AF/SSD-TR-61-9 R-3136

CHLORINE TRIFLUORIDE HANDLING MANUAL

ROCKETDYNE
A DIVISION OF NORTH AMERICAN AVIATION, INC.
6633 CANOGA AVENUE, CANOGA PARK, CALIFORNIA

CONTRACT AF33 (616)-6939
PROJECT No. 3148
TASK No. 30196

SEPTEMBER 1961

ROCKET TEST ANNEX
SPACE SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
EDWARDS AIR FORCE BASE, CALIFORNIA

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FOREWORD

This manual is one of the four propellant handling manuals prepared under Contract AF33(616)-6939, Supplement 1, PN 3148 TN 30196. The administrative and technical direction of this effort was provided by Messrs. F. S. Forbes, T. Marshall, and J. H. Smith of the AFFTC, Edwards Air Force Base, California. The manuals were prepared by the Analysis and Equipment Group of the Rocketdyne Engineering Department.

The propellant handling manuals were titled and identified as follows:

AF/SSD-TR-61-7	Hydrazine Handling Manual
AF/SSD-TR-61-8	Nitrogen Tetroxide Handling Manual
AF/SSD-TR-61-9	Chlorine Trifluoride Handling Manual
AF/SSD-TR-61-10	Pentaborane Handling Manual

A group of four design-criteria manuals were also prepared under Contract AF33(616)-6939, Supplement 1, PN 3148, TN 30196. These manuals were titled and identified as follows:

AF/SSD-TR-61-6	Mechanical System Design-Criteria Manual for Hydrazine
AF/SSD-TR-61-5	Mechanical System Design-Criteria Manual for Nitrogen Tetroxide
AF/SSD-TR-61-4	Mechanical System Design-Criteria Manual for Chlorine Trifluoride
AF/SSD-TR-61-3	Mechanical System Design-Criteria Manual for Pentaborane

ABSTRACT

This manual presents directly usable information for the safe handling of chlorine trifluoride. The properties of the propellant and techniques for hazards reduction and control are discussed in detail. Selection and preparation of equipment for use with the propellant are also presented and discussed. Propellant transfer procedures using both gas pressurization and pumping techniques are discussed in detail. Other pertinent information such as transportation, storage, and equipment decontamination are also presented.

(Unclassified Abstract)

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INTRODUCTION

This manual presents directly usable information for the safe handling of chlorine trifluoride. The material presented is that evolved from both applicable experience and a thorough evaluation of the available literature, especially that originating from the propellant manufacturer.

The need of reliable information for the safe handling of high-energy propellants is self-evident. Although pertinent literature abounds, the application of the published handling techniques to actual situations cannot be successfully realized in most cases. This frustrating situation can be attributed to the fact that most propellant handling information is obtained as a byproduct of hardware development programs. This manual represents one of the first concerted research efforts to formulate safe propellant handling techniques.

The material covered in this manual is that which is considered essential for the safe handling of the propellant. This material is presented in a very simple and direct manner to make it usable to all personnel involved in propellant handling operations. In addition, the techniques included are expected to provide additional background to the designer of storage and handling systems for chlorine trifluoride.

The safety practices in the manual are based on the principle that the prevention of hazardous situations is the most important consideration for the safe handling of high-energy propellants. However, it is acknowledged that a completely hazard-free facility cannot be ultimately realized; therefore, serious consideration is also given to the control of any hazardous situation that may arise.

This manual consists of eleven sections, each section dealing with a specific subject such as properties of the propellant, materials of construction, handling procedures, and others. This arrangement allows the user to obtain specific information expeditiously.

1.0 GENERAL DESCRIPTION

Chlorine trifluoride (CTF) is a halogen fluoride having the empirical formula, ClF_3. The color of the propellant in the solid state is white, very pale green-yellow in the liquid state, and nearly colorless in the gaseous state. The odor has been described as both sweet and pungent, similar to chlorine or mustard. It is insensitive to mechanical shock, nonflammable in air, and exhibits excellent thermal stability at ambient temperatures.

Chlorine trifluoride is an extremely hazardous propellant due to its toxicity and reactivity. The greatest hazard of the propellant lies in its reactivity, which is surpassed only by liquid fluorine. It reacts with the vast majority of organic and inorganic compounds, and under proper conditions, with most common metals.

As a storable liquid oxidizer, chlorine trifluoride offers for liquid-fueled rockets the readiness approaching that of solid-propellant rockets. Its fluorine content, about 62 percent by weight, places chlorine trifluoride as one of the most promising storable oxidizers presently available.

Chlorine trifluoride boils at about 53 F. As a result, refrigeration is not required to keep the propellant in the liquid state in conventional moderate-pressure vessels. When stored and transferred in clean, dry, compatible systems, and handled by properly trained personnel, chlorine trifluoride need present no serious storage or handling problems.

2.0 PROPERTIES

2.1 General Properties

Commercial chlorine trifluoride contains over 99 percent ClF_3, by weight. The most likely trace impurity is hydrogen fluoride. The properties of the propellant are reproducible.

2.1.1 Appearance

Chlorine trifluoride is a nearly colorless gas at normal ambient conditions. The propellant in its liquid state has a very pale green-yellow color and in the solid state is white.

2.1.2 Odor

Chlorine trifluoride odor has been reported as both sweet and pungent, similar to chlorine or mustard.

2.1.3 Chemical Composition

The specifications of chlorine trifluoride established by the manufacturer for the "as shipped" condition, and expressed in percentage by weight, is as follows:

ClF_3	99 min.
HF	1 max.

At 500 F chlorine trifluoride dissociates, presumably into fluorine and chlorine monofluoride, to the extent of about 1 percent.

2.1.4 Chemical Activity

Chlorine trifluoride is a very strong oxidizing agent. It reacts with most organic and inorganic substances, liberating large quantities of energy. Under ordinary conditions, it reacts violently with ice or water. At elevated temperatures, it will ignite most common metals. The reaction of the propellant with water forms hydrogen fluoride, oxygen, and some oxygen fluoride.

2.2 Physicochemical Properties

Molecular Weight	92.46
Boiling Point	53.15 F
Freezing Point	-105.38 F
Specific Gravity	1.85 at B.P.
Density	115.46 lb/ft^3 at B.P.
Vapor Pressure	17.2 psia at 60 F
	39.7 psia at 100 F
	80.6 psia at 140 F
Viscosity	3.21×10^{-4} $lb_m/ft\text{-}sec$ at B.P.
Surface Tension	0.00170 lb_f/ft at B.P.
Critical Temperature	307 F
Critical Pressure	837.7 psia
Heat of Fusion	35.4 Btu/lb at -103.3 F
Heat of Vaporization	128.1 Btu/lb at B.P.
Heat Capacity	0.303 Btu/lb-F at 41.16 F

The specific gravity and vapor pressure of chlorine trifluoride as a function of temperature are presented in Fig. 1 and 2, respectively.

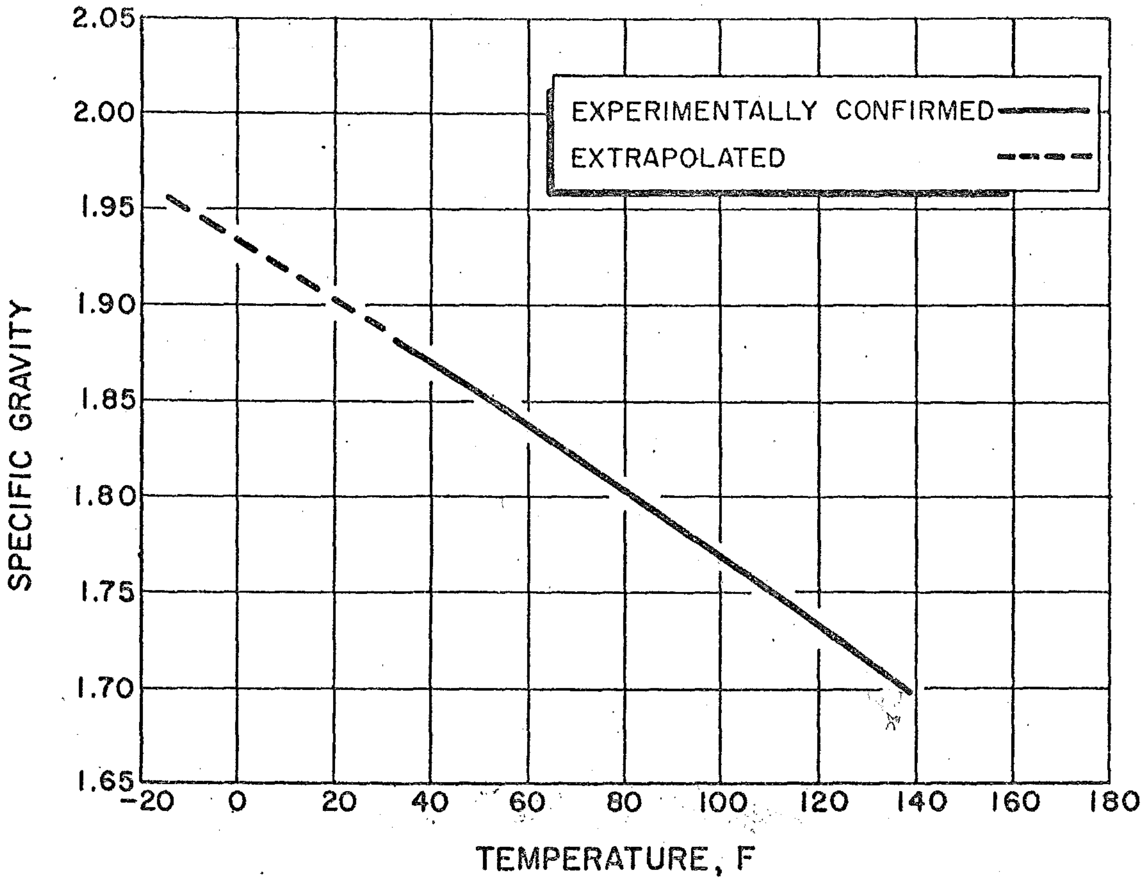

Figure 1. Specific Gravity of Liquid Chlorine Trifluoride

Sg = -0.0017T + 1.9352

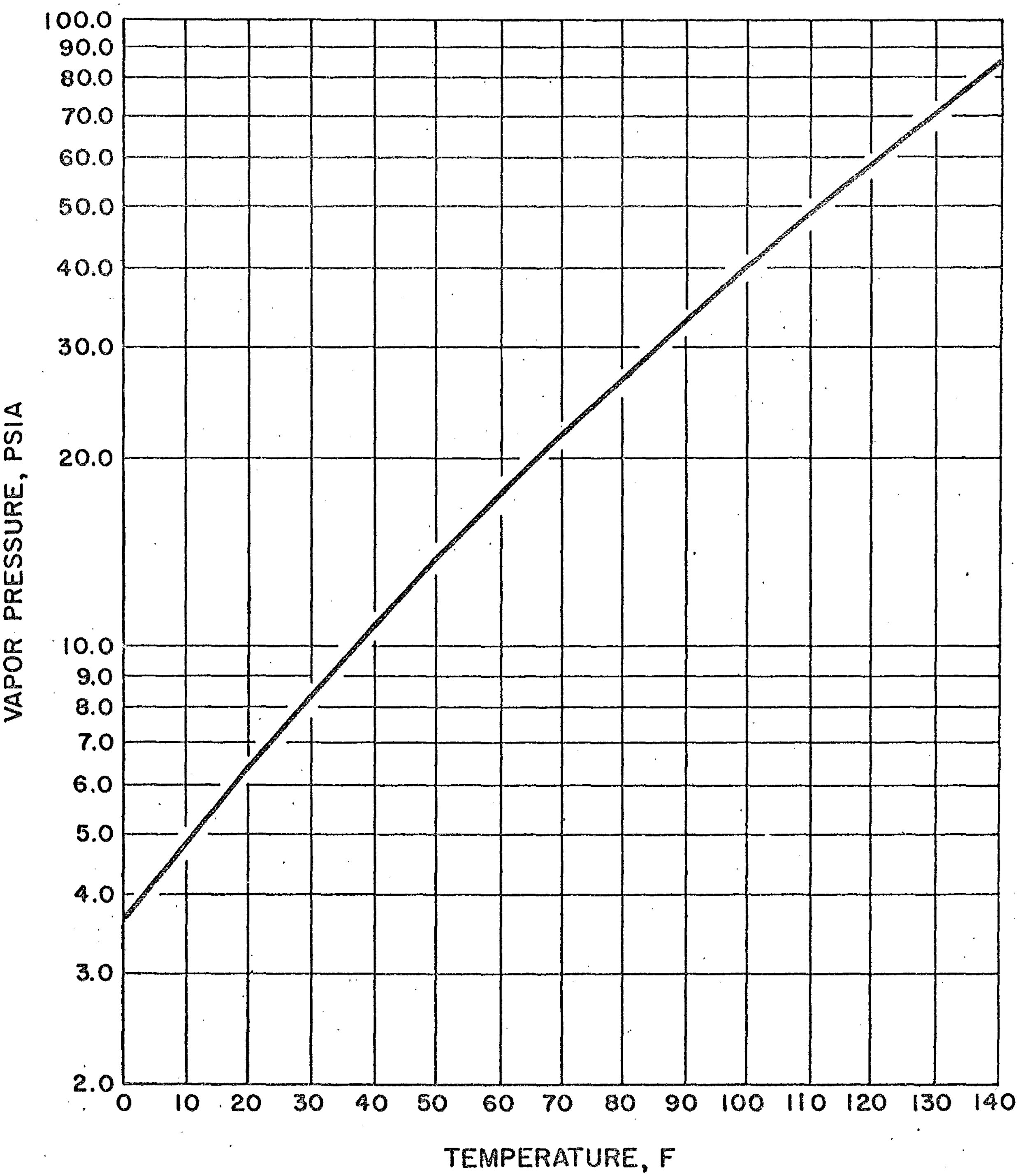

Figure 2. Vapor Pressure of Chlorine Trifluoride

3.0 MATERIALS

3.1 Materials Compatibility

Although chlorine trifluoride is extremely reactive, it can be stored in clean containers fabricated of compact metals such as Monel, copper, stainless steel, etc., due to the formation of a passive metal-fluoride film which protects the metal from further attack. In the presence of grease or other contaminants, chlorine trifluoride will ignite most common metals, including those mentioned above.

It is extremely important that all equipment for chlorine trifluoride service be fabricated of compatible materials, and properly cleaned and passivated. It should be noted that there are no lubricants or nonmetallic materials completely compatible with chlorine trifluoride.

All chlorine trifluoride systems must be dry and leakproof, in addition to being fabricated of compatible materials, and properly cleaned and passivated. This is necessary because the propellant reacts with water to form hydrofluoric acid which is more corrosive to most metals than chlorine trifluoride itself. In addition, the hazardous properties of the propellant are such that leaks and spills must be prevented.

3.1.1 Compatible Materials

The following materials, when properly cleaned and passivated, have been found to be compatible with chlorine trifluoride:

Stainless Steel Type 302	Stainless Steel Type 321
Stainless Steel Type 304	Stainless Steel Type 347
Stainless Steel Type 316	Chromium-Plated Steel

Aluminum Alloy No. 356	Nickel
Aluminum Alloy No. 1100	Monel
Aluminum Alloy No. 2024	K-Monel
Aluminum Alloy No. 5052	Nickel-Base Superalloy Rene-41
Aluminum Alloy No. 6061	
Aluminum Alloy No. 6063	Indium
Aluminum Alloy No. 6066	Boron Carbide
Aluminum Alloy Tens 50	Nitralloy
Copper	Kentanium 162 B

Under propellant static (nonflow) conditions, Teflon has been found to be compatible with chlorine trifluoride. For this application, however, the Teflon must be free of surface impurities and contamination.

3.1.2 Materials for Limited Service

Materials such as carbon, lead, Teflon, Kel-F, and carbon steels are usually considered satisfactory for limited service in chlorine trifluoride. Since these materials are attacked by the propellant under some expected conditions or time duration, their general use is not recommended. As mentioned in the previous paragraph, Teflon can be used on some special applications.

3.1.3 Incompatible Materials

The following materials and lubricants have been found to be incompatible with chlorine trifluoride and they must not be used in direct contact with the propellant:

Lubricants (all common types)	Columbium
Sealants (all common types)	Graphite with plastic binders
Titanium	

Carbon with plastic binders	Saran
Molybdenum	Rubber (all common types)
Polyethylene	Glass and glassed steel
	Nylon

3.2 Preparation of Materials

All components of a chlorine trifluoride transfer and/or storage system must be properly prepared prior to installation. In addition, the assembled system must be carefully dried and passivated. These procedures render all surfaces to be exposed to the propellant chemically inert.

Items such as valves, pumps, etc., cannot be cleaned in the assembled state because it is very difficult to remove all contaminants that might be present. Consequently, the cleaning of these items must be done before the component parts are assembled.

The preparation of materials generally consists of degreasing, descaling, passivating, and drying. The cleaning solutions utilized on these operations shall be applied by immersing, spraying, circulating, or any other manner, as long as the surfaces to be cleaned are completely wetted in the solutions. Any component which can trap or retain liquids shall be drained or emptied between applications of different cleaning solutions.

All solutions shall be made with distilled, deionized, or clean tap water and all chemicals shall be of chemically pure grade or better. The water shall be filtered through a 40-micron nominal-size filter.

3.2.1 Degreasing

Components fabricated of stainless steel, copper, and aluminum alloys can be degreased by cold flushing or vapor degreasing with trichloroethylene, or by flushing with a mild alkaline solution containing from 5 to 7 oz of Turco #4090* (or equivalent) per gallon of water at 140 to 160 F. The application of the mild alkaline solution shall be followed by a thorough water rinse.

Nonmetallic components, such as valve-stem packings fabricated of Teflon, can be degreased by immersion or scrubbing with the mild alkaline solution described above, followed by a thorough water rinse.

Items such as nonmetallic components or simple components fabricated of machine metal stock which are not to be cleaned any further, shall be dried by flushing with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

3.2.2 Descaling

Newly fabricated or reworked components which have scale resulting from welding or heat treatment, or impurities resulting from casting or forging, shall be descaled. Descaling solutions should not be used after precision machining unless the finished surfaces are protected.

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*Turco #4090 is furnished by Turco Products, Inc., 6135 So. Central Avenue, Los Angeles, California.

The descaling of stainless-steel components is accomplished as follows:

1. Etch at room temperature for a period of no longer than 60 minutes with an aqueous solution containing from 3 to 5 weight percent technical grade hydrofluoric acid and from 15 to 20 weight percent technical grade nitric acid.

2. Rinse thoroughly with water to remove all traces of the descaling solution.

NOTE: If the components are to be passivated immediately after descaling, they need not be dried. Otherwise, the components may be dried by purging with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

Components fabricated of copper can be descaled as follows:

1. Descale with an aqueous solution containing about 20 percent (by volume) technical grade hydrochloric acid, at room temperature, until the surfaces are bright and free of oxidation.

2. Rinse with water to remove all traces of the descaling solution.

NOTE: After being descaled, the components require no further chemical treatment. They should be dried by purging with dry, hydrocarbon-free filtered nitrogen gas or by heating in an oven at 140 to 160 F.

The descaling procedure for components fabricated of aluminum alloys is as follows:

1. Clean with Turco Smut-Go* solution (1 lb/gallon of water), or an approved equivalent cleaner, until the surfaces are visibly clean and shiny.

2. Rinse with water to remove all traces of the acid solution. If the components are to be passivated immediately after descaling, they need not be dried. Otherwise, the components may be dried by purging with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

3.2.3 Passivating

The passivation procedure for components fabricated of stainless steel is as follows:

1. Immerse for a minimum period of 30 minutes, at room temperature, in an aqueous solution containing from 45 to 55 percent (by weight) technical grade nitric acid.

2. Rinse with water to remove all traces of the passivating solution.

3. Dry by purging with dry, hydrocarbon-free, filtered nitrogen gas or by heating in an oven at 140 to 160 F.

NOTE: Acid passivation of components having polished or lapped surfaces may be omitted if the finished surfaces cannot be conveniently protected from the acid solution.

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*Turco Smut-Go is a chromic acid cleaner furnished by the Turco Products, Inc., 6135 So. Central Avenue, Los Angeles, California.

Components fabricated of aluminum alloys can be passivated as follows:

1. Immerse for a minimum period of one hour, at room temperature, in an aqueous solution containing about 45 percent (by weight) technical grade nitric acid.

2. Rinse thoroughly with water to remove all traces of the passivating solution.

3. Dry by purging with dry, hydrocarbon-free, filtered nitrogen gas, or by heating in an oven at 140 to 160 F.

3.2.4 Handling

Items that have been prepared for chlorine trifluoride service shall be handled, stored, or packaged in a manner to prevent recontamination. Large components such as valves, piping sections, tanks, etc., should have all openings capped with clean, compatible materials. Small items can be sealed in clean plastic bags.

3.2.5 System Passivation

After the chlorine trifluoride system has been assembled and leak-checked, it is necessary to propellant-passivate the system. This is accomplished by introducing a small amount of gaseous fluorine into the system. The gaseous fluorine not only reacts with any residual contaminating material, but also slowly attacks the containing metal surfaces forming an inert metal-fluoride film.

The passivation of chlorine trifluoride systems can be accomplished as follows:

1. Evacuate the system by means of a high-capacity vacuum pump for at least two hours to remove any volatile contaminant or water vapor that might be present.
2. Place a slight positive pressure in the system using dry, hydrocarbon-free nitrogen gas.
3. Disconnect the vacuum pump, and cap and seal the open system connection.
4. Introduce gaseous fluorine slowly into the system until a pressure of about 20 to 25 psig is obtained.
5. After the gaseous fluorine has been in the system for about 10 minutes, bleed the system slowly to ambient pressure.
6. Introduce gaseous fluorine slowly into the system until a pressure of about 100 psig is obtained, or to the maximum system working pressure, if less than 100 psig.
7. After the pressurized gaseous fluorine has been in the system for about 30 minutes, bleed the system slowly to ambient pressure.

After completion of the seven steps listed above, the system is considered passivated and ready to accept liquid chlorine trifluoride. Precautions must be taken to prevent the entry of moisture into the system. This can be accomplished by using dry pressurizing gas and by maintaining a positive pressure in the system at all times.

4.0 HAZARDS

4.1 Toxicity

Chlorine trifluoride is a very toxic propellant and inhalation of even dilute concentrations shall be avoided. Exposure of the skin to the propellant in either liquid or vapor form shall also be avoided. In 1960, a maximum allowable concentration (MAC) of chlorine trifluoride in air of 0.1 ppm was adopted by the American Conference of Governmental Industrial Hygienists. This MAC value is the average concentration over a normal work day to which most workers can be safely exposed day after day without adverse effects.

The odor threshold for chlorine trifluoride vapors is very low but has not been established reliably. However, it is known that the propellant vapor can be detected at sufficiently low concentrations and that personnel exposed to these concentrations do not experience toxic effects if they leave the hazard area immediately; thus, the propellant odor can serve as a warning of potential danger.

If an individual is exposed to strong vapor concentrations of chlorine trifluoride, he should hold his breath, if possible, until fresh air is reached; if unable to do this, breathing should be as shallow as possible. The exposed individual should be placed in the care of an authorized physician as soon as possible; in the meantime, first-aid treatment can be administered as directed by the local medical authority. For this purpose, it is recommended that at least one person permanently assigned to the storage area be properly trained in first-aid techniques. These techniques must be established only by the responsible medical authority.

4.2 Physiological Effects

Symptoms of chlorine trifluoride exposure are usually very obvious and will vary according to vapor concentration, duration of exposure, and the individual. Exposure to olfactory detectable concentrations for short periods of time usually results in watering of the eyes, dry throat, chest pain, and sometimes coughing.

Exposure to large concentrations of chlorine trifluoride will result in gasping for breath, swelling of eyes and eyelids, cloudiness of the cornea, lachrymation, severe salivation, coughing, breathing difficulty, and convulsions. In practice, fatal concentrations would be so irritating to the eyes and respiratory tract as to make the area intolerable. Concentrations of 50 ppm or more may be fatal in about 15 to 30 minutes.

Chlorine trifluoride is extremely corrosive and any contact of the propellant with living tissue will result in severe damage, especially to the eyes. If a person has suffered skin or eye exposure to liquid or vaporized chlorine trifluoride, the exposed areas should be washed immediately with copious quantities of water for a period of at least 15 minutes. The affected individual should be placed in the care of an authorized physician as soon as possible.

4.3 Fire and Explosive Hazards

Chlorine trifluoride is insensitive to mechanical shock and is nonflammable in air; however, the propellant readily ignites organic substances such as solvents and lubricants. The greatest hazard of the propellant lies in its extreme reactivity and the readiness to cause combustion of materials with which it comes in contact, and the violent explosions which may accompany the reaction. As a result, propellant spills and leaks must be avoided, and the storage and use areas must be maintained immaculately clean.

5.0 HAZARD REDUCTION

Spills of chlorine trifluoride present a hazard from both intoxication and fire as described in the HAZARDS section. For this reason, it is necessary to prevent propellant spills whenever possible and to control the spills and fires safely when they occur.

5.1 Spill Prevention

The prevention of chlorine trifluoride spills is one of the most important considerations for the safe handling of the propellant. Spills are the main cause of personnel intoxication and facility damage. Effective spill reduction is accomplished by the use of properly designed equipment and thoroughly trained personnel.

5.1.1 System Integrity

The integrity of the storage and transfer system cannot be overemphasized. The system should be reliable, operationally flexible, and easy to maintain. Some of the design criteria that shall be incorporated in the system are as follows:

1. Only materials of construction which are definitely known to be compatible with the oxidizer shall be employed
2. The number of mechanical joints shall be reduced to a minimum, thus reducing the probability of propellant leakage
3. The system shall be designed to withstand the maximum operating pressure safely
4. The transfer lines shall be free of liquid traps

5. An inert-gas system must be provided to purge the transfer lines without the necessity of dumping the residual propellant or disconnecting any system joints

6. The system components must be reliable, compatible with the oxidizer, and properly serviced

7. The chlorine trifluoride vents shall be ducted together and connected to a vapor scrubber or a high vent stack

8. Sufficient control equipment must be provided in order to isolate portions of the system during emergencies or components replacement

The continual observation of an operational system for possible malfunctions can prevent serious propellant spills. The leakage of chlorine trifluoride is not obvious because its vapors are nearly colorless. However, a sensitive halogen detector can be used effectively to check the system joints because a small propellant leak yields high local vapor concentrations. Thus, if a small leak is detected, corrective action must be taken as soon as possible.

5.1.2 Trained Personnel

Properly trained personnel are required to handle chlorine trifluoride safely. Operating personnel should be thoroughly familiar with the following:

1. The properties of chlorine trifluoride

2. Operation of the transfer and storage system

3. Toxicity and physiological effects of the propellant

4. Operation and use of safety equipment

5. Fire and spill prevention techniques

6. Fire and spill control measures

7. Disposal and decontamination techniques

8. Local operating procedures and regulations

No person should be allowed to handle chlorine trifluoride unless he is thoroughly familiar with the above-listed items and he is also confident that the propellant can be handled safely with the equipment and facilities available to him.

5.2 Fire Prevention

The most effective means of preventing chlorine trifluoride fires is by preventing propellant spills and leaks; therefore, a considerable amount of effort should be concerted toward this goal. However, since the probability of experiencing a propellant leak or spill cannot be completely eliminated, the storage area must be maintained immaculately clean at all times and free of litter, rubbish, solvents, and other combustibles.

6.0 HAZARD CONTROL

In case of chlorine trifluoride spillage or fire, all personnel shall report to their predesignated safe areas or emergency operating posts. Immediate evaluation of the hazardous situation is necessary so that appropriate control action be initiated in the shortest possible time.

The time period between the inception of the hazardous situation and initiation of control action shall be reduced to a minimum. This can be accomplished through proper planning, training, and organization. The following items shall be considered in the administration of the storage and handling areas:

1. Safe areas and evacuation routes shall be pre-established
2. Only authorized personnel shall be allowed to enter these areas
3. At least two operating personnel shall wear protective clothing and equipment during propellant handling operations
4. Periodic drills shall be performed to ensure personnel proficiency during emergency operations

6.1 Spill Control

A propellant spill can be most efficiently controlled by performing the following steps chronologically:

1. Stop the propellant handling operations
2. Isolate the propellant tanks from the transfer lines by closing the necessary valves

3. Locate the source of spill

4. Isolate the affected components by closing the necessary valves

5. Dispose of the spilled propellant

The performance of the first four steps listed above should be automatic and can be performed in a very short time.

The disposition of the spilled propellant should not be too difficult, especially when propellant handling is performed only during satisfactory weather conditions and the first four steps listed above are quickly executed. The disposition method depends greatly on the quantity of propellant spilled, prevailing weather conditions, location of storage and/or handling area, etc. Therefore, the discussion presented herein will be limited to general criteria which will be applicable to most situations.

Chlorine trifluoride spills can be best controlled by allowing the propellant to vaporize. In this case, a large amount of propellant would initially vaporize (flash) resulting in the cooling of the residual propellant. Shortly thereafter, steady-state vaporization is experienced. It should be noted that a large amount of toxic vapors are generated over a long period of time, thus dictating the need of performing propellant handling operations during satisfactory weather conditions.

As an alternate method, the spilled propellant can be deluged with large quantities of water. In this case, however, the water reacts with the propellant forming hydrofluoric acid and generating large quantities of energy. This method is not recommended because of the following reasons:

1. A large quantity of hydrofluoric acid is formed which might be difficult to dispose of subsequently

2. The water might react with the propellant explosively

3. The energy liberated by the reaction would cause rapid vaporization of the residual propellant which can result in prohibitive vapor concentrations

After the spill is controlled, the entire area must be thoroughly decontaminated. Decontamination techniques are presented in another section of this document.

6.2 Fire Control

Chlorine trifluoride fires result in the generation of intense heat for a short period of time. Since the propellant fires cannot be extinguished efficiently, if at all, the fire control techniques are based on preventing facility damage. This is accomplished by deluging the area with copious quantities of water. Fog-type water injection nozzles are particularly suitable for this application.

The reduction or prevention of facility damage resulting from fires can be attained when the following items are considered in the design, fabrication, and operation of the storage area:

1. The facility must be designed as fireproof as possible

2. The area must be maintained clean, uncluttered, and free of combustible materials

3. The facility must be equipped with a properly designed water-deluge system, preferably of the fog type

4. The storage tanks must be diked in order to limit the potential propellant burning zone

It is emphasized again that the most important way of preventing facility damage is by preventing chlorine trifluoride leaks and spills.

7.0 SAFETY EQUIPMENT

The toxic and extreme reactive properties of chlorine trifluoride dictate the need of adequate safety equipment for the protection of the storage and handling areas, and operating personnel. Although it is recognized that the type of personal safety equipment depends upon several factors such as facility design and personnel assigned tasks, it is suggested that only one type of safety equipment be specified and enforced. This criteria reduces the misunderstanding among operating personnel as to what degree of protection is required for a specific job assignment and prevents "short-cut" methods which are difficult to spot check and which can result in serious accidents.

7.1 Facility Safety Equipment

Facility safety equipment shall consist of personnel emergency showers, eye wash fountains, a water deluge system preferably of the fog type, fire blankets, portable fire extinguishers, fire hoses, a chlorine trifluoride detector, and first-aid kits. This equipment shall be strategically located and easily accessible.

All operating personnel shall be thoroughly familiar with the location and operation of each piece of safety equipment. The operation of the equipment must be verified periodically.

7.2 Personal Safety Equipment

All personnel in the chlorine trifluoride handling and storage areas shall wear flameproof clothing at all times. In addition, all personnel performing propellant transfer operations shall wear fully protective personal equipment. If the operations are performed remotely, it is still recommended that at least two operating personnel be fully protected to facilitate proper spill and fire control.

The following personal protective equipment, or its equivalent, is recommended:

1. Flameproof coveralls
2. Suit, Gra-Lite
3. Gloves, Charco HySol
4. Hood, Gra-Lite
5. Boots, Gra-Lite
6. Respirator, self-contained or air-line connected

This equipment must be maintained clean and in good operating order. A contaminated suit, for example, can become a definite safety hazard.

NOTE: The above equipment recommendation is based only on its commercial availability. However, this equipment is incompatible with the oxidizer, heavy, uncomfortable, and bulky. A need for more appropriate personal safety equipment definitely exists.

8.0 DECONTAMINATION

Decontamination involves the removal of chlorine trifluoride, hydrofluoric acid, and other fluorides following a propellant spill or fire, and the deactivation of facility equipment previously exposed to chlorine trifluoride. Decontamination procedures are employed to protect both personnel and equipment. Personnel performing these operations shall wear the fully protective equipment described in the SAFETY EQUIPMENT section.

8.1 Area Decontamination

The contaminants remaining from a chlorine trifluoride spill or fire are hydrofluoric acid, solid fluorides, and in some cases, liquid chlorine trifluoride. Since these fluorine compounds are corrosive and toxic, they must be removed. This can be accomplished by washing the area with copious quantities of water. The drained water in turn becomes contaminated and must be disposed of as stipulated by local regulations.

8.2 Equipment Decontamination

The removal of a component from a chlorine trifluoride system must be preceded by a thorough gaseous nitrogen purge to remove any residual propellant. If the removed component is to be reused without service or modification, no further decontamination operations are required; otherwise, the removed component is purged thoroughly with water and dried by purging it with gaseous nitrogen.

All components removed from a chlorine trifluoride system must be labeled clearly, describing the extent of decontamination and operational status.

9.0 TRANSPORATION

Shipment of chlorine trifluoride by common carrier is authorized by the Interstate Commerce Commission which classifies chlorine trifluoride as a "Corrosive Liquid," and as such, is subject to those regulations established for this group. In transit, cylinders must be affixed with an ICC-approved WHITE label. Highway vehicles carrying 2500 lb or more of chlorine trifluoride must be identified with "Dangerous" placards in letters at least 3 inches high on a contrasting background.

Chlorine trifluoride is conveniently available in 5-, 10-, 25-, and 100- to 200-lb-net cylinders. These cylinders are equipped with one shutoff valve. One-ton cylinders are also available and are equipped with two shutoff valves. The ICC specifications applicable to the shipping cylinders are as follows:

ICC 3A150	seamless steel
ICC 3AA150	seamless steel
ICC 3B240	seamless steel
ICC 3E180	seamless steel
ICC 4B240	welded and brazed steel
ICC 4BA240	welded and brazed steel

Shipment of chlorine trifluoride in carboys, tank cars, and tank trucks is not authorized.

10.0 STORAGE

Chlorine trifluoride may be stored safely for several years provided the storage system is kept reasonably free of excessive heat and moisture. Instances of chlorine trifluoride storage at ambient temperatures for periods of 10 to 11 years have been reported. The shipping containers in which the propellant is received are satisfactory for storage. However, frequent inspection of the valves and fittings is mandatory because of the corrosive nature of the propellant.

Storage of tonnage quantities of chlorine trifluoride can be effected in large storage tanks fabricated of compatible stainless steels, aluminum alloys, or Monel. For this application, the Monel tanks are most suitable.

The U. S. Air Force General Safety Procedures for Chemical Guided Missiles Propellants (T.O. 11C-1-6 dated 1956) does not include chlorine trifluoride in its propellant classification list. However, since both liquid nitrogen tetroxide and liquid fluoride are assigned the same classification, it is assumed that chlorine trifluoride belongs to the same group (Group 11, Class A, Poisonous Substance). This Technical Order is the present authority for establishing quantity-distance relationships for storing most of today's liquid rocket propellants. The quantity-distance values presently established for Group 11 propellants "Poison Hazard" are presented in Table 1.

Chlorine trifluoride cylinders must be located and positioned so that they are secured against rolling or being inadvertently tipped over. This can be accomplished by placing them in cradles. Cylinders up to 200-lb-capacity can be placed vertically and secured with chains; individual cylinder support is preferred.

TABLE 1

APPLICABLE QUANTITY-DISTANCE VALUES FOR GROUP 11 PROPELLANTS

(T.O. 11C-1-6, dated 1956)

Quantity of Propellant		Barricaded Distance in Feet To:				Unbarricaded Distance in Feet to:
Pounds over	Pounds not over	Inhabited or Service Building**	Passenger Railroad*	Public Highway*	Magazine or Another Group XI Storage (Z)	Magazine or Another Group XI Storage
10	1,000	5,000	305	155	100	200
1,000	5,000	5,000	450	225	150	300
5,000	10,000	10,000	520	260	200	300
10,000	50,000	10,000	840	420	200	400
50,000	100,000	10,000	1,090	545	400	500
100,000	250,000	10,000	1,295	650	500	800

* American Table of Distances (double these if storages are unbarricaded).

** For distances from storages (except ready storages) to operating building, use (Z) inhabited building distances.

11.0 HANDLING

Chlorine trifluoride handling operations, as described herein, includes the unloading of the shipping cylinders, loading of storage tanks, venting, and disposal operations. Personnel performing handling operations must wear the fully protective equipment described in the SAFETY EQUIPMENT section. Another activity closely associated with the above functions is the handling of the shipping cylinders. The shipping cylinders can be handled safely without the need of fully protective equipment.

11.1 Handling of Shipping Cylinders

The shipping cylinders must be handled gently and carefully. They must not be dropped, tumbled, rolled, dragged, or allowed to bump into other cylinders, walls, or projections. The cylinder shutoff valve(s) cap must be installed at all times during cylinder handling operations.

The cylinders may be transferred by means of truck, crane, forklift, or any other piece of equipment capable of handling them safely. While in transfer, the cylinders must be firmly secured.

Storage of the shipping cylinders should be restricted to those areas specifically designated for this purpose. The condition of each cylinder (full, empty, contaminated, etc.) must be marked clearly.

11.2 Transfer of Chlorine Trifluoride

Chlorine trifluoride can be discharged from its storage container either by its own vapor pressure, by pressurizing the container with dry nitrogen or helium, or by connecting a transfer pump in the container discharge line. Gas pressurization unloading is used almost exclusively at the present time since this technique is extremely reliable. The transfer pump technique is practical when large quantities of the propellant must be transferred in a relatively short period of time. Vapor pressure unloading is impractical and sometimes impossible.

The propellant transfer system must be chemically compatible with the propellant, leakproof, and in excellent operating order.

In preparing for a transfer operation, all personnel not directly concerned with the operation shall evacuate the hazard area. Appropriate warning lights and signs shall be displayed to keep out unauthorized personnel.

Personnel performing the transfer operation shall wear the fully protective equipment described in the SAFETY EQUIPMENT section. If the operations are performed remotely, at least two operating personnel should be fully dressed to facilitate proper spill and fire control. Sufficient safety equipment should be available for all personnel allowed to remain in the hazard area.

Supervisory and emergency support personnel shall be notified prior to executing any hazardous operation in the storage area.

The propellant transfer procedures are dependent upon numerous factors such as transfer system design, type of propellant container, training of operating personnel, prevailing weather conditions, etc. Establishing proper operating procedures for each specific situation in a single document is practically impossible. Therefore, the procedures presented below are general in nature. The transfer system schematics presented are not finalized designs; they are provided only to facilitate the explanation of typical procedures.

11.2.1 Transfer From Single-Opening Containers

The 5-, 10-, 25-, and 100- to 200-lb-net chlorine trifluoride cylinders are single-opening containers in which only one opening is available to perform the propellant transfer operation. The opening is sealed by a compatible shutoff valve, which in turn is protected by a gas-tight cap. These cylinders are not equipped with dip tubes and as such should be avoided since they present additional propellant transfer complexity.

The propellant can be transferred from these cylinders by prepressurizing the cylinders with dry nitrogen prior to the transfer, or by allowing the propellant to flow under its own vapor pressure. The vapor transfer technique is inefficient, and in some cases, impossible unless the collecting tank is cooled. Therefore, the prepressurization technique is recommended for most operations involving single-opening cylinders.

The prepressurization of the cylinder with dry nitrogen can be accomplished as follows:

1. Remove the protective cap from the cylinder shutoff valve.
2. Connect a regulated dry nitrogen supply line to the shutoff valve of the cylinder.

3. Regulate the pressure supply to the desired value. The regulated pressure level determines the rate of propellant transfer. A value ranging from 50 psig to 100 psig is usually adequate. The pressure should never exceed 10 psig less than the cylinder design pressure.

4. Open the pressure supply shutoff valve.

5. Slowly open the cylinder shutoff valve.

6. When the cylinder pressure equalizes the regulated source pressure, close the supply and cylinder shutoff valves.

NOTE: Two basic techniques can be used to determine when pressure equalization is attained. First, the noise generated by the gas flow through the pressurizing line ceases. Second, the regulated pressure gage registers the regulated pressure value prior to gas flow.

7. Bleed the trapped gas between the two shutoff valves by opening the transfer-line bleed valve.

8. Disconnect the pressurizing line from the cylinder shutoff valve.

9. Cap the opened connections to prevent contamination.

The transfer of chlorine trifluoride from the pressurized cylinder to the storage tank (Fig. 3) can be performed as follows:

1. Turn the cylinder upside down and place it in a transfer cradle. The cylinder must be properly secured and care must be exercised to prevent damage to the cylinder shutoff valve.

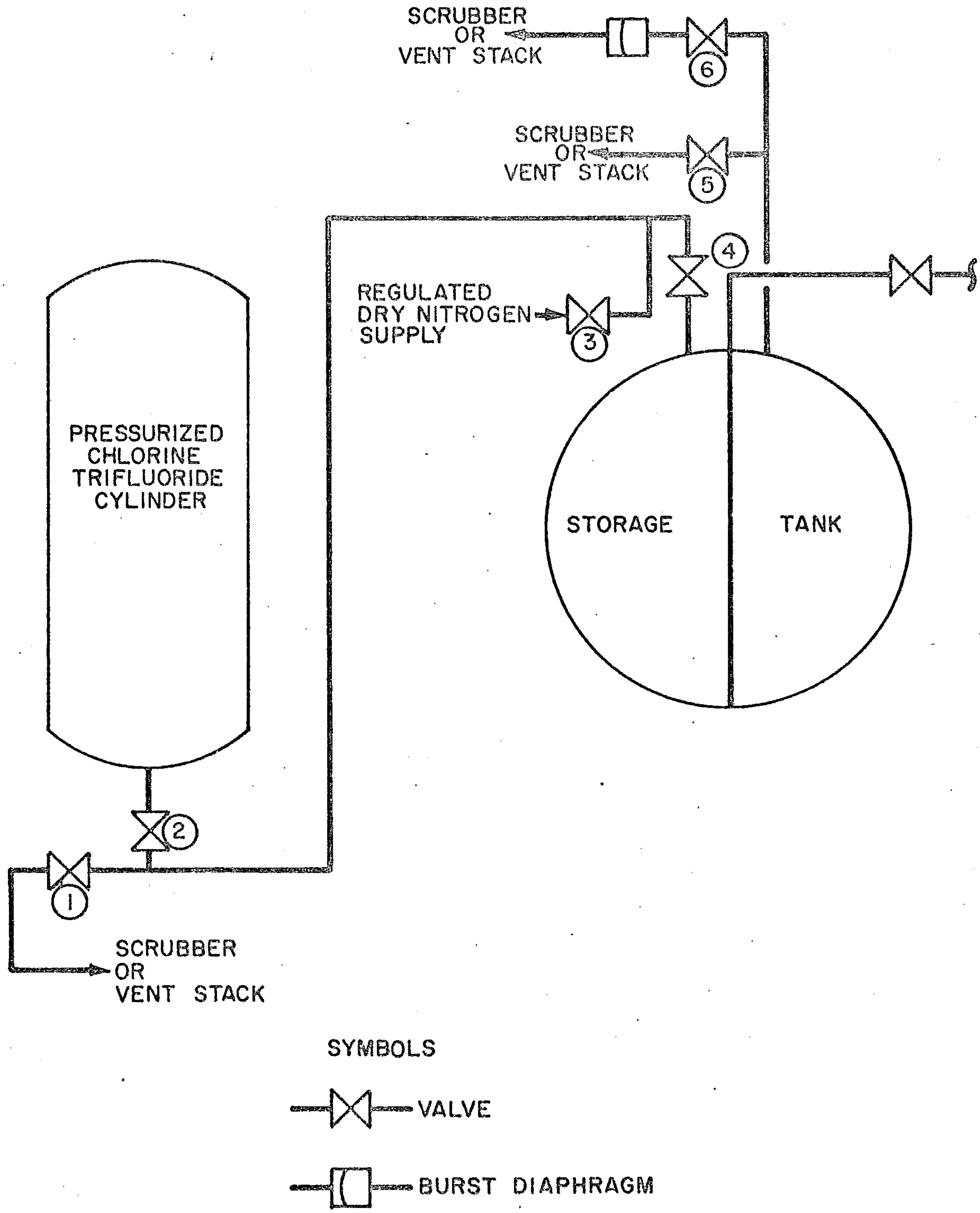

Figure 3. Transfer of Chlorine Trifluoride from Pressurized Single-Opening Cylinders

2. Connect the cylinder shutoff valve to the propellant transfer system as shown in Fig. 3.

3. Close all system valves except valve No. 6 which must be maintained open. The purpose of valve No. 6 is to prevent the continuous escape of chlorine trifluoride in case of burst-diaphragm failure and to facilitate the removal of the burst diaphragm whenever required.

4. Purge the propellant transfer line to remove trapped water vapor. This is accomplished by opening valves No. 3 and 1. When the purge operation is completed (about 3 minutes), close valves No. 3 and 1.

5. Open valve No. 2 slowly and check for leaks. If a leak develops, close the valve, open valve No. 1, and take the action necessary to stop the leak. (Valve No. 1 must be closed and valve No. 2 opened before proceeding with Step 6.)

6. Open valve No. 4.

7. After valves No. 2 and 4 have been opened, the propellant flows from the cylinder into the storage tank until the liquid in the cylinder is depleted or the pressure in the two containers equalizes. If the pressure in the two containers equalizes, close valve No. 4, and open valve No. 5 momentarily to depressurize the storage tank. The flow can be resumed by reopening valve No. 4.

8. When the desired quantity or all of the available propellant has been transferred, close valves No. 2 and 4.

NOTE: There are several devices which can be used to detect the completion of the propellant transfer operation.

Combinations of two or more devices are usually required to provide the desired flexibility of the transfer system. Some of these devices are:

a. A flowmeter installed in the transfer line

b. A scale or other weight-sensing device attached to the container being unloaded

c. A calibrated level indicator mounted on the storage tank

9. Purge the transfer line thoroughly by opening valves No. 1 and 3. When the purging operation is completed (about 3 to 5 minutes), close valves No. 3 and 1.

10. Depressurize the storage tank by opening valve No. 5 momentarily.

11. Disconnect the cylinder shutoff valve from the transfer system and cap the opened components.

12. Turn the cylinder to the upright position, mark it adequately, and dispose of it according to operating procedures.

13. Notify all personnel concerned that the transfer operation is completed and the area is clear.

11.2.2 Transfer From Double-Opening Containers

The one-ton chlorine trifluoride shipping cylinder is a double-opening container and is equipped with two shutoff valves. During transfer operations, one opening can be used to pressurize or vent the container and the other opening to discharge the propellant.

The propellant can be discharged from the shipping container either by pressurizing the container with dry nitrogen, or by connecting a transfer pump in the product discharge line. Although the pressurization unloading technique has been used nearly exclusively in the past, procedures for both transfer techniques are discussed in detail below.

11.2.2.1 Pressurization Unloading

As mentioned previously, the transfer of liquid chlorine trifluoride from shipping containers can be performed reliably by pressurizing the containers with dry nitrogen. The following procedure is basically applicable to the transfer of the propellant from double-opening containers into a storage tank using gas pressurization:

1. Place the one-ton cylinder in the horizontal position with the shutoff valves aligned with the vertical centerline axis.

2. Remove the protective cap from the cylinder and connect the cylinder shutoff valves to the transfer system as shown in Fig. 4. When the cylinder is positioned per Step 1, above, the upper shutoff valve is connected to the regulated pressure supply and the lower shutoff valve is connected to the transfer line.

3. Ensure that all system valves are closed except valve No. 9 which must be maintained open. The purpose of valve No. 9 is to prevent the continuous escape of chlorine trifluoride in case of burst-diaphragm failure and to facilitate the replacement of the burst diaphragm whenever required.

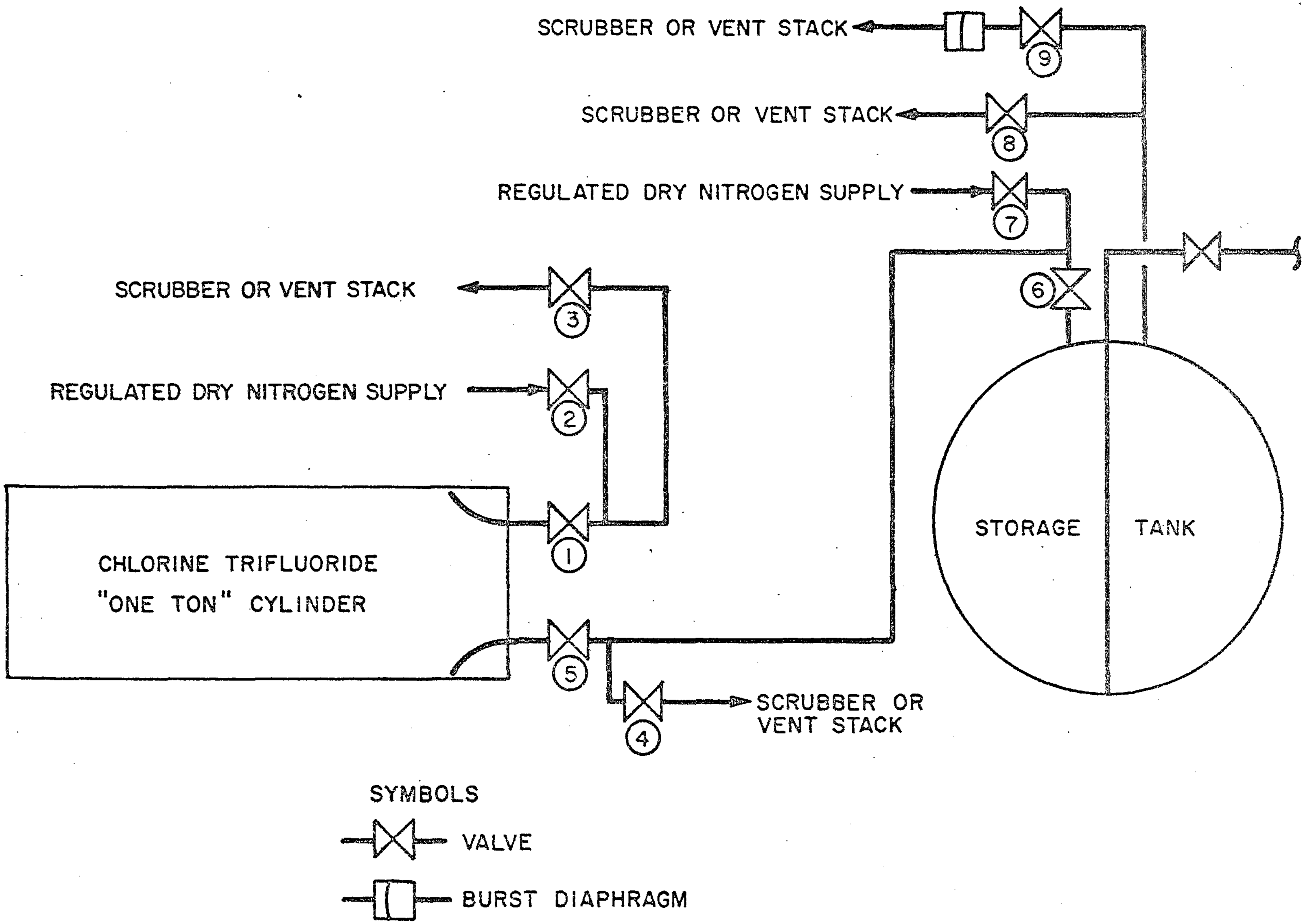

Figure 4. The Transfer of Chlorine Trifluoride from Double-Opening Containers Using Gas Pressurization

4. Set the dry nitrogen regulator to the desired pressure level. This pressure level determines the propellant discharge flow. A value ranging from 50 to 100 psig is usually adequate. The pressure should never exceed 10 psig less than the container design pressure.

5. Purge the propellant lines to remove trapped water vapor. This can be accomplished as follows:

   a. Open valves No. 4 and 7, and purge for about 3 minutes.

   b. Close valve No. 7 and 4.

   c. Open valves No. 3 and 2, and purge for about 3 minutes.

   d. Close valves No. 2 and 3.

6. Open valve No. 5 and check for leaks. If a leak develops, close the valve, open valve No. 4, and take the necessary action to stop the leak. (Valve No. 4 must be closed and valve No. 5 opened before proceeding with Step 7.)

7. Open valve No. 6.

8. Establish the propellant flow by pressurizing the chlorine trifluoride cylinder. This is accomplished by opening valves No. 1 and 2. A propellant flow is experienced until the liquid in the shipping container is depleted or the pressure in the two containers equalizes. If the pressure in the two containers equalizes, close valves No. 2 and 6, and open valve No. 8 momentarily. The flow can be resumed by reopening valves No. 6 and 2.

9. When the desired quantity or all of the available propellant has been transferred, close valves No. 2 and 5.

NOTE: There are several devices which can be used to detect the completion of the propellant transfer operation. Combinations of two or more devices are usually required to provide the desired transfer system flexibility. Some of these devices are:

a. A flowmeter installed in the transfer line

b. A scale or other weight-sensing device attached to the container being unloaded

c. A calibrated level indicator mounted on the storage container

10. Depressurize the shipping container by opening valve No.3. When the container is depressurized, close valves No. 1 and 3.

11. Close valve No. 6 and purge the transfer line by opening valves No. 4 and 7. When the transfer line is properly purged (usually takes from 3 to 5 minutes at a pressure level of about 50 psig), close valves No. 7 and 4.

12. Depressurize the storage container by opening valve No. 8 for a short period of time.

13. Disconnect the shipping cylinder shutoff valves from the transfer system and cap the opened components.

14. Mark and dispose of the shipping container according to operating procedures.

15. Notify all personnel concerned that the transfer operation is completed and the area clear.

11.2.2.2 Transfer Pump Unloading

As mentioned previously, pump unloading is an alternate method of transferring chlorine trifluoride from the shipping cylinders into storage tanks. This technique is highly applicable when large quantities of the propellant must be transferred in a relatively short period of time.

The following procedure is basically applicable to the transfer of chlorine trifluoride from the "one-ton" shipping cylinder into a storage tank by means of a transfer pump:

1. Place the shipping cylinder in a horizontal position with the shutoff valves aligned with the vertical centerline axis.
2. Remove the cylinder protective cap and connect the cylinder shutoff valves to the transfer system as shown in Fig. 5. With the cylinder positioned per Step 1, above, the upper shutoff valve is connected to the vapor-return line and the lower shutoff valve is connected to the transfer line.
3. Ensure that all system valves are closed, except valves No. 11 and 7, which must be opened. The object of valve No. 11 is to prevent the continuous escape of chlorine trifluoride in case of burst-diaphragm failure and to facilitate the removal of the burst diaphragm whenever required. Valve No. 7 prevents pump damage due to overpressures resulting from the possible vaporization and expansion of trapped propellant in the pump.
4. Set the dry nitrogen regulator to the desired pressure level. A value ranging from 20 to 30 psig should be adequate.

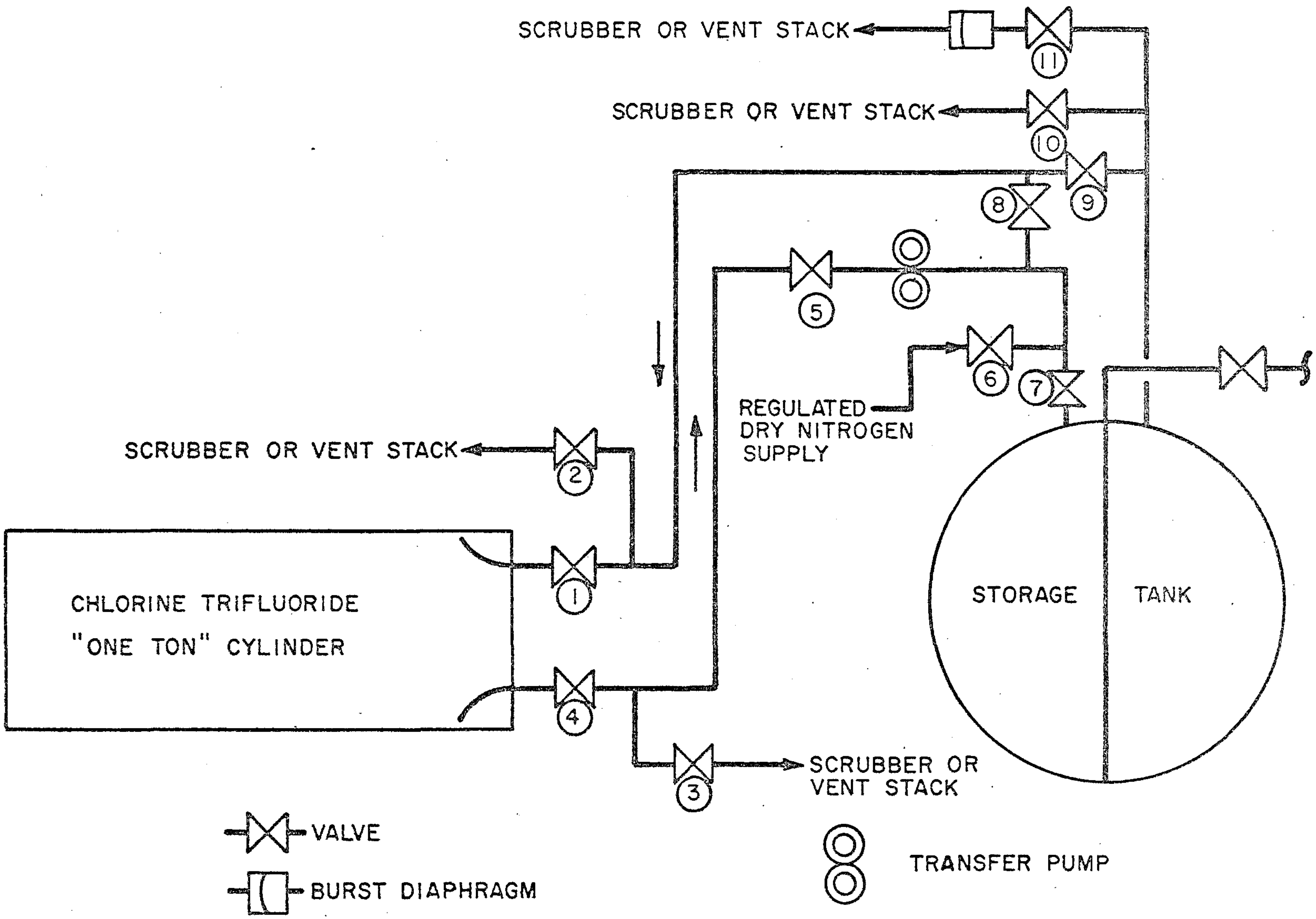

Figure 5. The Transfer of Chlorine Trifluoride from Double-Opening Containers Using a Pump

5. Purge the propellant lines to remove trapped water vapor. This can be accomplished as follows:

   a. Close valve No. 7.

   b. Open valves No. 3, 5, and 6, and purge for about 3 minutes.

   c. Close valves No. 6, 5, and 3.

   d. Open valves No. 2, 8, and 6, and purge for about 3 minutes.

   e. Close valves No. 6, 8, and 2.

   f. Open valve No. 7.

6. Pressurize the chlorine trifluoride cylinder to permit the proper priming of the transfer pump, if required. This is accomplished as follows:

   a. Close valve No. 7.

   b. Open valves No. 1, 8, and 6.

   c. When the gas flow stops, close valves No. 6, 8, and 1.

   d. Open valve No. 7.

7. Open valve No. 4 and check for leaks. If a leak develops, close the valve, open valve No. 3, and take the necessary action to stop the leak. (Valve No. 3 must be closed and valve No. 4 opened before proceeding with Step 8.)

8. Open valve No. 5 and check for leaks. If a leak develops, close valves No. 5 and 7, open valves No. 8 and 2, and take the action necessary to stop the leak. (Valves No. 2 and 8 must be closed and valves No. 5 and 7 opened before proceeding with Step 9.)

9. Open valve No. 1.

10. Start the transfer pump and open valve No. 9. A closed-loop pump transfer operation is thus established.

11. When the desired quantity or all of the available propellant have been transferred, stop the transfer pump and close valve No. 7.

NOTE: There are several devices which can be used to detect the completion of the propellant transfer operation. Combinations of two or more devices are usually required to provide the desired transfer system flexibility. Some of these devices are:

a. A flowmeter installed in the transfer line

b. A scale or other weight-sensing device attached to the container being unloaded

c. A calibrated level indicator mounted on the storage tank

12. Close valve No. 9 and depressurize the shipping cylinder by opening valve No. 2. When the container is depressurized, close valve No. 2.

13. Depressurize the storage tank by opening valve No. 10 for a short period of time.

14. Purge the propellant transfer line as follows:

a. Open valve No. 6 for about 2 to 3 minutes or until the gas flow stops.

b. Close valve No. 6.

c. Close valve No. 4.

d. Open valves No. 3 and 6, and purge the line for about 3 to 5 minutes.

e. Close valves No. 6, 5, and 3.

15. Depressurize the shipping cylinder by opening valve No.2. When the cylinder is depressurized, close valves No. 1 and 2.

16. Purge the vapor-return line as follows:

a. Open valves No. 2, 8, and 6, and purge for about 3 to 5 minutes.

b. Close valves No. 6, 8, and 2.

17. Open valve No. 7.

18. Disconnect the shipping cylinder shutoff valves from the transfer system and cap the opened components.

19. Mark and dispose of the shipping cylinder according to operating procedures.

20. Notify all personnel concerned that the transfer operation is completed and the area clear.

11.3 Venting

The depressurization of chlorine trifluoride containers occurs quite frequently. In this operation, a considerable amount of vapor is released which must be handled safely. Two basic methods can be used for handling the propellant vapor. These methods are:

1. The transfer system vent lines are connected to a scrubber system which removes the propellant vapor from the

vented gases. Many types of scrubbers and solutions for absorbing the propellant vapors can be used.

2. The transfer system vent lines are connected to a vent stack which discharges the vented gases at least 60 feet above the highest working point in the area. A low-pressure nitrogen purge can be installed in the stack to further dilute the vented propellant vapor before being discharged into the atmosphere.

Chlorine trifluoride containers should be vented only under controlled conditions. These conditions are dependent upon area location, weather conditions, etc.

11.4 Disposal

Disposal involves the controlled release of chlorine trifluoride from a shipping or storage container into a system capable of disposing of the propellant safely. Military regulations, at the present time, limit the disposal of Group 11 propellants to a maximum of 1000 lb for any one disposal operation. As mentioned previously, chlorine trifluoride is not included as a propellant in the regulations; however, it is assumed that it belongs to the Group 11 propellants.

The following items are essential for the proper selection and safe operation of the chlorine trifluoride disposal area:

1. The disposal area shall be isolated in accordance with the quantity-distance table presented in the STORAGE section.

2. The disposal area shall be clear of trees, weeds, brush, and other combustibles.

3. The area must be provided with adequate facility safety equipment (See SAFETY EQUIPMENT section).

4. One person shall never be allowed to work in the disposal area alone.

5. The personal safety equipment which was described in the SAFETY EQUIPMENT section must be worn during disposal operations.

6. All personnel not participating in the disposal operation shall evacuate the area.

7. Disposal operations shall be performed only under controlled conditions. These conditions are dependent upon area location, weather conditions, etc.

The following methods can be employed to dispose of chlorine trifluoride:

1. The slow release of the propellant through a high vent stack. The outlet should be at least 60 feet above the disposal area. A dry nitrogen purge may be installed in the stack to dilute the propellant vapor before being exhausted into the atmosphere.

2. The controlled burning of chlorine trifluoride by the use of a fuel such as alcohol or kerosene. This is accomplished by placing a quantity of fuel approximately equal to the quantity of chlorine trifluoride to be disposed of into a burn basin. The oxidizer is then fed slowly into the burn basin and allowed to react with the fuel. For this operation, the chlorine trifluoride supply tank should be located at least 50 feet from the burn basin.

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