Designation: D 5632 – 95

An American National Standard

Standard Specification for Halon 1301, Bromotrifluoromethane (CF_3Br)[1]

This standard is issued under the fixed designation D 5632; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers requirements for Halon 1301 as a fire-fighting medium.

1.2 This specification does not address the fire-fighting equipment or hardware that employs Halon 1301 or the conditions of employing such equipment (for example, handhelds, fixed installations, etc.).

1.3 This specification does not address the storage or transportation of Halon 1301. Storage, handling, and transportation issues may be addressed in future ASTM specifications.

1.4 The following safety hazards caveat pertains only to the test methods portion, Section 6, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 4081 Specification for Drycleaning Grade Perchloroethylene[2]

2.2 *ISO Standards:*

ISO 3363 Fluorochlorinated Hydrocarbons for Industrial Use—Determination of Acidity—Titrimetric Method[3]

ISO 3427 Gaseous Halogenated Hydrocarbons (Liquified Gases)—Taking of a Sample[3]

ISO 5789 Fluorinated Hydrocarbons for Industrial Use—Determination of Nonvolatile Residue[3]

2.3 *U.S. Military Standards:*

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes[4]

MIL-STD-129 Marking for Shipment and Storage[4]

MIL-STD-1188 Commercial Packaging of Supplies and Equipment[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *halon*—a halogenated hydrocarbon used as a fireextinguishing medium.

3.1.1.1 *Discussion*—The halon terminology system provides a convenient means to reference halogenated hydrocarbon fire extinguishants. Halogenated hydrocarbons are acyclic saturated hydrocarbons in which one or more of the hydrogen atoms have been replaced by atoms from the halogen series (that is, fluorine, chlorine, bromine, and iodine). By definition, the first digit of the halon numbering system represents the number of carbon atoms in the compound molecule; the second digit, the number of fluorine atoms; the third digit, the number of chlorine atoms; the fourth digit, the number of bromine atoms; and the fifth digit, the number of iodine atoms. Trailing zeros are not expressed. Unaccounted for valence requirements are assumed to be hydrogen atoms.

$$\text{number of hydrogen atoms} = [((\text{number of carbon atoms} \times 2) + 2) - (\text{sum of halogen atoms})] \quad (1)$$

For example,

$$\text{bromotrifluoromethane} - CF_3Br - \text{Halon 1301} \quad (2)$$

4. Material Requirements

4.1 *Type I*:

4.1.1 The nitrogen (N_2) partial pressure shall be such that the safe working pressure of the receiving vessel is not exceeded. To prevent excessive pressure, the fill density of the halon within a container should not exceed that needed to achieve complete filling of the container at the maximum envisaged storage temperature. For example, the U.S. DOT 4BA500 cylinder partial pressure shall not exceed 12.1 bar at 21°C (161 psig at 70°F) for a 1121-kg/m^3 (70-lb/ft^3) fill density (yielding a total pressure of 25.8 bar at 21°C (360 psig at 70°F)). For this example, the safe working pressure of the 4BA500 cylinder is not exceeded for temperatures below 54°C (130°F).

4.1.2 Halon 1301 shall conform to the requirements prescribed in Table 1 when tested by the appropriate test method(s) listed in Section 6.

4.1.3 When a material analysis is required, by agreement between the purchaser and the supplier, the total pressure in the Halon 1301 container, partial pressure of the N_2, the fill density of the halon within the container, and the maximum safe storage temperature shall be part of the material analysis

[1] This specification is under the jurisdiction of ASTM Committee D-26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.09 on Recycled Halon 1301.

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[2] *Annual Book of ASTM Standards*, Vol 15.05.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Available from Standardization Documents Order Desk, Bld. 4 Section D, 700 Robbins Av., Philadelphia, PA 19111-5904, Attn: NPODS.

TABLE 1 Requirements

Property	Requirement
Halon 1301 purity, %, mol/mol, min	99.6 (exclusive of any N_2 present)
Acidity, ppm by mass, max	3.0
Water content, ppm by mass, max	10
Nonvolatile residue, % by weight, max	0.01
Halogen ion	passes test
suspended matter or sediment	none visible

(certification). The pressure shall be reported in bar (preferred) or pound-force per square inch gage. The fill density shall be reported in kilograms per cubic metre at 21°C (preferred) or pounds per cubic foot at 70°F. The maximum safe storage temperature of the Halon 1301 container shall be reported in degrees Celsius (preferred) or in degrees Fahrenheit and shall conform to applicable regulations for the Halon 1301 container design and use.

4.2 *Type II*—Halon 1301 shall conform to the requirements of Type I, as listed in 4.1, and shall contain no more than 1.5 % by volume fixed gases in vapor phase, expressed as air when tested by the appropriate test method(s) listed in Section 6.

4.3 By agreement between the purchaser and the supplier, analysis may be required and limits established for elements or compounds not specified in Table 1.

NOTE 1—**Warning:** Exposure to concentrations of Halon 1301 in excess of 10 % (halon volume/air volume) during periods of elevated adrenaline could produce cardiac arrhythmia in some personnel.

4.4 Unless otherwise specified, Type I is assumed.

5. Sampling

5.1 Samples of halons, taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427 or MIL-STD-105, Inspection Level S-4. The sampling bottle shall be capable of safely resisting the vapor pressure of the sample at the highest temperature that could be encountered.

5.2 The Halon 1301 selected in accordance with 5.1 shall be tested for quality conformance in accordance with Section 6. The presence of one or more defects shall be cause for rejection.

6. Test Methods

6.1 *Purity*:

6.1.1 Determine the purity by gas-liquid chromatography in accordance with the technique described in 6.1.2-6.1.5 or another acceptable laboratory technique providing equivalent results.

6.1.2 *Apparatus*—The following special apparatus is required to determine the percent of Halon 1301:

6.1.2.1 *Gas Chromatograph*, equipped with a 1-mV recorder and thermal conductance detector.

6.1.2.2 *Column*, 3 m by ⅛-in. (3.175-mm) outside diameter thin-wall stainless steel tubing, packed with 80 to 100 mesh Porapak Q[5] or equivalent.

6.1.2.3 *Gas Sampling Valve*, 5-mL volume or a volume sufficient to achieve proper separation in the specified column.

6.1.3 *Reagents*—The carrier gas shall be a commercial grade of helium. The column packing shall consist of a standard solution, for example, 20 % (weight/weight) practical hexadecane, on 80 to 100-mesh Porapak Q[5] or equivalent.

6.1.4 *Procedure*:

6.1.4.1 Install the column and adjust the temperature of the column oven to 80°C, injection port to 160°C, and detector block to 100 to 110°C. The temperature should be programmed to rise 10 to 15°C/min, to a maximum of 180°C.

6.1.4.2 Adjust the helium flow to 20 mL/min.

6.1.4.3 Adjust the detector voltage to 8 V or to the mid-range of the thermal conductivity detector (TCD) instrument being used and allow the instrument to stabilize.

6.1.4.4 Take the sample from the liquid phase (inverted cylinder). Flush the sample loop and sample valve for approximately 30 s before sampling.

6.1.4.5 Rotate the gas sampling valve to transfer the sample into the chromatographic system and note the time.

6.1.4.6 Close the sample cylinder valve.

6.1.4.7 Allow the sample to elute, for approximately 18 min, attenuating as necessary to make the peak heights a convenient size. Under proper instrument settings, the Halon 1301 should elute after approximately 5 min.

6.1.5 *Calculation*:

6.1.5.1 Calculate percent Halon 1301 as follows:

$$\%\ CF_3Br = \frac{A(CF_3Br)}{A_s} \times 100 \quad (3)$$

where:

$A\ (CF_3Br)$ = area of monobromotrifluoromethane peak multiplied by recorder range setting, and

A_s = sum of the relative peak heights excluding the nitrogen (air) peak height.

6.1.5.2 Percent nitrogen may be calculated as follows:

$$\%\ N_2 = \frac{A_n}{A_s + A_n} \times 100 \quad (4)$$

where:

A_n = area of nitrogen peak multiplied by the recorder range setting, and

$A_s + A_n$ = sum of the relative peak heights including the nitrogen peak.

It is useful to calculate percent nitrogen in order to judge a safe fill density. Percent Halon 1301 below that specified in Table 1 shall constitute failure of this test method.

6.2 *Acidity*—Vaporize a large sample in the presence of distilled water. Determine the acidity of the solution by the appropriate method specified in ISO 3363, titration in accordance with 6.2.1.2-6.2.2.3, a pH indicator, or another acceptable laboratory technique providing equivalent results.

6.2.1 *Sodium Hydroxide Titration*:

6.2.1.1 *Reagents*:

(1) Sodium Hydroxide, 0.01 *N* solution, standardized against reagent grade potassium acid phthalate.

(2) Methyl Red Indicator, 0.1 % solution.

[5] Available from Alltech, 2051 Waukegan Road, Deerfield, IL 60015. Phone 1-800-255-8325.

6.2.1.2 *Procedure*—Place 10 mL of a crushed ice-distilled water slurry in a 250-mL stoppered Erlenmeyer flask and add 50 g of monobromotrifluoromethane to the slurry. Place the stopper in the flask loosely, and swirl the flask gently from time to time until the ice is melted completely. Add 1 drop of methyl red indicator, and if a reddish color remains, titrate to a yellow endpoint with 0.01 *N* sodium hydroxide solution. Run a crushed ice-distilled water blank (with no Halon 1301) along with the sample.

6.2.1.3 *Calculation*—Calculate parts per million acid halides, as HBr, as follows:

$$\text{acid halides, ppm} = \frac{(A - B) \times N\ \text{NaOH} \times 0.0809 \times 10^6}{\text{weight of sample, g}} \quad (5)$$

where:

A = NaOH for sample, mL, and
B = NaOH for blank, mL.

Acid halides in excess of the amount specified in Table 1 shall constitute failure of this test.

6.2.2 *Acidity by Universal Indicator*:

6.2.2.1 *Apparatus*:

(1) Fritted Glass Sparger, of coarse porosity, contained in a 100-mL glass scrubbing bottle provided with inlet and outlet tubes.

(2) Neoprene Connecting Tubing.

(3) Wet Test Meter, 0.1 ft^3 revolutions.

(4) Needle Valve Control, No. 55-660, Matheson Co.,[6] or equivalent.

6.2.2.2 *Reagent Universal Indicator*, [7] with color chart, or equivalent.

6.2.2.3 *Procedure*—Prepare neutralized distilled water by adding 0.4 mL of Universal Indicator solution to 100 mL of distilled water, and titrate with 0.01 *N* sodium hydroxide until the water shows a pH of 7.0 when compared to the Universal Color Chart. Add 50 mL of the neutralized water to the glass scrubbing bottle fitted with the glass gas sparger. Attach a needle valve control to the sample cylinder, and connect the cylinder, inverted, to an empty safety trap. Connect the safety trap outlet to the scrubbing bottle inlet. Connect the scrubbing bottle outlet to the inlet of the wet test meter. Open the needle valve slowly and pass 20 L of sample through the scrubber at a flow rate of approximately 500 mL/min. Turn off the needle valve and disconnect the sample cylinder from the scrubbing bottle. Transfer 10 to 12 mL of water solution to a clean test tube. Add 0.3 mL of Universal Indicator solution and swirl. Read the pH of the solution by comparison with the Universal Color Chart. Report the pH reading. No observable change in pH indicates an acidity of less than 3.0 ppm.

6.3 *Water Content*—Test Halon 1301 for water content. The analysis may be conducted by the phosphorus pentoxide method, infrared absorption, electrolytic moisture analysis, piezoelectric analyzer, or another acceptable laboratory technique. The accuracy of the results and the standard method shall be by orthodox Karl Fischer method. Water content greater than specified in Table 1 shall constitute failure of this test.

6.4 *Qualitative Test for Halogen Ion*—Test a sample for the presence of halogen ions in accordance with 6.4.1-6.4.3 or by another acceptable laboratory technique providing equivalent results. Generally, a sample treated with an alcoholic solution of silver nitrate shall exhibit no turbidity or precipitation of silver halide.

6.4.1 *Apparatus*:

6.4.1.1 *Fritted Glass Gas Sparger*, of coarse or A porosity, contained in a 100-mL glass scrubbing bottle provided with inlet and outlet tubes.

6.4.1.2 *Neoprene Tubing*.

6.4.1.3 *Wet Test Meter*, 0 to 1 ft^3/revolutions.

6.4.1.4 *Needle Valve Control*, No. 55-660, Matheson Co.,[6] or equivalent.

6.4.2 *Reagents*:

6.4.2.1 *Methyl Alcohol*, absolute.

6.4.2.2 *Silver Nitrate*, saturated solution in methyl alcohol.

6.4.3 *Procedure*—Add 10 mL of methyl alcohol into the scrubber assembly and add 3 to 4 drops of saturated alcoholic silver nitrate solution. Attach a needle valve control to the sample cylinder. Connect the sample cylinder in the upright position to an empty safety trap with neoprene tubing. connect the outlet of the safety trap to the inlet of the scrubbing bottle assembly with neoprene tubing. Connect the outlet of the scrubbing bottle assembly to the inlet of the wet test meter. Open the needle valve slowly and pass approximately 2 L of sample through the scrubber at a flow rate of approximately 100 mL/min. Turn off the needle valve and disconnect the scrubber assembly from the sample cylinder and the wet test meter. Examine the contents of the scrubber visually for the presence of turbidity. Report the halide present if any turbidity develops. The appearance of any turbidity shall constitute failure of this test.

NOTE 2—The approximate ratio of methyl alcohol to monobromotrifluoromethane must be maintained. An excess of sample will cause the silver nitrate to precipitate, yielding a false turbidity reading as halide.

6.5 *Suspended Matter and Sediment*—Examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

6.6 *Nonvolatile Residue*—Determine the nonvolatile residue in accordance with the method specified in ISO 5789 or another accepted laboratory technique providing equivalent results.

6.7 *Fixed Gases in the Vapor Phase*—Test Halon 1301 for air in the vapor phase by the determination of gases not absorbable in perchloroethylene, by gas chromatography, or another accepted laboratory technique providing equivalent results.

6.7.1 *Perchloroethylene Absorption*—Halon 1301 may be tested for air in the vapor phase of the container by the determination of gases not absorbable in perchloroethylene using water as a sealant. The test may be conducted by the determination of gases not absorbable in perchloroethylene using mercury as a sealant or by the determination of gases remaining when the extinguishant is frozen. The determination

[6] Available from Matheson Co., 430-T Caredean Road, Horsham, PA 19044. Phone 215-674-0686.

[7] Available from Fisher Scientific Co., Mid-Atlantic Region, 585 A Drive, Pittsburgh, PA 15238. Phone 1-800-766-7000.

of gases not absorbable in perchloroethylene using water as a sealant shall be the standard.

6.7.1.1 *Apparatus*:

(1) Absorption Vessel, Lab Glass Co., No. LG-11119,[8] or equivalent.

(2) Leveling Bulb and Connecting Tubing.

(3) Perchloroethylene Reagent—Solvent quality in accordance with Specification D 4081.

(4) Silicone Solution—One part Organo Silane Ester A-1230 or Amino Silane A-1106,[9] or equivalent, in 100 parts of water.

6.7.1.2 *Procedure*—Wash the absorption vessel thoroughly and rinse in distilled water. Coat the inside with the silicone solution, let stand 5 min, and discard the solution. Rinse again with distilled water and dry at 212°F for 10 min. When cool, add 20 mL of distilled water and fill with perchloroethylene. Add sufficient water to the leveling bulb to give a water seal. Collect a 100-m^3 sample from the vapor space of the Halon 1301 cylinder directly into the absorption vessel. A slight positive pressure is needed to prevent entry of extraneous air. Tilt the vessel in such a way as to remove all the water lock that separates the perchloroethylene from the gas. Allow the perchloroethylene to flow into the upper chamber of the vessel and gently agitate to aid in the absorption of the gas. Return the vessel to an upright position and allow the water layer to flow back into the burette section. Adjust the leveling bulb so that the liquid layers are at the same level. Read percent nonabsorbable gas from the burette.

6.7.1.3 *Calculation*—Make a blank run with a sample from the liquid phase of the cylinder and subtract the result from the analysis of the vapor phase. The presence of air in the vapor phase in excess of 1.5 % by volume shall constitute failure of this test.

6.7.2 *Gas Chromatography*—Halon 1301 may be tested for the concentration of air in the vapor phase by gas chromatography. A concentration of air in excess of 1.5 % by volume shall constitute failure of this test.

7. Container, Packaging, and Package Marking

7.1 Containers used for shipping and storage of Halon 1301 conforming to this specification shall be marked in accordance with MIL-STD-129 or MIL-STD-1188, whichever is applicable, and with the following information as a minimum:

7.1.1 Supplier's name and address,

7.1.2 Halon 1301,

7.1.3 Statement that material conforms to Specification D 5632, and

7.1.4 For storage or transportation within the United States, a warning label shall be affixed to the container conforming with United States Environmental Protection Agency requirements, in accordance with Section 611 of the Clean Air Act, as amended.

8. Keywords

8.1 bromotrifluoromethane; CF_3Br; fire fighting; firefighting agent; fire protection; fire suppressant; fluorochlorinated hydrocarbon; Halon 1301

[8] Available from Lab Glass Co., P.O. Box 610, Dept. G, Vineland, NJ 08360. Phone: 1-800-220-2164.

[9] Available from OSI Specialties, Inc., P.O. Box 38002, South Charleston, WV 25303-3802.