiliary-blowing agent in the production of slabstock flexible polyurethane foams for the furniture and bedding industries. Evaporation of the chlorinated organic during production of the urethane polymer expands the cells of the foam, reducing its density without making it stiff or rigid. The auxiliary blowing agent also helps to control the reaction temperature, which otherwise could get sufficiently high to burn or scorch the foam interior. In foam blowing applications, use of methylene chloride can eliminate the possibility of hard seams and can allow for ready compliance with flammability requirements for upholstered furniture. Special Grade Methylene Chloride is manufactured at the Geismar, LA plant and drums available at the terminal in Harvey, LA. Special Grade Methylene Chloride can also be sourced from the Lemont, IL terminal facility.

**Technical Grade:** OxyChem's Technical Grade Methylene Chloride is inhibited with one of two stabilizers: amylene or cyclohexane. Methylene chloride's aggressive solvency makes it an ideal paint remover and paint mask cleaner. Methylene chloride is the active ingredient in many formulations of paint removers including industrial paint and commercial furniture strippers, home paint removers, and products used for aircraft maintenance. The chemical has a unique ability to penetrate, blister, and lift a wide variety of paint coatings. Formulations of the chemical are used extensively in both flow-over and immersion (dip) tanks in furniture refinishing operations. For the maintenance of military and commercial aircraft, a methylene chloride based product has commonly been used to inspect the surface for damage. It has no flash point under normal use conditions and can be used to reduce the flammability of other substances, decreasing the chance of in-plant fire

or explosion. Methylene chloride is widely used in the electronics industry for the production of printed circuit boards. Methylene chloride is employed in the manufacture of polycarbonate resin used for the production of thermoplastics. It is used as a chlorinated organic in the production of cellulose triacetate, which serves as a base for photographic film. Other applications include its use in the solvent welding of plastic parts and as a releasing agent to prevent the manufactured part from permanently bonding to the mold. This grade meets the test requirements of the *Food Chemical Codex*, the *ACS Specification for Reagent Chemicals*, General Use, ASTM Specification D4701-00; and *National Formulary 29* (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)). The manufacturing process for Technical Grade Methylene Chloride does not incorporate all of the measures specified in the Food and Drug Administration's current Good Manufacturing Practices (cGMP). It is the responsibility of the user to assess their use of Technical Grade Methylene Chloride products in food, feed, or pharmaceutical related applications and to determine whether appropriate regulatory requirements are being met. The Technical Grade Methylene Chloride is produced at both the Geismar, LA and Wichita, KS locations. Technical Grade Methylene Chloride can also be sourced at the Lemont, IL terminal facility.

## PERCHLOROETHYLENE

Perchloroethylene (Perc) is primarily used as a chemical intermediate in the production of several fluorinated compounds. Its other major uses are as a solvent in commercial and industrial cleaning and degreasing of fabricated metals, and as a solvent in automotive aerosols. OxyChem does not sell perchloroethylene into the dry cleaning application.

**Isomerization Grade:** Isomerization (Isom) Grade Perc is used as a chloriding agent that provides a source of the chloride ion ( $\text{Cl}^-$ ) that acts as a catalyst promoter and reformer. The general requirements for chlorinated organics in isomerization and regeneration applications are for a high purity product with a minimum amount of oxygen and nitrogen compounds. OxyChem's Isomerization Grade Perc with a minimum assay of 99.995% (wt.) and less than 15 ppm organically combined oxygen and nitrogen meets these requirements. This grade meets the requirements of UOP, LLC, a developer of catalyst used in the refining industry. Isom Grade Perc is manufactured at the Geismar, LA facility with drums available from the Harvey, LA terminal.

**Fluorocarbon Grade:** Fluorocarbon Grade Perc is used as a basic raw material in the manufacture of chlorofluorocarbons (CFC's), principally trichlorotrifluoroethane (CFC-113), which is used in the electronics industry. This grade of perc is also used in the synthesis of hydrofluorocarbon 134a (HFC-134a), and can be used for the synthesis of hydrochlorofluorocarbon (HCFC) HCFC-123, HCFC-142b, and HCFC-141b. Fluorocarbon Grade Perc is produced at the Geismar, LA facility with drums available from the Harvey, LA terminal.

**Technical Grade:** Technical Grade Perc serves as a carrier solvent for fabric finishes, rubber, silicones and adhesives. It is used in paint remover formulations and printing inks and as an extraction medium. In addition, perc is a component of chemical masking formulations used to protect surfaces from chemical etchings in the aerospace and other industries. Perc is also used in wool scouring and as a solvent carrier in dyes and water repellents. The textile industry uses perc as a spotting agent for the removal of spinning oils and lubricants. Perchloroethylene is used as an insulating fluid in some electrical transformers as a substitute for polychlorinated biphenyls (PCBs). Relatively small quantities of perc are used in aerosol specialty products and paper coatings. Perchloroethylene is used in aerosol formulations for the automotive aftermarket, particularly for brake cleaning. These formulations provide auto repair shops with highly effective, nonflammable products. Perc Technical Grade is manufactured at the Geismar, LA facility with drums available from the Harvey, LA terminal. Technical Grade Perc can also be sourced from the Lemont, IL terminal facility.

**Industrial Grade:** Industrial Grade Perc is a slightly higher stabilized version of Tech Grade Perc. Applications for this grade of perc are similar to those of Tech Grade, where higher inhibitor content is required such as cold-cleaning operations. Perchloroethylene's non-flammability and low vapor pressure make it an effective cold (room temperature) metal cleaner, when used in compliance with applicable regulatory requirements. Its low vapor pressure contributes to reduced emissions from cold cleaning operations where it is employed. The use of Industrial Grade versus Technical Grade is usually at the user's discretion. Industrial Grade Perc is produced at the Geismar, LA facility with drums available from the Harvey, LA terminal. Industrial Grade Perc can also be sourced from the Lemont, IL terminal facility.

**Vapor Degreasing Grade (VDG):** Many industries, including aerospace, appliance, and automotive

manufacturers, use perchloroethylene for vapor degreasing metal parts during various production stages. Perc's high boiling point (250°F), greater volume of condensate, and resultant longer cleaning cycle are advantageous in removing "difficult" soils. These include removal of high melting point pitches and waxes, and removal of large quantities of water for jewelry manufacturers. Perc is particularly effective in cleaning of spot-welded seams and fine orifices and straight vapor cleaning of light metal parts. This grade meets test requirements of FED OT-236C, Grade B (replaced by ASTM D4376-02); and ASTM D4376-02 (See current product specification sheet at [www.oxychem.com](http://www.oxychem.com)). VDG Perc is manufactured at the Geismar, LA facility with drums available from the Harvey, LA terminal.

### Safety & First Aid Hazards

All of OxyChem chlorinated organic products are hazardous chemicals and should be handled with extreme care. Personnel should be properly trained in the handling of chlorinated organics and should always wear the proper protective equipment when working around chlorinated organics. All users should read the proper Material Safety Data Sheet (MSDS) before handling chlorinated organics. The MSDS for any OxyChem chlorinated organic can be found at the OxyChem website: [www.oxychem.com](http://www.oxychem.com)

The primary health hazard associated with the use of chlorinated organics is overexposure due to the inhalation of vapors above the OSHA PEL (Occupational Safety and Health Association established Permissible Exposure Limits). Limits are updated in the current MSDS for each product.

The PEL is the allowable average concentration to which workers may be exposed, 8 hours per day, five days a week, without adverse effects. OSHA has also established a Short Term Exposure Limit (STEL) for methylene chloride, which is a 15 minute average exposure that should not exceed 125 ppm. Likewise, the American Conference of Governmental Industrial Hygienists (ACGIH) has established Threshold Limit Values (TLV) and these values can also be found in the current MSDS for each product.

Work areas employing chlorinated organics should be isolated and contained. A local exhaust should provide adequate ventilation to limit solvent vapors to below exposure limits.

It is recommended that employees be provided with and required to use chemical impervious clothing, gloves, boots, splash proof goggles and other appropriate protective clothing necessary to prevent any possibility of skin contact with chlorinated organics. Material types which may be considered for this service include: Teflon®, Viton®, PE/EVAL, CPF3, Responder®, and Tychem®, PVC, Nitrile, PVA. Face shields can augment protection provided by splash-proof goggles and safety glasses, but are not intended to replace these safety appliances. (Figure 3)

#### References:

EPA Guidance Document for the Halogenated Solvent Cleaner NESHAP, EPA-453/R-94-081  
Halogenated Solvents Industry Association, White Papers, March 2008.

[http://www.hsia.org/white\\_papers/paper.shtml](http://www.hsia.org/white_papers/paper.shtml)

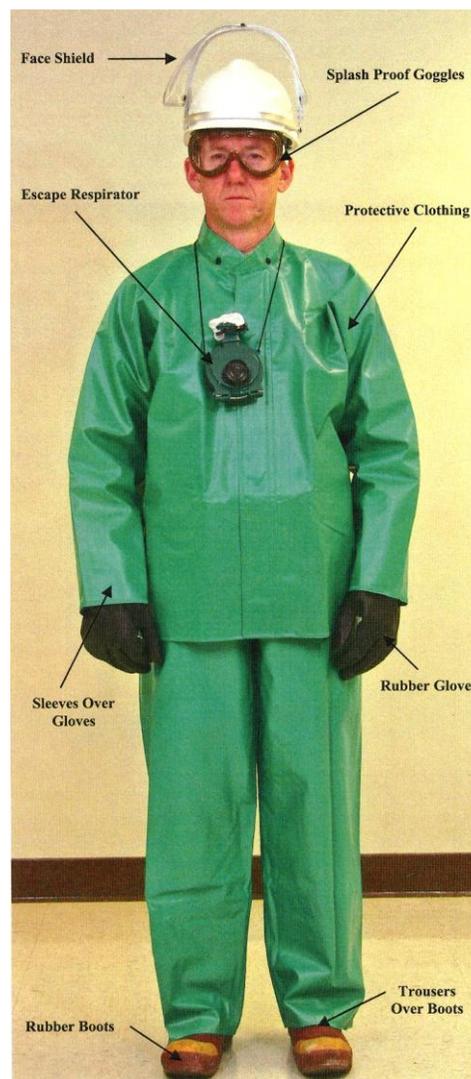


Figure 3: Personal Protective Equipment

HSDB, Hazardous Substances Databank, Last Revision Date: 20050624.

## Respiratory Protection

OSHA requires that employees using respirators should be properly fitted and trained in their use.

**Basically there are 3 types of respirators:**

**1. Escape:** In areas where the unexpected release of chlorinated organic vapors may lead to potentially dangerous exposure, appropriate escape respirators should be carried by or be readily accessible to each employee. The most common respirator used for this purpose is the mouthpiece respirator. This respirator contains a single cartridge with a mouthpiece and nose clip. Employees should only use this respirator when escape times are short and airborne concentrations of chlorinated organic vapors are low. (Figure 4)



Figure 4: Mouthpiece Respirator (Escape Only)

**2. Air Purifying Respirators:** Air purifying respirators contain cartridges or canisters of absorbent or reactive material to remove harmful gases from breathing air. These respirators are available as either half face or full-face units. For chlorinated organic service, use a cartridge or canister respirator designed for chlorinated organic service. (Figure 5)

Where vapor concentration exceeds or is likely to exceed OSHA or NIOSH recommended exposure levels, a cartridge or canister respirator should be used. When working in environments where airborne concentrations are irritating to the eyes, full-face piece respirators should be used. Cartridge or canister respirators are strictly air-purifying devices and must never be used in an oxygen deficient atmosphere (less than 19.5% oxygen by volume), in environments immediately

dangerous to life or health (IDLH), or areas containing unknown concentrations of chlorinated organics.



Figure 5: Full Face Air Purifying Respirator

It is important to remember that cartridges and canisters have a limited service life. Conditions such as humidity, chemical concentrations in the workplace, other chemicals in the workplace, and frequency of use will affect cartridge and canister service life. Therefore, an evaluation of workplace conditions should be made to determine the appropriate cartridge/canister replacement schedule.

**3. Self-Contained Breathing Apparatus:** Self-contained breathing apparatus (SCBA) can provide respiratory protection in an oxygen-deficient environment and in situations where unknown concentrations of chlorinated organic vapors are present. The SCBA can also provide protection in emergency situations. (Figure 6)

The SCBA is an atmosphere-supplying respirator for which the breathing air source is designed to be carried by the user (OSHA definition). A full-face mask is always used with this type of apparatus. OSHA requires that when wearing the SCBA in an Immediately Dangerous to Life and Health (IDLH) atmosphere, the SCBA must be operated in the pressure demand mode and be certified by NIOSH for a minimum service life of thirty minutes. Escape SCBA devices are commonly used with full face pieces or hoods, and depending on the supply of air, are usually rated as 3 to 60 minute units.

OSHA requires that all respirators must be NIOSH approved and shall use breathing gas containers marked in accordance with the NIOSH respirator certification standard, 42 CFR Part 84. For further information on regulations pertaining to respirator equipment, see 29 CFR 1910.134 and 30 CFR § 57.5005 For additional information see DHHS

(NIOSH) Publication No. 2005-100, *NIOSH Respirator Selection Logic 2004* or Publication No. 87-116, *NIOSH Guide to Industrial Respiratory Protection*.



Figure 6: Self-Contained Breathing Apparatus

## Safety Precautions

In the absence of air and water, chlorinated organics are stable in cool dry conditions. In contact with air or water it is subject to very slow hydrolysis or oxidation, either reaction offers the possibility of the formation of trace amounts of HCl. This can lead to discoloration in storage tanks. Chlorinated organics can be stored in vessels made of common materials of construction, except aluminum, magnesium, zinc, and their alloys.

Avoid contact with pure oxygen, strong alkalis, alkali metals, open flames and welding arcs, or other high temperature sources which induce thermal decomposition to irritating and corrosive HCl.

Dry chemical, carbon dioxide, foam, water fog or spray should be used to extinguish fires. Water may be ineffective, but should be used to keep fire-exposed containers cool.

Safety showers and eye wash fountains should be located in the immediate work area and clearly marked. These units should be tested on a regular basis. Portable or temporary systems are available. Every precaution should be taken to ensure that a suitable system is in place and operational before handling chlorinated organics. ANSI Standard Z358.1 contains placement and performance criteria for emergency eyewash and shower equipment. Only trained and properly protected personnel should be allowed to enter areas where chlorinated organics are present.

Before entering tanks or opening pipelines that have contained chlorinated organics, they should be completely emptied and checked for vapors before entering. Do not enter a confined space (which includes tanks or pits) without following proper entry procedures such as 29 CFR 1910.146.

Good housekeeping practices are important where chlorinated organics are used. If a chlorinated organic leak occurs, evacuate the area and eliminate all ignition sources. Response personnel should wear self-contained breathing air and protective equipment to prevent contact with vapor, liquid or hazardous decomposition products.

See Page 21 of this handbook for specific recommendations on spills and precautions.

## First Aid

The physiological effects of the chlorinated hydrocarbons may vary from product to product. The following information is therefore general. If more specific information is needed on any chlorinated organic, refer to the applicable OxyChem Material Safety Data Sheet or contact OxyChem Technical Service Department in Wichita, KS at 800-733-1165 ext. 1.

**Eye Contact** - Direct contact of chlorinated organics with the eyes will result in pain, watering, and inflammation. To prevent eye contact, protective eye wear (such as splash goggles, a full face shield, or safety glasses with side shields) must be worn. If eyes come in contact with chlorinated organics, hold the eyelids apart and flush the eye gently with a large amount of lukewarm water for at least 15 minutes, forcibly holding eyelids apart to ensure complete irrigation of all eye and lid tissues. Washing eyes within several seconds is essential to achieve maximum effectiveness. **GET MEDICAL ATTENTION IMMEDIATELY.**

**Skin Contact** - Prolonged exposure of the skin to chlorinated organics will remove the skin's natural oils, causing the affected area to become red, rough and dry. If exposure continues, the chlorinated organics may produce a burning sensation. Prolonged contact with chlorinated organics can cause contact dermatitis. To prevent irritation, wear protective clothing (including gloves) when working with chlorinated organics. If skin comes in contact with chlorinated organics, immediately wash exposed skin with plenty of water while removing contaminated clothing, jewelry and shoes. Wash clothing and thoroughly clean shoes

before reuse. GET MEDICAL ATTENTION IMMEDIATELY.

**Inhalation** – The principle hazard associated with chlorinated organics usually arises from accidental or prolonged overexposure to product vapors. Exposure to high vapor concentrations may cause a depression of the central nervous system resulting in cardiac arrest, fainting, unconsciousness, and possible death.

The effects of overexposure include headache; nausea; dizziness; stimulation or depression; vomiting; staggering; blurring of vision; loss of appetite; fast, irregular or weak pulse.

The hazards encountered in limited overexposure are lack of alertness, light-headedness, and impaired coordination. The worker may become a hazard to him/herself and to others working in the area.

Concentrations capable of causing serious effects are highly unlikely if work and storage areas are well ventilated and proper protective equipment is used.

To avoid overexposure to chlorinated organic vapors, monitor the vapor concentration in the work place. If vapors are found above the OSHA PEL, install engineering controls (such as localized ventilation) to reduce chlorinated organic vapor concentrations to a safe operating level. In areas where the concentrations of chlorinated organic vapors are either unknown or cannot be maintained below the limits, workers must be equipped with NIOSH approved respirators as per the OSHA Respiratory Protection standard.

If chlorinated organics are inhaled, remove individual to uncontaminated area. Give artificial respiration if not breathing. If breathing is difficult, oxygen should be administered by qualified personnel. If respiration or pulse has stopped, have a trained person administer Basic Life Support (Cardio-Pulmonary Resuscitation/Automatic External Defibrillator) and CALL FOR EMERGENCY SERVICES IMMEDIATELY.

**Ingestion** – Accidental ingestion of chlorinated organics may result in irritation of the gastrointestinal tract, nausea, vomiting, diarrhea, bloody stools, unconsciousness and death. Carbon tetrachloride and chloroform are liver and kidney poisons. Proper labeling, handling and storage of chlorinated organics will reduce the likelihood of accidental ingestion. If ingestion occurs, never give anything by mouth to an unconscious or convulsive

person. If swallowed, do not induce vomiting. If vomiting occurs spontaneously, keep airway clear. Do not give fluids. GET MEDICAL ATTENTION IMMEDIATELY.

**NOTE TO PHYSICIAN:** All chlorinated organics listed in this handbook are an aspiration hazard. Risk of aspiration must be weighed against possible toxicity of the material (see ingestion) when determining whether to induce emesis or to perform gastric lavage. This material sensitizes the heart to the effects of sympathomimetic amines. Epinephrine and other sympathomimetic drugs may initiate cardiac arrhythmias in individuals exposed to this material.

**CARBON TETRACHLORIDE:** May cross the placenta and may be excreted in breast milk. Alcohol may enhance the toxic effects. Those exposed to alcohols, ketones, phenobarbital, methamphetamine, or other barbiturates, other brominated or chlorinated solvents, DDT, PBB, chlordecone, nicotine, carbon disulphide and other alkyl disulphides or experiencing hypoxia also might be more sensitive. Stimulants such as adrenaline may induce ventricular fibrillation.

**METHYLENE CHLORIDE:** This material is metabolized to carbon monoxide. Consequently, elevations in carboxyhemoglobin as high as 50% have been reported, and levels may continue to rise for several hours after exposure has ceased. Data in experimental animals suggest there is a narrow margin between concentrations causing anesthesia and death.

**Traumatic Shock** - Whenever injured persons are being cared for, the person administering first aid should watch for signs of traumatic shock. Traumatic shock may follow serious injury and is a depressed condition of many body functions due to inadequate blood circulation throughout most of the body. Signs of shock are pale, moist, cool skin; shallow and irregular breathing; and weak pulse.

Beads of perspiration may be noted about the lips, forehead, palms, and armpits. The patient may become nauseated.

To treat shock, keep the patient lying down and as warm and comfortable as possible. Raise the patient's feet eight to twelve inches unless there is head injury, breathing difficulty, or if the patient complains of added pain.

## Toxicity

Up-to-date toxicity information is available from the Material Safety Data Sheets. Read the MSDS before use.

The OxyChem Technical Service and Product Stewardship organizations, as well as many trade associations, including the Chemical Manufacturers Association (CMA), have trained personnel to answer questions about the toxicity of these products.

Chronic Health Effects for chlorinated organics vary in degree of chronic (long term) toxicity. The body may experience liver, kidney, and other internal organ damage from overexposure to chlorinated organic vapors. The effects of chlorinated organics will vary with the product, duration and concentration of the chlorinated organic exposed to, as well as the individual being exposed. Specific chronic effects of chlorinated organics can be found in the MSDS.

All of the chlorinated organics have been studied to determine the potential to cause cancer in humans. Five different agencies have classified the chlorinated organics related to their ability to cause cancer. Carcinogenicity classifications vary between agencies, and can be found in the MSDS.

Carbon tetrachloride is classified by NTP as a Reasonably Anticipated Human Carcinogen; and by IARC as a Human Inadequate Evidence, Animal Sufficient Evidence, Group 2A carcinogen and is listed by OSHA as causing cancer.

Perchloroethylene is classified by NTP as an Anticipated Human Carcinogen, and by IARC as a Human Limited Evidence, Animal Sufficient Evidence, Group 2A.

## Regulatory

The following is a summary of regulations and sources that may pertain to chlorinated organics.

### U.S. Environmental Protection Agency

- Clean Air Act: All the chlorinated organics listed in this handbook are included on the Clean Air Act, Section 112(b) list of Hazardous Air Pollutants.

#### CHLOROFORM:

Urban Air Toxics Strategy: Identified as one of 33 HAPs that present the greatest threat to public health in urban areas

NSPS: Manufacture of substance is subject to certain provisions for the control of Volatile Organic Compound (VOC) emissions

#### PERCHLOROETHYLENE

A New Source Performance Standard has been developed for perchloroethylene under Clean Air Act, Section 111.

<http://www.epa.gov/air/caa/>

- Clean Water Act: Perchloroethylene is included on the Clean Water Act, Section 126 list of Priority Pollutants.

Effluent limitation guidelines have been developed for perchloroethylene under Section 304B of the Clean Water Act.

<http://www.epa.gov/water/>

- Comprehensive Environmental Response, Compensation, and Liability Act: Releases of Chlorinated organics in excess of the Reportable Quantity must be reported. See Table 3 for Reportable quantities for individual products

	Reportable Quantities
Carbon Tetrachloride	10 lbs.
Chloroform	10 lbs.
Methylene chloride	1000 lbs.
Perchloroethylene	100 lbs.

<http://www.epa.gov/lawsregs/laws/cercla.html>

- Emergency Planning and Community Right-to-know Act: OxyChem chlorinated organics are listed substances that are subject to reporting requirements under Section 313 of the act. These reports are often called Toxic Release Inventory (TRI) Reports.

<http://www.epa.gov/lawsregs/laws/epcra.html>

- Resource Conservation and Recovery Act
  - Carbon Tetrachloride: Listed Hazardous Waste Codes in which listing is based wholly or partly on carbon tetrachloride include U211, D019, F001, F024, F025, K016, K019, K020, K021, K073, K116, K150, K151, and K157.
  - Chloroform: Listed Hazardous Waste codes in which listing is based wholly or partly on chloroform - K044, K009, K010, K019, K020, K021, K029, K073, K116, K149, K150, K151, K158
  - Methylene Chloride: Listed Hazardous Waste Codes in which listing is based wholly or partly on methylene chloride include U080 and F002.
  - Perchloroethylene: Listed Hazardous Waste Codes in which listing is based wholly or partly on perchloroethylene include U210, F002, and D039.

<http://www.epa.gov/lawsregs/laws/rcra.html>

- Safe Drinking Water Act— Effluent limitation guidelines have been developed for the listed chlorinated organics under Section 304B

<http://www.epa.gov/lawsregs/laws/sdwa.html>

#### CARBON TETRACHLORIDE:

Consumer Product Safety Commission: Carbon tetrachloride and mixtures containing it (with the exception of chemicals containing unavoidable residues of carbon tetrachloride that do not result in concentrations of carbon tetrachloride greater than 10 ppm) are banned in consumer products.

#### Stratospheric Ozone Depletion

In 1992, the EPA issued its final rule implementing Section 604 of the Clean Air Act. That section implemented the United States' obligations under the Montreal Protocol by limiting the production and consumption of substances with a potential to deplete stratospheric ozone. **Carbon Tetrachloride** was identified as Ozone Depleting Substances (ODS). This rule, published as 40 CFR Part 82, Subpart A, required the phase out of production of these products for emissive uses on December 31, 1995.

Exceptions to the ban include product manufactured for transformation (chemical intermediate) processes, and 'essential' uses as approved by the EPA.

Additionally, 40 CFR Part 82, Subpart E requires that products containing or manufactured with Ozone Depleting Substances (ODS) must be

pecially labeled identifying the presence of an ODS.

### **Significant New Alternatives Policy**

The Clean Air Act also required the EPA to establish a program to identify alternatives for ODS, and to publish lists of acceptable and unacceptable substitutes. The program, the Significant New Alternatives Policy (SNAP) program is described in 40 CFR Part 82, Subpart G. **Methylene chloride** and **perchloroethylene** are listed as acceptable replacements under the SNAP program.

This regulatory summary also does not address any similar state or local regulations, some of which may impose additional or different obligations from those imposed by federal regulations. All users are responsible for a complete review of the applicable regulations pertaining to their own operation.

### **US Department of Homeland Security**

#### **CHLOROFORM:**

Listed as Release Chemical of Interest on Appendix A of the CFATS security regulations, 6 CFR 27.

# Handling and Storage

## Shipping

Chlorinated organics may be delivered in bulk by either tank trailers or by tank cars. Since there are significant differences in the equipment configurations of these two modes of transport, different unloading procedures are required.

The typical unloading procedures presented in this section are designed for deliveries to top-loading storage tanks. These procedures may require modifications for deliveries to bottom-loaded storage tanks, or for other variations in storage or delivery equipment.

### TANK TRAILER or TANK CAR UNLOADING PROCEDURES

**Only properly trained personnel should be involved in unloading operations. An operator must be present throughout the unloading procedure. Written procedures must be readily available, and followed. A safety shower and eyewash must be readily accessible. The unloading area must be adequately lit. All DOT regulations in CFR, Title 49 must be obeyed.**

*OxyChem recommends adhering to the requirements found in 49 CFR 177.834 regarding the unloading of tank trailers as being a good work practice.*

Review all customer safety policies with the delivery driver upon entry into the customer's site. The driver should be shown the location of the nearest safety shower and eyewash station, and the location of any other safety equipment necessary. Additionally, the driver should verify with a knowledgeable customer representative the unloading line and storage tank intended to convey and hold the delivered product, respectively. DOT requirements in 49 CFR 177.834 state qualified person "attends" the loading or unloading of a cargo tank if, throughout the process, he is alert and is within 7.62 m (25 feet) of the cargo tank. The qualified person attending the unloading of a cargo tank must have an unobstructed view of the cargo tank and delivery hose to the maximum extent practicable during the unloading operation. If these requirements cannot be met solely by the driver then a representative from the customer site will be asked to be present during the entire unloading.

## Pump Unloading

This is the preferred method for unloading tank trailers. Transfers of chlorinated organics by pump allow the option of vapor recovery, in which vapors are returned from the storage tank to the transportation vessel via hose. However, if vapor recovery is not utilized, the tank trailer dome lid or vapor recovery inlet must be opened before commencing the transfer process to prevent tank trailer or pump damage.

Product can be transferred by use of a customer-supplied pump. Customer pumps must be of a self-priming design and have a pressure relief bypass valve set at 35 psig. maximum. (Customers requiring a pump on the delivery trailer must specify when placing orders with OxyChem's Customer Service Department).

1. Tank trucks or tank cars containing OxyChem chlorinated organics should be located on a level, paved surface in a designated unloading area.
2. All unloading and receiving areas for chlorinated organics should be surrounded by total containment for the control of potential spills and leaks. Where large scale containment (i.e., diking) is not practical, drip pans or other suitable containers must be placed under connections in the event of a leak.
3. Set hand brakes and wheel chocks, and establish an electrical ground for the tank truck or tank car, unloading lines, pump, and storage container.
4. Verify the tank truck's or tank car's contents by sampling or check the placards, bill of lading, and certificate of analysis. Verify that the receiving tank and lines are properly labeled, and that the tank has sufficient volume to hold the delivered product. **DO NOT BEGIN THE UNLOADING PROCESS IF ADEQUATE CAPACITY DOES NOT EXIST.**

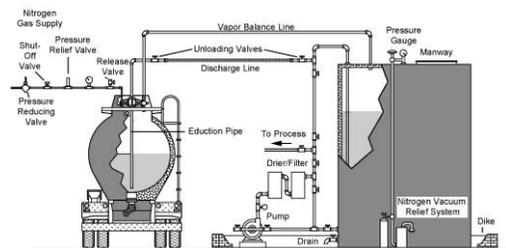


Figure 7: Tank Truck or Tank Car Top Unloading Diagram

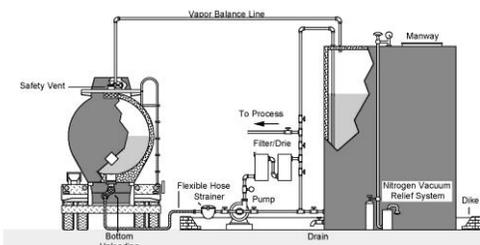


Figure 8: Tank Truck or Tank Car Bottom Unloading Diagram

5. Check all transfer lines and hoses to make sure they are clean, dry, and free of any contamination. Whenever possible, use pumps and flexible hoses dedicated to the certain product service. Tank trucks can be provided with their own pumps upon request and several sections of chemically resistant hoses. Verify that they are compatible with the product received. The size and location of the valves and/or outlets will differ somewhat from truck to truck. Inspect all connections and the pump to ensure they contain the proper gasket material and packing, and that they are clean, dry and free of contamination. Tank cars have valves on the top of the car under the dome cover.
6. Connect one section of the 2-inch flexible hose to the tank truck's 2-inch bottom or rear unloading valve and to the suction side of the pump. If using the tank truck's pump, connect another section of the hose from the pressure side of the pump to the storage tank or permanent unloading line. Connect the unloading line to the liquid valve connection at the manway on the tank car and connect return vapor line to the vapor valve connection on tank car. Check to make sure all connections are secure.



Figure 9: Truck Trailer Rear Product Outlet

7. Ensure that workers involved in the unloading procedures are fitted with the proper safety

equipment including safety shoes, hard hats, gloves, protective glasses or safety shield, and the appropriate respiratory equipment. Only properly trained personnel should be involved in unloading operations. An operator must be present throughout the unloading procedure.

8. Release any pressure in the trailer or car by slowly and carefully opening one of the safety vents on top of the trailer or car. Chlorinated organic vapors should be vented through an emission control system during unloading. Whenever possible, connect the vents from the storage container and the shipping vessel allowing them to breathe the same vapor space, thereby eliminating vapor emissions to the atmosphere.

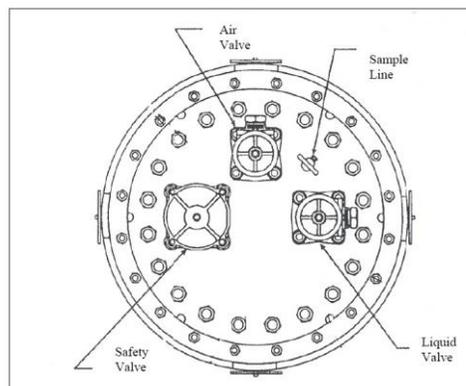


Figure 10: Typical Dome Valve Arrangement

9. Open the storage tank inlet valve, followed by the tank truck's or tank car's unloading valve. Turn on the pump and begin transferring product. At the same time, have another operator check all connections for any sign of leaks.
10. After the tank truck or tank car is empty (or the pump can no longer remove chlorinated organic), shut off the pump, close the tank inlet valve, then close the trailer's or car's unloading

valve. Disconnect the flexible 2-inch unloading line from the trailer or tank car and drain as much product as possible. Disconnect the flexible line to the suction side of the pump, and collect any dripping in a suitable container. Close the valve on the pressure side of the pump, and disconnect the hose.

11. If the tank truck's pump was used to unload the container, close the valve on the pressure side of the pump, then close the tank inlet valve or the valve at the end of the dedicated unloading line. Disconnect the flexible hose and collect any product remaining in a suitable container.
12. Reseal the tank truck or tank car by closing the safety vent. Replace any caps that may have been used to keep the valves clean. Remove hand brakes and wheel chocks before releasing the trailer or tank car.

### Tank Trailer Equipment

Both single and multi-compartment tank trailers are available for shipment of chlorinated organics. Each compartment is equipped with a bottom outlet valve, a vapor recovery inlet and a pressure activated vent. All OxyChem Private Carriage tank trailers are equipped with two 20-foot sections of unloading hoses. The receiving line should have a two(2) inch male quick connect fitting to complete the tank trailer hose connection.

**To facilitate unloading services, specific requests for a trailer equipped with a pump; unique hose length requirements; or special unloading line connectors should be made when orders are placed.**

### Pressure Unloading

In this method of delivery, air is supplied either by customer compressor or by tractor air. Tractor air is most common and utilizes the delivery tractor's brake system air supply. Regardless of air supply source, an air supply free of oil, moisture, and particulate matter is required for product purity. Sites utilizing air transfers must have a pressure gauge; a pressured regulator set at no higher than 30 psig; a pressure relief valve set at 35 psig maximum; a safety shut off valve, as well as a pressure bleed-off valve. Depending upon compressor station design and other factors, installation of air filtering/purifying devices in the air line delivering compressed air may be required.

NOTE: Vapor recovery cannot be used when unloading by air.

Tank trucks or tank cars containing OxyChem chlorinated organics may also be pressure unloaded using nitrogen or air (dew point less than -40°C/-40°F) to pressure offload moisture sensitive products.

**NOTE: The transfer procedures are written for top loading storage tanks only. The exact sequence for filling bottom-loaded storage tanks may vary.**

### Storage & Equipment

Written procedures detailing requirements for proper handling, safety equipment, first aid training, unloading and loading procedures are good practice and in many cases, required by law. They should include detailed instructions for handling and reporting spills.

Considerable care should be taken when transferring chlorinated organics to maintain high product quality. These same careful practices will ensure the health and safety of workers, and ensure that no product is allowed to escape into the air, soil or water.

An above ground tank must be used whenever a large volume of chlorinated organics is to be stored at a customer or terminal site. Storage tanks should be large enough to contain a minimum of 150% of the normal delivery volume.

Top-filled tanks are preferred over bottom-filled tanks. Use of bottom-filled tanks requires installation of check valves in the product transfer line to prevent catastrophic tank draining, should the transfer line fail.

Storage tanks previously used to contain other products may be used for storage, but must be thoroughly cleaned, water tested, drained and dried prior to being placed in chlorinated organics service. New tanks should be prepared in the same manner. Since water testing is not always sufficient to detect small liquid or vapor leaks, the tank should be monitored closely during the initial filling with product to check for leaks not detected during water testing. A halide meter (or equivalent) should be used for monitoring. It is recommended that the halide meter be used as part of a routine maintenance program.

Storage tanks may be mounted horizontally or vertically. Storage tanks used for chlorinated organics should meet the American Petroleum

Institute (API) Standard 650, Welded Tanks for Oil Storage; API standard 620, Design & Construction of Large, Welded, Low-pressure Storage Tanks; ASME Section VIII, or other suitable design and fabrication standards. All local regulations concerning above ground storage tanks should be reviewed and all permits obtained before installing a bulk storage system.

Bulk storage containers should be constructed of either carbon or stainless steel. **Aluminum or fiberglass reinforced plastic storage tanks are prohibited for chlorinated organic service.** Storage tanks should not be constructed of, nor contain, any non-compatible plastic components.

The storage tanks exterior should be cleaned, primed and painted with a white or aluminum colored paint to aid in keeping the tank and its contents cool.

#### **Storage Tank Pads or Saddles**

Saddles used to support horizontal tanks may be constructed of reinforced concrete or steel. Vertical tanks may be supported by an oiled sand base over crushed stone or on a concrete pad. The design of the concrete pad or saddle foundation (if horizontal) should be based on at least the total weight of the tank filled with product also soil conditions, frost line, wind loads, and other factors which may influence safe support of a tank full of product..

#### **Secondary Containment**

All storage facilities should be designed to protect the environment from contamination through the use of secondary containment. Typical secondary containment systems employ impermeable surfaces such as double-walled tanks, sumps, dikes (non-earth). All storage tanks should be diked to contain the tank contents in the event of a spill or tank rupture. They should be large enough to contain the tank's volume and an additional appropriate volume as a safety factor. **(Containment volumes and diking requirements are often defined and mandated by individual states and localities. Regulations must be reviewed prior to construction.)**

An effective secondary containment system will include not only the tank area, but also the pump site and the unloading (hose connection) area. Dikes may be constructed of concrete or concrete treated with a chlorinated organic resistant epoxy-phenolic sealant. Avoid the use of asphalt-based materials or non-coated concrete in secondary containment systems or in the unloading area. Where diking does not apply, chlorinated organic

resistant buckets or other appropriate portable spill containers should be used. Tank trailer unloading areas should avoid the use of French drains, storm water sewers, or unlined pits.

#### **Manways**

Manways on tanks are necessary for access to the tank for inspection and cleaning. Two manways, 24 inches in diameter, are desirable. One manway should be located on top of the tank, while the second should be positioned on the side near the base for empty tank inspection and clean out procedures.

#### **Piping**

Piping of seamless Schedule 40 black iron, carbon steel or stainless steel is usually satisfactory. **Galvanized steel, aluminum or plastic pipe should not be used.** Transfer pipes are typically 2-inch in diameter, but may be as large as 6-8 inches for barge and vessel deliveries. Fittings may be carbon steel or stainless steel, and may be threaded, flanged or welded. Threaded piping is only recommended for sizes less than 3/4" in diameter. It is essential that the pipe be threaded two full turns before applying threading compound or Teflon® tape to eliminate the possibility of contaminating the interior of the pipe. All pipes should be free of oils and any other contaminants prior to being placed in service.

#### **Drain Lines**

Storage tanks should be equipped with a flanged and valved outlet drain at the floor level to allow the tank complete drainage if necessary. This outlet should be located such that it can be tied into a pump. Since this outlet is rarely used, the valve should be plugged and equipped with a locking system to prevent any accidental releases.

#### **Outlet Line**

An outlet line for normal product withdrawal should be located 3-4 inches above the tank floor. It should be tied into the drier-filter pump system. This piping system ensures that clean, dry product can be loaded without duplication of equipment. A safety valve should be provided between the tank and the pump as a means for shutting off the flow of product should a problem arise with the pump.

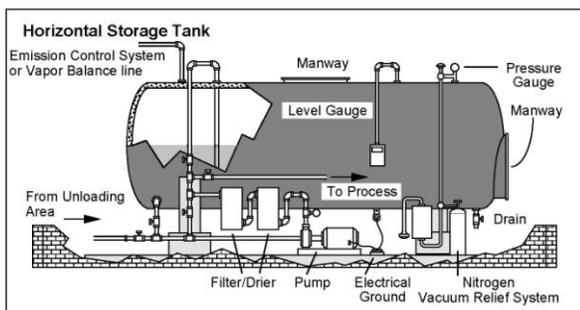
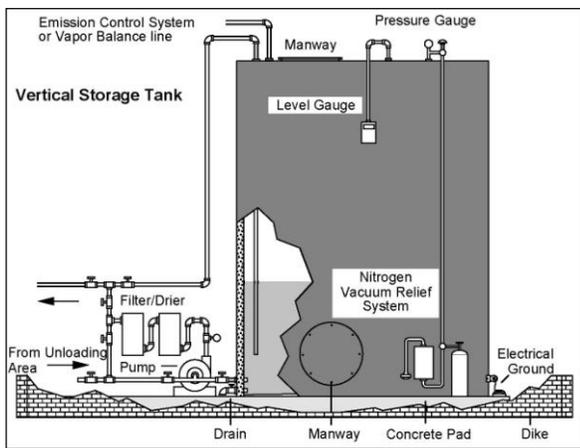


Figure 11: Storage Tank diagrams

## Valves

Carbon steel, ball, globe (for bypass), plug, and butterfly valves are satisfactory. Ball valves should have a PTFE seat; globe valves and all-metal seat valves should be of stainless steel or nickel alloy. Valve stem packing can be of Teflon® construction.

Ball or plug valves are preferred where full line flow is desired. Where throttling is necessary, globe valves are recommended. Globe angle valves can be used in piping system to eliminate some additional fittings.

## Pumps

Pumps may be either positive displacement or centrifugal type, and can be constructed of steel, stainless steel, or alloy 20. All packings must be made of chlorinated organic resistant materials such as PTFE. Pumps used for drumming should supply product at a rate of approximately 50 gpm. The pump should be located so that a positive head pressure is always maintained on the suction side.

If unusual pressures or pumping conditions are encountered, a positive displacement pump may be used. Positive displacement pumps must be equipped with a bypass or relief valve to eliminate possible damage should the pump be operated while the discharge valve is closed.

Pumping facilities, which transfer product through a filter, should employ a pump discharge pressure gauge. Gauge readings can help determine the filter replacement frequency. Placement of a strainer in front of the pump's intake is suggested to prevent pump damage from rust and particulate matter found in the tank. See "filters" section for additional information.

## Bypass Line

A bypass line must be provided when utilizing a positive displacement pump, and is recommended for centrifugal pumps to protect against pipe damage and undue strain on the pump.

## Emergency Shut-Off Valve

An emergency shut-off valve should be located at the outlet of the vessel feeding the pump.

## Meters

Chlorinated organics meters must be suitable for use with the specific product being handled. Data on suitability is best obtained directly from the meter manufacturer. For best results, it should also contain an air eliminator, strainer, and temperature compensator. Meters can be constructed of bronze, stainless steel or carbon steel.

**Chlorinated organics meters must not be constructed of any reactive metals such as aluminum, zinc, brass or magnesium alloys.**

## Tank Level Indication

Several gauging methods are used to determine the chlorinated organics volume inside storage tanks. Typically dP cells, sonic, or a float with electronic read out are used.

## Electrical Ground

An electrical ground line is a required safety feature that provides a point of discharge for any build-up of static electricity from pumping dry chlorinated organics.

## Hoses

Recommended materials of construction include braided or seamless stainless steel, polytetrafluoroethylene (PTFE or Teflon®), and some grades of Viton®. If a composition other than stainless steel or Teflon® is used, it must first be tested to ensure compatibility with the specific chlorinated organic product. Fixed piping should be incorporated in place of hose to minimize long-term maintenance costs where possible. Two-inch diameter seamless solvent resistant hose is recommended for use in tank trailer unloading, while three-inch hose is suggested for tank car

service. Two sections of 20-foot hose is provided with the delivery vessel (tank trailers only).

### **Gaskets**

Gaskets used in the service of chlorinated organics may be constructed of PTFE or Teflon® envelope gasket material or graphite with stainless steel metal inserts. Rubber-based products such as neoprene or Buna N gasketing should not be used. If a composition differing from those mentioned above is to be used, it must first be tested with the specific chlorinated organic product to ensure compatibility.

### **Other Non-Metal Wetted Parts**

All non-metal wetted surfaces such as internal pump, valve and meter parts must be constructed of components that are inert to the specific chlorinated organic product, such as Teflon®. **Neoprene and natural rubber parts cannot be used for chlorinated organic service.** Other plastic products such as ultra-high molecular weight, high molecular weight, irradiated, or cross-linked polyethylene, polyvinyl alcohol, or other common polymeric compounds must first be tested with the specific chlorinated organic product to ensure compatibility.

### **Line Filters**

An inline filter is recommended to eliminate any particulate contamination which might develop in handling or storage. This filter should be installed so that it will serve dual purpose of filtering product going into, as well as out of, the storage tank. Filters of small micron pore size can be used to remove insoluble contaminants that can accumulate through product transfers. Filters should be made of solvent resistant materials, and should be adequately sized for the required flow rate.

### **Line Driers**

Chlorinated organics can absorb moisture from the air during product transfer. Desiccants such as Drierite® (calcium sulfate), molecular sieve (4A), alumina or certain ion exchange resins (Dow X811®) may be used for drying liquid chlorinated organics. Before using another desiccant material, laboratory tests should be run to determine compatibility and performance. The chlorinated organic filter and drier should be arranged in series such that product passes through each during transfer to and from storage.

### **Pressure/Vacuum Relief System**

Tanks should also be equipped with a pressure-vacuum relief system to maintain the integrity and safety of the tank. The set points for such a system are typically design values of the vessel.

### **Safety Seal**

If the storage tank is fitted with a liquid overflow line, the line must be equipped with a seal. A filled U-trap can serve as a seal, but the use of a seal pot is recommended.

### **Vent Scrubbers**

An emission control device such as a scrubber or low temperature condenser may be fitted to the vent (and may be required by law). Vent scrubbers may contain a chlorinated organic trapping media, such as activated carbon, to prevent emissions to the atmosphere.

All chlorinated organics storage tanks should be equipped with a desiccant dryer in the tank vent line. The dryer is necessary to prevent the entry of atmospheric moisture into the tank as the tank breathes due to temperature and barometric changes or when the product is withdrawn. Moist air can cause corrosion inside the tank and lead to possible product degradation. The dryer can be charged with anhydrous calcium chloride nuggets, anhydrous calcium sulfate, silica gel, or other suitable desiccant. It must be checked on a periodic basis and maintained as necessary.

## **Maintenance of Storage Facilities**

Testing of tanks to be used above atmospheric pressure will usually be specified by local codes. A minimum hydrostatic test pressure of one and one-half times the working design pressure is recommended. Normally the tank fabricator will perform the tests required before delivery, if specified. Before placing the tank into service, an inspection should be made to ensure that the tank is thoroughly dried and that all foreign material and particles are removed. Tank cleaning is especially important when a used tank that may have contained organic-based material is intended for chlorinated organic storage.

One of the primary hazards associated with the use of chlorinated organics occurs when employees are exposed to high concentrations of the vapor in an enclosed area. Adequate ventilation must be supplied to remove vapors before hazardous concentrations can accumulate. Chlorinated organic vapors are expected to be highest during storage tank clean out. During confined area entries, special care must be taken to ensure employee safety. **The following procedures were adapted from the ASTM D 4276, Standard Practice for Confined Area Entry and are listed as a starting point for developing**

**procedures. You must follow the OSHA requirements given in 29 CFR 1910.146.**

(For the purpose of these instructions, a confined area entry refers to the entering of any tank vessel, sump, pit, duct, tank car, tank truck, van trailer, or enclosed space in which a chlorinated organic product was contained.)

**Clean Out Preparation**

All personnel involved in tank clean out procedures should be properly trained in safe entry and rescue procedures. They should have a working knowledge and understanding of the hazards that may exist.

Entry into confined spaces should be by written entry permit, issued by the responsible supervisor or other qualified person. The purpose of the entry permit is to ensure that a checklist of precautions has been reviewed prior to entry. This permit is an authorization and approval in writing certifying that all existing hazards have been evaluated, and necessary protective measures have been taken to ensure the safety of the worker. The permit should be valid for a limited time only (usually 8-10 hours), and a new permit should be required in the event of any job interruption or any indication of changes in job conditions. Issuance of the entry permit should address all of the following considerations:

**Vessel Entry**

1. All process lines exiting or entering the confined space should be disconnected, capped off, and blinded. Closing of valves alone is not adequate protection.
2. Pumps connected to the enclosed area or any other mechanical or electrical equipment, especially conveyors, are to be locked out by locking the main electrical switch in the "OFF" position.
3. A system for positive ventilation should be provided prior to, and during, entry periods. Fans, exhaust vents, air movers, or natural drafts that are capable of ventilating the confined air space within several minutes are necessary.
4. Prior to entry, the enclosed space must be tested for oxygen content, lower explosive limit, and toxic vapor concentration. The oxygen content must be between 19.5% and 22.0% in all levels of the tank. The atmosphere must be absent of toxic vapors. The concentration of chlorinated organic vapors may be determined using the appropriate NIOSH approved gas detection tubes, a calibrated halide meter, portable flame ionization gas chromatograph, or a portable infrared (IR) analyzer.

5. Personal protective equipment should include an approved respirator (Self Contained Breathing Apparatus [SCBA]) or supplied air respirators are recommended), hard hat, safety glasses or splash goggles, gloves, and long sleeves. In addition, any person entering the enclosed area must be fitted with a safety harness and lifeline. The lifeline should be secured outside the entrance.
6. When entry into the vessel must be made through a top opening, a hoisting device or other effective means must be provided to lift the worker out of the space. Ladders must be in place for entrances and exits where the drop or climb involves a depth of more than three feet.
7. Self-contained breathing apparatus or supplied air respirators should be available at the vessel entrance. These devices must be worn if testing finds the chlorinated organic level to be above the OSHA Permissible Exposure Limit (PEL).
8. A second person must be available at the area entrance and in sight of the person inside at all times. They should be equipped with proper safety equipment and adequate communications equipment for summoning additional help if necessary (i.e. a two way radio, whistle, etc.). Under no conditions should this observer enter the affected area without others standing by. If the observer is required to leave his or her post, the person inside the enclosed space must exit.
9. Approved low-voltage electrical equipment must be used where the atmosphere in the confined area may contain flammable vapors or where the atmosphere could contain chlorinated organic vapors within their flammable limits. All electrical circuits should be equipped with a ground-fault interrupter.
10. Entrances to confined space should be posted, identifying the area as a confined space and that a permit is required for entry. During the work, when there is more than one entrance to the confined area, signs indicating that workers are inside, posted at each entrance, are advised.
11. Before entering a storage tank or other chlorinated organic vessel for cleaning or inspection, any remaining product should be drained or removed by vacuum, and placed in a safe container awaiting disposal or reuse. The tank should then be nitrogen dried or steamed or filled with water, drained, ventilated, and checked for presence of vapors.

12. Remove metal fines or chips, rust, and all other insoluble contaminants from the tank bottom. Scrape any baked on contaminants if necessary using an appropriate wire brush to avoid sparking. Repair defects to the tank surface as necessary.
13. After removing insoluble contaminants, rinse all chlorinated organic wetted surfaces thoroughly with an alcohol or water rinse. If cleaning a tank where acidity has been a problem, all wet surfaces should be scrubbed with a wire brush and a 5% soda ash (sodium carbonate) or baking soda (sodium bicarbonate) solution to remove metal chlorides and visible patches of corrosion.
14. After cleaning, rinse the tank thoroughly with several successive water rinses to remove any remaining impurities. Dry the tank completely by ventilating for several hours. Do not refill with product until the tank is completely dry.
15. Close manways and all other openings and secure vessel. Open the appropriate entrance and exit lines, and prepare the tank to be filled with product. (New gaskets are recommended to ensure the integrity of the product and to prevent leaks.) For large bulk storage tanks, purge the tank with nitrogen to a dew point of -40°F. An oxygen content of less than 1% may also be desired.

## Storage of Non-bulk Containers

Non-bulk containers such as drums of chlorinated organics should be stored in a cool, dry, well ventilated area away from direct sunlight. Avoid stacking drums more than one high unless drum-racking systems are employed. Drums should be stored in the vertical position with the bungs (openings) located on top. Bungs should always be adequately tightened to prevent accidental leakage or vapor loss. Precautions should be taken to protect these containers from prolonged contact with moisture, since water contamination entering through the bung openings may result in corrosion and subsequent solvent degradation. Although all chlorinated organics listed in this handbook are non-flammable, empty or full chlorinated organic drums should never be stored near a direct heat source, such as flames, or arcs.

Always store chlorinated organic drums in areas equipped with secondary containment systems. Containment systems should be adequate to hold

110% of the largest expected amount of drummed product to be stored, and should be impermeable to chlorinated organics.

## Labeling

All storage vessels (tanks, drums, etc.) should be properly labeled to comply with OSHA Hazard Communications Standards (29CFR Part 1910.1200). In-plant product containers must be labeled, tagged or marked with the identity of the product and appropriate hazard warnings. Never store chlorinated organics in open or unlabeled containers.

## Spills and Precautions

Employees should be familiar with the purpose, use, and maintenance of the appropriate safety equipment. Personal protective equipment must never be used as a substitute for good, safe working practices.

Where direct contact with liquid chlorinated organics is likely, solvent resistant gloves made of neoprene, Viton®, or polyvinyl alcohol should be worn. Chemical safety goggles and a solvent resistant apron are also recommended, especially where splashing is a problem. Use of contact lenses is not recommended.

Good housekeeping practices are mandatory where chlorinated organics are stored or used. Storage facilities and work areas should be designed to handle accidental spillage or leakage. Spills should be cleaned up immediately to prevent buildup of excessive vapor concentrations. Chlorinated organics should never be flushed into a sanitary sewer or other outlet which connects to waterways or uncontrolled runoff streams. It is recommended that dikes and emergency planning procedures be provided for spills and emergencies. Contact federal, state or local authorities for applicable regulations.

## Odor Threshold

All chlorinated organics exhibit unique characteristic odors. Although smell can be useful in detecting the presence of chlorinated organic vapors, it is dangerous to rely on smell to determine solvent vapor levels. Odor detection varies with the individual products. Additionally, individuals exposed to chlorinated organics for long periods of time experience olfactory fatigue, requiring a higher concentration of vapors before the nose is capable of detection. Before initiating work where vapor levels are unknown, vapor monitoring should be conducted to identify the actual vapor concentrations.

### **Educating Employees**

Chlorinated organics are safe chemicals when they are treated with respect by those who handle or use them. All employees should be educated as to the proper handling of these chlorinated organics and informed of their potential hazards. Employees should be instructed in the proper action to take in the event of an accidental spill and/or exposure. Locations of eyewash fountains, and safety showers, and points of building egress should be easily accessible and clearly marked.

### **Good Personal Hygiene**

Good personal hygiene practices will minimize the accidental intake of chlorinated organics. Employees should never eat, drink, or smoke near chlorinated organics. After working with chlorinated organics, always thoroughly wash hands with soapy water.

### **Flammability**

One of the principle advantages of chlorinated organics is the non-flammability. None of the chlorinated organics listed in this manual exhibit flash or fire points as determined by standard laboratory testing procedures.

Methylene chloride has a limited flammability range when high vapor concentrations are mixed with air and exposed to a high-energy ignition source. Under these conditions, methylene chloride can burn while exposed to a heat source, but will not support combustion. These conditions are unlikely during normal process operations.

Chlorinated organics, when exposed to elevated temperatures such as those encountered in open flame or electric heaters and welding operations, can decompose to form a highly toxic mixture of phosgene and hydrogen chloride gases. Extreme care should be exercised in all operations involving welding or cutting on chlorinated organic storage tanks and piping. Do not weld or cut on empty chlorinated organic drums.

## Technical Data

### Physical Properties

Some of the physical properties of OxyChem Chlorinated Organics are listed below:

#### Carbon Tetrachloride

CAS: 56-23-5  
Other names: Tetrachloromethane  
Molecular Weight: 153.81  
Physical State: Clear Liquid, Colorless  
Odor Threshold: 50 ppm  
Boiling Point: 170F (76.7C)  
Freezing Point: -9F (-23C)  
Vapor Pressure: 91 mm Hg@20C  
Specific Gravity: 1.59@25/25 C  
Vapor Density (air=1): 5.32  
Water Solubility: 0.08% @ 25C  
pH: No data  
Volatility: 100%  
Evaporation Rate (ether=1): 0.3  
Partition Coefficient (n-octanol/water): 2.83  
Flash point: None  
Autoignition Temperature: None  
IDLH: 200 ppm

#### Chloroform

CAS: 67-66-3  
Other names: Trichloromethane  
Molecular Weight: 119.37  
Physical State: Clear Liquid, colorless  
Odor Threshold: 205-307 ppm (causes olfactory fatigue)  
Boiling Point: 142 F (61.1C)  
Freezing Point: -83F (-63.9C)  
Vapor Pressure: 160 mmHg@20C  
Specific Gravity: 1.4 @ 25/25C  
Vapor Density (air=1): 4.1  
Water Solubility: 0.8% @ 25C (alcohol stabilized)  
pH: Not applicable  
Volatility: 100%  
Evaporation Rate (ether=1): 0.56  
Coefficient of water/oil distribution: log Kow=1.97  
Flash point: None  
Autoignition Temperature: >1832 F (>1000C)  
IDLH: 500 ppm

#### Methylene Chloride

CAS: 75-09-2  
Other names: Dichloromethane  
Molecular Weight: 84.94  
Physical State: Clear Liquid, colorless  
Odor Threshold: 200-300 ppm (causes olfactory fatigue)  
Boiling Point: 104 F (40C)  
Freezing Point: -139F (-95C)  
Vapor Pressure: 350 mmHg@20C  
Specific Gravity: 1.3 @ 25/25C

Vapor Density (air=1): 2.9  
Water Solubility: 1.32% @ 25C  
pH: Not applicable  
Volatility: 100%  
Evaporation Rate (ether=1): 0.7  
Coefficient of water/oil distribution: log Kow=1.25  
Flash point: None  
Autoignition Temperature: 1033 F (>556.1C)  
IDLH: 2300 ppm

#### Perchloroethylene

CAS: 127-18-4  
Names: Tetrachloroethylene, Perc, PCE  
Molecular Weight: 165.82  
Physical State: Clear Liquid, colorless  
Odor Threshold: 50 ppm ( may cause olfactory fatigue)  
Boiling Point: 250 F (121C)  
Freezing Point: -2F (-19C)  
Vapor Pressure: 13 mmHg@20C  
Specific Gravity: 1.62 @ 25/25C  
Vapor Density (air=1): 5.8  
Water Solubility: 0.015% @ 25C  
pH: Not available  
Volatility: 100%  
Evaporation Rate (ether=1): 0.1  
Coefficient of water/oil distribution: log Kow=2.88  
Flash point: None  
Autoignition Temperature: Not applicable  
IDLH: 150 ppm

### Product Sampling

1. In general, an eight ounce sample is adequate for confirming product specifications. Before transferring bulk shipments of OxyChem chlorinated organics, the shipping vessel should be sampled and analyzed to ensure product quality.
2. Sampling should only be accomplished with a closed system that has built-in capabilities to handle vents, provide nitrogen, process unused liquid volume and results in a sample in a closed container.
3. Samples should be stored in glass with polyseal screw caps or caps lined with poly-fluorocarbons. Caps made of other HDPE or irradiated PE may also be used if non-volatile residue is not important. If caps are metal lined, make sure neither tin nor aluminum are used. Do not use plastic coated (inside or outside) sample bottles when sampling chlorinated organics since the plastic coating can contaminate the product.

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4. For barges, vessels, and one compartment trucks, a single sample is sufficient. For multiple compartment trucks or barges, samples should be taken and analyzed for each compartment.
  5. Samples taken as retains should be labeled with the product name and grade, name and address of supplier, date sent, date received, barge, vessel, truck ID number, and lot number. Samples should be stored in a safe, dry place, away from direct sunlight for an appropriate period, generally ninety days. Afterwards, they should be added to the customer's inventory, or disposed of in accordance with all federal, state, and local regulations.

## Where to Find Equipment

The equipment suppliers listed here are believed to be reliable. This is however only a partial listing as space will not permit a listing of every supplier of each type of equipment.

### Chillers

NuTemp, Inc.  
9655 Industrial Dr.  
Bridgeview, IL 60455  
800.323.3977  
[www.nutemp.com](http://www.nutemp.com)

Xchanger, Inc.  
1401 South 7<sup>th</sup> St.  
Hopkins, MN 55343-7868  
952.933.2559  
[www.xchanger.com](http://www.xchanger.com)

### Filters

The Cary Co.  
1195 W. Fullerton Ave.  
Addison, IL 60101  
630.376.2400  
[www.thecarycompany.com](http://www.thecarycompany.com)

Parker Hannifin Corporation  
Process Filtration Division  
1515 W. South St.  
Lebanon, IN 46052  
317.482.3900  
800.272.7537  
[www.parker.com](http://www.parker.com)

3M Purification  
400 Research Parkway  
Meriden, CT 06450  
203.237.5541  
800.243.6894  
[www.cuno.com](http://www.cuno.com)

### Gaskets

John Crane Inc.  
6400 West Oakton St.  
Morton Grove, IL 60053  
847.967.2400  
800.SEALING  
[www.johncrane.com](http://www.johncrane.com)

Garlock, Inc.  
1666 Division St.  
Palmyra, NY 14522  
315.597.4811  
877.GARLOCK (427.5625)  
[www.garlock.com](http://www.garlock.com)

### Gaskets Cont.

Allstate Gasket & Packing  
31 Prospect Place  
Deer Park, NY 11729-3713  
631.254.4050  
[www.allstategasket.com](http://www.allstategasket.com)

### Flexible Metal Hoses

Senior Flexonics, Inc.  
Hose Division  
815 Forestwood Dr.  
Romeoville, IL 60441  
905.456.4074  
800.267.1975  
[www.senior-flexonics.com](http://www.senior-flexonics.com)

Flexible Metal, Inc.  
2467 Mountain Industrial Blvd.  
Tucker, GA 30085  
770.493.1100  
[www.flexiblemetal.com](http://www.flexiblemetal.com)

### Solvent Resistant Hoses

Crane Resistoflex  
1 Quality Way  
Marion, NC 28752  
828.724.4000  
[www.resistoflex.com](http://www.resistoflex.com)

The Gates Rubber Company  
1551 Wewatta St.  
Denver, CO 80202  
303.744.1911  
[www.gates.com](http://www.gates.com)

### Hose Connections

PT Coupling  
1414 E. Willow  
Enid, OK 73701  
800.654.0320  
[www.ptcoupling.com](http://www.ptcoupling.com)

Civacon  
4304 Mattox Rd.  
Kansas City, MO 64150  
816.741.6600  
888.526.5657  
[www.civacon.com](http://www.civacon.com)

### Meters

Rosemount Measurement Division of Emerson Process  
8200 Market Blvd.  
Chanhassen, MN 55317  
800.999.9307  
[www2.emersonprocess.com](http://www2.emersonprocess.com)

## Meters Cont.

Brooks Instrument  
407 West Vine St.  
Hatfield, PA 19440  
888.554.FLOW  
[www.brooksinstrument.com](http://www.brooksinstrument.com)

The Foxboro Company/Invensys  
5601 Granite Parkway III, Suite 1000  
Plano, TX 75024  
469.365.6400  
[www.foxboro.com](http://www.foxboro.com)

## Pressure Gauges

AMETEK, Inc., U.S. Gauge Division  
900 Clymer Ave.  
Sellersville, PA 18960  
215.257.6531  
[www.ametekusg.com](http://www.ametekusg.com)

Ashcroft Inc.  
Instrument Division  
250 East Main St.  
Stratford, CT 06614-5145  
203.378.8281  
800.328.8258  
[www.ashcroft.com](http://www.ashcroft.com)

## Pressure & Vacuum Relief Valves

The ProTecToSeal Company  
225 West Foster Ave.  
Bensenville, IL 60106  
630.595.0800  
800.323.2268  
[www.protectoseal.com](http://www.protectoseal.com)

Anderson Greenwood Crosby  
PO Box 944  
Stafford, TX 77497  
281.274.4400  
[www.andersongreenwood.com](http://www.andersongreenwood.com)

Varec  
5834 Peachtree Corners East  
Norcross, GA 30092  
770.447.9202  
866.698.2732  
[www.varec.com](http://www.varec.com)

Sentry Equipment Corp.  
966 Blue Ribbon Circle N.  
Oconomowoc, WI 53066  
262.567.7256  
[www.sentry-equip.com](http://www.sentry-equip.com)

## Pumps

Flowserve Corporation  
5215 N. O'Conner Blvd., Suite 2300  
Irving, TX 75039  
972.443.6500  
[www.flowserve.com](http://www.flowserve.com)

Aurora Pump Group  
800 Airport Rd.  
North Aurora, IL 60542  
630.859.7000  
[www.aurorapump.com](http://www.aurorapump.com)

Goulds Pumps, Inc.  
240 Fall St.  
Seneca Falls, NY 13148  
315.568.2811  
[www.goulds.com](http://www.goulds.com)

Sundyne Corp.  
14845 W. 64<sup>th</sup> Ave.  
Arvada, CO 80007  
303.425.0800  
[www.sundyne.com](http://www.sundyne.com)

Vanton Pump & Equipment Corp.  
201 Sweetland Ave.  
Hillside, NJ 07205  
908.688.4216  
[www.vanton.com](http://www.vanton.com)

Waukesha Cherry Burrell  
611 Sugar Creek Rd.  
Delavan, WI 53115  
262.728.1900  
800.252.5200  
[www.goweb.com](http://www.goweb.com)

## Solvent Dryers

Hankison International  
1000 Philadelphia St.  
Canonsburg, PA 15317-1700  
724.745.1555  
[www.hankisonintl.com](http://www.hankisonintl.com)

Lectrodryer  
PO Box 2500  
Richmond, KY 40476-2602  
859.624.2091  
[www.lectrodryer.com](http://www.lectrodryer.com)

AFC-Holcroft  
49630 Pontiac Trail  
Wixom, MI 48393  
248.624.8191  
[www.afc-holcroft.com](http://www.afc-holcroft.com)

## Tanks

Kennedy Tank & Manufacturing Co.  
833 East Sumner Ave.  
Indianapolis, IN 46227-1345  
317.787.1311  
800.445.1344  
[www.kennedytank.com](http://www.kennedytank.com)

RECO Constructors, Inc.  
710 Hospital St.  
Richmond, VA 23219  
804.644.2611  
[www.recoconstructors.com](http://www.recoconstructors.com)

CB&I  
One CB&I Plaza  
2103 Research Forest Dr.  
The Woodlands, TX 77380-2624  
832.513.1000  
[www.cbi.com](http://www.cbi.com)

Brown Tank LLC  
6995 55<sup>th</sup> St. N.  
St. Paul, MN 55128  
651.747.0100  
[www.browntank-mn.com](http://www.browntank-mn.com)

## Tank Gauges

John C. Ernst Co., Inc.  
21 Gail Court  
Sparta, NJ 07871  
973.940.1600  
888.463.7678  
[www.johncernst.com](http://www.johncernst.com)

Emerson  
8200 Market Blvd.  
Chanhassen, MN 55317  
800.999.9307  
[www2.emersonprocess.com](http://www2.emersonprocess.com)

Jogler, Inc.  
9715 Derrington Rd.  
Houston, TX 77064  
281.469.6969  
800.223.8469  
[www.jogler.com](http://www.jogler.com)

The ProTecToSeal Company  
225 West Foster Ave.  
Bensenville, IL 60106  
630.595.0800  
800.323.2268  
[www.protectoseal.com](http://www.protectoseal.com)

## Tank Content Gauges Cont.

Brooks Instruments  
407 West Vine St.  
Hatfield, PA 19440  
888.554.FLOW  
[www.brooksinstrument.com](http://www.brooksinstrument.com)

Magnetrol International, Inc.  
5300 Belmont Rd.  
Downers Grove, IL 60515-4499  
800.624.8765  
[us.magnetrol.com](http://us.magnetrol.com)

## Tank Vent Dryers

Sentry Equipment Corp.  
966 Blue Ribbon Circle N.  
Oconomowoc, WI 53066  
262.567.7256  
[www.sentry-equip.com](http://www.sentry-equip.com)

W.A. Hammond Drierite Co.  
PO Box 460  
Xenia, OH 45385  
937.376.2927  
[www.drierite.com](http://www.drierite.com)

Hankison International  
1000 Philadelphia St.  
Canonsburg, PA 15317-1700  
724.745.1555  
[www.hankisonintl.com](http://www.hankisonintl.com)

## Valves

The William Powell Co.  
2503 Spring Grove Ave.  
Cincinnati, OH 45214  
513.852.2000  
[www.powellvalves.com](http://www.powellvalves.com)

Crane Co.  
19241 David Memorial Dr.  
Shenandoah, TX 77385  
936.271.6500  
[www.cranevalves.com](http://www.cranevalves.com)

Cincinnati Valve Co.  
PO Box 14151  
Cincinnati, OH 45250-1451  
800.471.8258  
[www.lunkenheimercvc.com](http://www.lunkenheimercvc.com)

## Valves Cont.

Flowserve Corporation  
5215 N. O'Conner Blvd., Suite 2300  
Irving, TX 75039  
972.443.6500  
[www.flowserve.com](http://www.flowserve.com)

## Vapor Monitoring Devices & Analysis

Assay Technology  
1382 Stealth St.  
Livermore, CA 94551  
925.461.8880  
800.833.1258  
[www.assaytech.us](http://www.assaytech.us)

3-M Corp.  
Occupational Health & Safety Div.  
3-M Center, Building 275-6W-01  
St. Paul, MN 55144-1000  
888.3M.HELPS  
[solutions.3m.com](http://solutions.3m.com)

SKC, Inc.  
863 Valley View Rd.  
Eighty Four, PA 15330-9613  
724.941.9701  
800.752.8472  
[www.skcinc.com](http://www.skcinc.com)

ESafety, Inc.  
119 Foster St., Building 6  
Peabody, MA 01960  
978.532.7330  
900.462.1103  
[www.esafetyinc.com](http://www.esafetyinc.com)

National Draeger, Inc.  
101 Technology Dr.  
Pittsburgh, PA 15275-1057  
412.787.8383  
[www.draiger.com/us](http://www.draiger.com/us)

Sensidyne, Inc.  
16333 Bay Vista Dr.  
Clearwater, FL 34620  
800.451.9444  
[www.sensidyne.com](http://www.sensidyne.com)

These test methods are provided as a reference in establishing a quality control program. More detailed methods may be found in the American Society for Testing and Materials (ASTM) procedures published in the **Annual Book of ASTM Standards**, Volume 15.05.

### **Specific Gravity**

Determine the specific gravity of the sample at 25/25°C. by means of a precision specific gravity balance or by any other equally accurate means. ASTM D 2111

### **Water Content by Karl Fischer**

This is the preferred method to be used in determining moisture content in chlorinated solvents. ASTM D-3401.

### **Non-Volatile Residue**

Evaporate 1000 mls of sample in a tared platinum evaporating dish to dryness on a steam bath, using inverted volumetric flask technique. Cool in a desiccator and weigh. ASTM D 2109

### **Acidity**

Mix 100 mls of sample with 100 mls of neutral distilled water and shake vigorously for one minute. Add methanolic bromothymol blue indicator and titrate to a green endpoint with 0.01 N. NaOH solution. ASTM D 2989

$$\frac{\text{Mls NaOH} \times 3.65}{\text{SpGr of Solvent}} = \text{ppm Acidity as HCl}$$

### **Acid Acceptance**

This procedure is applicable to degreasing grades of perchloroethylene and methylene chloride only. The acid acceptance is determined by reaction with non-aqueous hydrochloric acid (in isopropyl alcohol) in excess. The excess acid is neutralized with standard sodium hydroxide solution. ASTM D 2942.

#### **I. Apparatus**

1. Iodine or Erlenmeyer flasks 250 ml with ground-glass stoppers.
2. Burettes, 25 ml or 50 ml, graduated to 0.1 ml.
3. Pipettes, calibrated to deliver 25 ml and 10 ml at ambient temperature.

#### **II. Reagents**

1. *Bromophenol Blue Indicator Solution* (1.0 g/liter).

Prepare by dissolving 1 g of bromophenol blue in a mixture of 800 ml of water and 200 ml of denatured ethyl alcohol. The pH range is 3.0 to 4.6. The color change is from yellow on the acid side through blue-green to blue on the alkaline side.

2. *Hydrochlorination Reagent* (0.1 N HCl).

Prepare by dissolving 4.4 ml of concentrated hydrochloric acid (sp. gr. 1.19) in isopropyl alcohol and diluting to 500 ml with alcohol

3. *Sodium Hydroxide, Standard Solution* (0.1N). Prepare and standardize a 0.1 N solution of sodium hydroxide (NaOH).

#### **III. Procedure**

1. Blank. Pipette 25 ml of hydrochlorination reagent and 25 ml of isopropyl alcohol with a 25 ml pipette into a 250 ml Erlenmeyer flask. Add 3 drops of bromophenol blue indicator solution and titrate to a stable blue-green end point with 0.1 N NaOH solution. Record the milliliters of NaOH solution required as *A* (see Section IV for calculation).

2. Sample. Transfer 25 ml of hydrochlorination reagent by means of a 25 ml pipette to a 250 ml glass-stoppered Erlenmeyer flask. Add 10 ml of the dry solvent and 25 ml of isopropyl alcohol. Shake the mixture thoroughly and allow to stand at room temperature for 10 minutes. Add 3 drops of bromophenol blue indicator solution to the sample flask and titrate with 0.1 N NaOH solution to the blue-green end point. Record the milliliters of NaOH solution needed as *B* (see Section IV for calculation).

#### **IV. Calculations**

Calculate the acid acceptance as equivalent NaOH in weight percent as follows:

Equivalent NaOH,

Acid Acceptance as Weight Percent NaOH

$= (A-B) N \times 0.04 \times 100W$

where:

*A* = milliliters of NaOH solution required for blank titration (III.1)

*B* = milliliters of NaOH solution required for sample titration (III.2)

*N* = Normality of NaOH solution

*W* = grams of sample used (volume in ml x specific gravity)

#### **Color**

Compare 100 ml of the sample with 100 ml of a Platinum-Cobalt standard of 10 in Nessler tubes. Report the color of the sample as greater or less than that of the standard. As an alternate, check color with a suitable APHA color scale comparator. ASTM D 2108.

#### **Chlorides**

Place 10 ml of chloride-free water in a 50 ml glass stoppered graduated cylinder and add 40 ml of sample. Shake for 2 minutes; allow settling 5 minutes and adding 2 drops of concentrated nitric acid add 1ml of 0.1N silver nitrate solution. Observe water layer for presence of turbidity as compared to a blank of water and silver nitrate. Presence of turbidity is reported as chlorides present. ASTM D 2988

### ***Free Halogens***

Mix 20 mls of sample with 5 mls of a 10% potassium iodide solution in a glass-stoppered graduated cylinder and shake for one minute. Add 1 or 2 mls of starch solution. The presence of free halogens is indicated by a blue coloration. Report as either positive or negative. ASTM D 4755

### ***Distillation Range***

Conduct a standard ASTM distillation for lacquer solvents and diluents using a thermometer of suitable range calibrated for 100 mm immersion, graduated in 0.2°C units, and capable of being read to 0.2°C. Conduct the distillation at or correct the results to 760 mm of mercury. ASTM D 1078

## FURTHER INFORMATION

More detailed information on OxyChem chlorinated organics or any specific application is available on request through the OxyChem Technical Service Department.

Technical Service Department  
OxyChem  
PO Box 12283  
Wichita, KS 67277-2283  
800-733-1165 Ext. 1  
[www.OxyChem.com](http://www.OxyChem.com)

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