

AEROSPACE MATERIAL SPECIFICATION



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Trichloroethane 1, 1, 1, (Methyl Chloroform) Inhibited, Vapor Degreasing

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1. SCOPE:

- 1.1 This specification covers the requirements of inhibited 1,1,1 trichloroethane for vapor degreasing, (see 6.1).

2. APPLICABLE DOCUMENTS:

The following publications, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

2.1 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

QQ-A-250/4C	Aluminum Alloy 2024, Plate and Sheet
QQ-M-44	Magnesium Alloy, Plate and Sheet (AZ-31B)
PPP-D-729	Drums, Metal, 55-Gallon (For Shipment of Non-corrosive Material)
PPP-P-704	Pails, Shipping, Steel (1 through 12 Gallon)

MIL-S-7952	Steel, Sheet and Strip, Uncoated Carbon (1020 and 1025)
MIL-T-9046	Titanium and Titanium Alloy, Sheet, Strip and Plate

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129	Marking for Shipment and Storage

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

D-1078-63	Distillation Range of Volatile Organic Liquids
D-1364-64	Water in Volatile Solvents (Fischer Reagent Titration Method)
D-2108-64	Test for Color of Halogenated Organic Solvents and Their Admixtures
D-2109-64	Test for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures
D-2111-64	Test for Specific Gravity of Halogenated Organic Solvents and Their Admixtures

3. REQUIREMENTS:

3.1 Material:

The material shall consist of inhibited 1,1,1 trichloroethane. The inhibitors and impurities shall consist of materials such that the toxicity of the solvent mixture shall not exceed that of the uninhibited 1,1,1 trichloroethane as demonstrated by acute and chronic toxicity studies. The material shall be and remain homogeneous, and its utility shall not be adversely affected by storage up to 6 months in the original shipping containers under any climatic condition.

3.2 Chemical composition:

The material shall contain not less than 93.0 percent 1,1,1 trichloroethane by weight. The material shall not contain any individual halogenated constituent, other than 1,1,1 trichloroethane, in excess of 0.5 percent by volume. In addition, the sum total of all halogenated constituents, other than 1,1,1 trichloroethane, shall not exceed 1.0 percent by volume. The composition of 1,1,1 trichloroethane, shall be determined by a gas chromatography procedure of sufficient sensitivity and accuracy to detect minute quantities of contaminants as described in 4.4.5.

3.3 Physical and chemical requirements:

The material shall meet the requirements of table I when tested in accordance with the indicated method.

TABLE I. Properties of 1,1,1 Trichloroethane

Property	Requirement	Test Method Paragraph
Nonvolatile Residue, % wt, max	0.001	4.4.1
Specific Gravity 25°/25°C	1.300-1.327	4.4.2
APHA Color, max	15	4.4.3
Appearance	Free from suspended matter and sediment	4.4.4
1,1,1 Trichloroethane content (% by wt) min	93.0	4.4.5
Water Content, max	100 ppm	4.4.6
Acidity (as HCl)		
As Received (% by wt) max	0.001	4.4.7.1
After 48-hour Accelerated Oxidation, max	0.020	4.4.7.2
Free Halogen	None	4.4.8
Metals Corrosion	No visual corrosion	4.4.9
Distillation Range		4.4.10
Initial Boiling Point, min	70°C (158°F)	
Dry Point, max	88°C (190°F)	
Acid Acceptance		
As % wt as NaOH, min	0.10	4.4.11
Residue Soluble in CCl ₄ (% by wt as Iso-Octane) max	0.0005	4.4.12

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for inspection:

The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Sampling:

4.2.1 Lot: A lot shall consist of the 1,1,1 trichloroethane produced by one manufacturer, with no change in process or materials, provided the operation is continuous. In the event the process is a batch process, each batch shall constitute a lot. (See 6.3).

4.2.2 Sampling for inspection of filled containers: A random sample of filled containers shall be selected from each lot offered for inspection in accordance with Military Standard MIL-STD-105 at inspection level I, and acceptable quality level (AQL) = 2.5 percent defective to verify compliance with this specification regarding fill, closure, marking and other requirements not involving tests.

4.2.3 Sampling for tests: Two containers shall be selected at random from each inspection lot. If more than one lot is represented in the shipment, each lot represented shall be treated as a separate shipment for sampling purposes. The contents of each container having been selected at random for sampling, shall be thoroughly mixed by rolling and inverting immediately prior to sampling. Each sample shall be tested to determine compliance with this specification.

4.3 Quality assurance inspection:

4.3.1 Inspection of filled containers: Each sample filled container selected in accordance with 4.2.2 shall be examined for defects of the container and the closure, for evidence of leakage, and for unsatisfactory marking; each sample filled container shall also be weighed to determine the amount of contents. Any container in the sample, having one or more defects, or under required fill, shall be rejected and if the number of defective containers in any sample exceeds the acceptance number for the applicable sampling plan of Military Standard MIL-STD-105, the lot represented by the sample shall be rejected.

4.3.2 Lot acceptance tests: The samples selected in accordance with 4.2.3 shall be subjected separately to the tests specified in 4.4. If either sample fails one or more of these tests, the lot shall be rejected.

4.4 Test procedures:

- 4.4.1 Nonvolatile residue: This determination shall be made in accordance with ASTM Method A, D-2109-64. A stream of warm (40° - 50°C) dry, oil-free nitrogen or compressed air may be used to accelerate evaporation. The results shall be reported on a weight basis.
- 4.4.2 Specific gravity: The specific gravity shall be determined in accordance with ASTM D-2111-64.
- 4.4.3 APHA color: Test in accordance with ASTM Method D-2108-84.
- 4.4.4 Appearance: A portion of the well shaken sample shall be placed in a test tube and allowed to stand stoppered and undisturbed until bubbles have entirely disappeared. Examination shall be made by transmitted light for uniformity and freedom from particles.
- 4.4.5 1,1,1 Trichloroethane content: The content of 1,1,1 trichloroethane, inhibited, shall be assayed in accordance with the following gas chromatographic procedure or one of equal or better sensitivity and accuracy.
- 4.4.5.1 Apparatus: A gas chromatograph having the following minimum capabilities is recommended:
- Temperature control system capable of maintaining the chromatographic column at prescribed temperature $\pm 0.5^{\circ}\text{C}$.
- Heated sample inlet.
- A differential katharometer (hot wire), thermistortype thermal conductivity detector or hydrogen flame detector.
- 4.4.5.2 Materials: The following materials are recommended:
- Chromatographic column - Annealed copper or stainless steel tubing, 12 feet long by 1/8-inch outside diameter (0.055-inch inside diameter).
- Column packing - Acid washed calcined-pink firebrick (Chromosorb P), 30 to 50 U.S. Standard mesh, coated with 15 percent by weight of UNCON LB 1800X liquid phase. The completed column should be conditioned prior to initial use at 125°C, with helium gas flowing through it for 24 hours at a rate of 15 ml per minute.
- Carrier gas - Dry helium of 99.95 mole percent purity.
- Standard - Uninhibited 1,1,1 trichloroethane of at least 98.0% purity.

4.4.5.3 Analysis conditions: The following analysis conditions are recommended:

Column temperature - 60°C isothermal.

Detector temperature - 225°C.

Sample inlet temperature - 200°C.

Sample size - 5 microliters (to be injected by means of a precision 10 ul syringe via a heated inlet).

Carrier gas flow - 15 ml per minute.

Detector sensitivity - Sufficient to easily meet the 0.5 percent detection requirement set forth in 3.2.

Employing the operating conditions recommended, all constituents of methyl chloroform solvents should elute from the column in approximately 30 minutes.

4.4.5.4 Determination of 1,1,1 Trichloroethane content: Quantitative measurement of concentration of a constituent in a sample depends upon a determination of the peak area or peak height of the constituent's chromatographic peak or area. Peak areas may be measured by any accepted method (planimeter, ball and disc integrator, electronic digital integrator, etc.) or may be approximated by multiplying the height of the peak by the width of the peak at the half-height. To obtain the percent concentration of the 1,1,1 trichloroethane in a sample, a direct comparison is made of its peak area with the peak area of 1,1,1 trichloroethane in the standard. Both sample and standard must be analyzed under identical operating conditions.

4.4.6 Water content: The water content shall be determined in accordance with ASTM Method D-1364-64.

4.4.7 Acidity (as HC1):

4.4.7.1 The acidity of the "as received" material shall be determined by standard titration with .02 normal sodium hydroxide in anhydrous methanol using bromothymol-blue as an indicator.

4.4.7.2 Acidity after accelerated oxidation: The general arrangement of the apparatus used is shown in figure 1. Two-hundred ml of the sample shall be placed in the boiling flask. One strip of steel 1/2 inch by 2 inches by 1/16 inch shall be suspending by means of an uncoated copper wire so it remains above the liquid level, and another strip 1/4 inch by 3/4 inch by 1/16 inch shall be placed in the bottom of the flask. The strips shall conform to S.A.E. steels within the range of 1010 to 1040 inclusive. The oxygen delivery tube shall extend to within 1/4 inch of the bottom of the flask. The water shall be started through the condenser. The oxygen flow shall be regulated to 10-12 bubbles per minute by adjusting the height of the water level above oxygen outlet in the pressure control cylinder. The heating of the solution shall be started and the solvent refluxed for 48 hours. The solvent shall be cooled to ambient temperature, 25 ml shall be withdrawn and the acidity determined by the method described in 4.4.7.1.

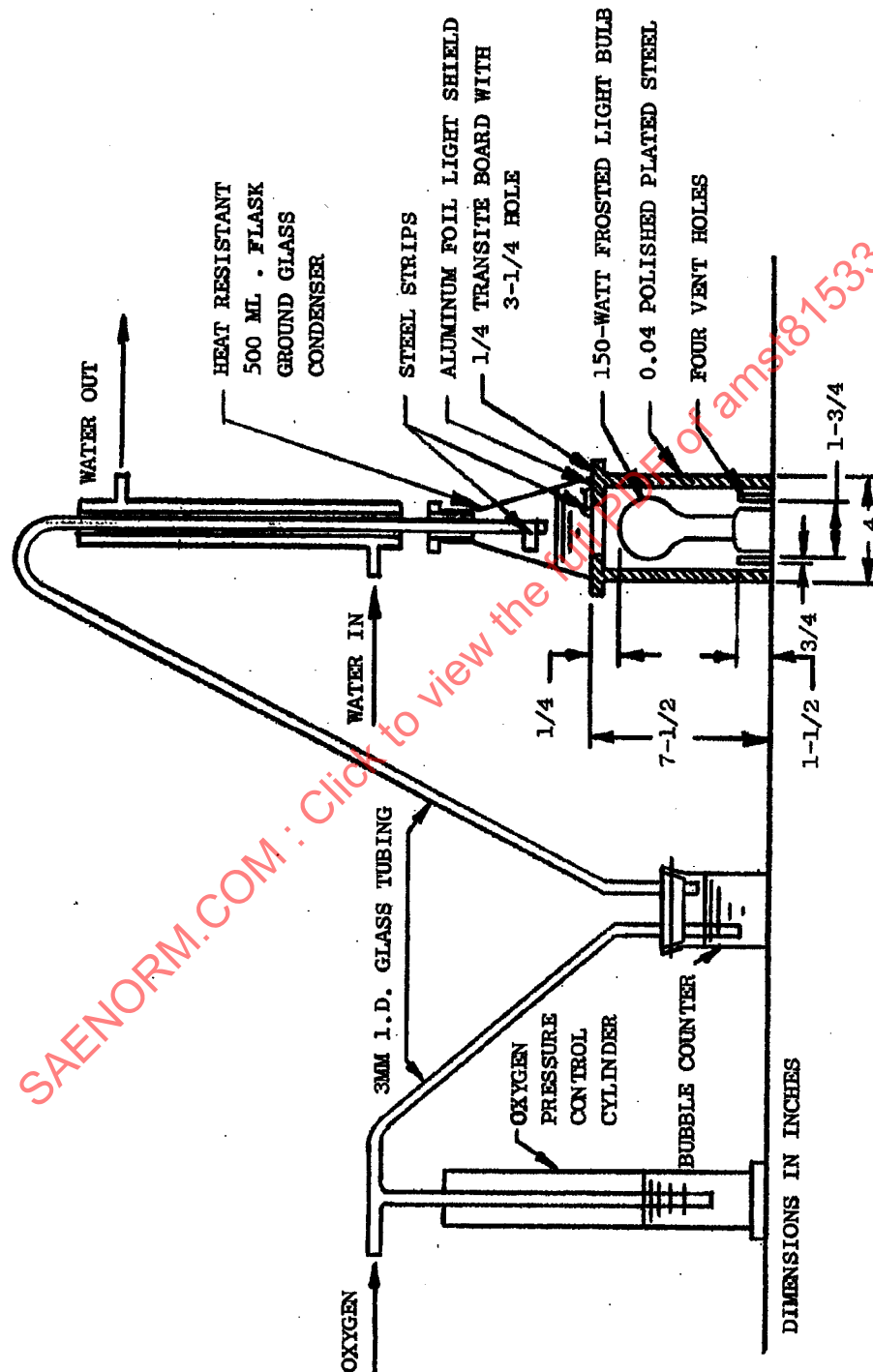


FIGURE 1. Accelerated Oxidation Test Apparatus

- 4.4.8 Free halogen: Transfer 10 ml of sample to a clean test tube. Add 10 ml of distilled water, 5 drops of 10 percent potassium iodine solution (ACS Reagent) and one ml of starch solution (ACS Reagent). Shake for one minute. A blue color in the aqueous layer indicates the presence of free halogen.
- 4.4.9 Metals corrosion: Prepare 1/2 inch by 5 inch panels of each of the following metals: Aluminum alloy (QQ-A-250/4C temper T-4), Steel (MIL-S-7952) Magnesium (QQ-M-44) and Titanium (MIL-T-9046). Polish panels with No. 1 emery cloth until the entire panel is bright, shiny and free from any oxide film or tarnish. Do not handle the panels with bare hands after polishing. Wash with soap or detergent and water and rinse with distilled water. Wash with acetone and allow to air-dry. Place each panel in separate 500 ml Erlenmeyer flask which has a ground glass neck, add sufficient 1,1,1 trichloroethane to form a layer of solvent one inch deep. Attach a reflux condenser and heat over a light shielded 150 watt light bulb and reflux for 24 hours. Allow to cool to ambient temperature, rinse with sample but do not wipe, and examine for corrosion.
- 4.4.10 Distillation range: Test in accordance with ASTM Method D-1078-63.
- 4.4.11 Acid acceptance: (as percent NaOH by weight)
- 4.4.11.1 Reagents:
- Sodium hydroxide - 0.01 Normal, standardized.
- Hydrochlorination reagent (hydrochloric acid) - Dissolve 1.5 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) in 400 ml of absolute methanol in a 500 ml glass stoppered bottle and mix thoroughly.
- Indicator solution - Dissolve one gram of phenolphthalein in 50 ml of ethanol and dilute to 100 ml with distilled water.
- 4.4.11.2 Procedure: Pipet 25 ml of the hydrochlorination reagent into a 250 ml ground glass stoppered Erlenmeyer flask. Add 15 ml of the solvent under test, stopper and swirl the flask to mix. Let stand for a minimum of 5 minutes to allow completion of the reaction. While the reaction is taking place, pipet 25 ml of the hydrochlorination reagent to another flask, add 25 ml of distilled water and one ml of indicator solution. Titrate with 0.01N NaOH solution to a faint pink that remains for 15 seconds. Record the ml of NaOH used as the Blank (A). At the end of the reaction period, remove the stopper from the flask containing the sample and add 25 ml of distilled water and one ml of indicator solution. Titrate with 0.01N NaOH to same end point as specified for the blank. Record the ml of NaOH used, (B).