



Designation: ~~D5632-01~~ Designation: D 5632 - 08

Standard Specification for Halon 1301, Bromotrifluoromethane (CF₃Br)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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the 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 are addressed in Practice D 5631.

1.4

1.4 The values stated in inch-pound units are to be regarded as standard. The values in parentheses are for information only.

1.5 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 4.3.

2. Referenced Documents

2.1 *ASTM Standards:*²

D 5631 Practice for Handling, Transportation, and Storage of Halon 1301, Bromotrifluoromethane (CF₃Br)~~BR~~

2.2 *ISO Standards:*³

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

ISO 3427 Gaseous Halogenated Hydrocarbons (Liquefied Gases)—Taking of a Sample

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

2.3 *U.S. Industry Standard:*⁴

ASQ Z1.4 Sampling Procedures and Tables for Inspection by Attributes (ANSI Approved, DoD Adopted)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *halon 1301*—The halogenated hydrocarbon compound Bromotrifluoromethane used as a fire extinguishing 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.

number of hydrogen atoms =

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

¹ This specification is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents, and is the direct responsibility of Subcommittee D26.09 on Fire Extinguishing Agents.

Current edition approved Oct. 10, 2001. Published December 2001. Originally published as D5632-94a. Last previous edition D5632-95.

Current edition approved Jan. 1, 2008. Published January 2008. Originally approved in 1994. Last previous edition approved in 2001 as D 5632 - 01.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* Vol 15.05, volume information, refer to the standard's Document Summary page on the ASTM website.

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

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 100 Robbins Ave., Philadelphia, PA 19111-5904, Attn: NPODS.

For example,
bromotrifluoromethane – CF₃Br – Halon 1301

(2)

4. Material Requirements

4.1 *Type I*—Mixtures of Halon 1301 and Nitrogen:

4.1.1 The nitrogen (N₂)

4.1.1 The nitrogen (N₂) 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 4BA 500 cylinder partial pressure shall not exceed ~~12.1 bar~~ 161 psi at ~~21°C~~ (161 psi) ~~70°F~~ (12.1 bar at 70°F) 21°C for a ~~1121 kg/m³~~ 70 lb/ft³ ~~(70 lb/ft³)~~ (1121 kg/m³) fill density (yielding a total pressure of ~~25.8 bar~~ 360 psig at ~~21°C~~ (360 psig) ~~70°F~~ (25.8 bar at 70°F) 21°C). For this example, the safe working pressure of the 4BA500 cylinder is not exceeded for temperatures below ~~54°C~~ (130°F) ~~130°F~~ (54°C).

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 N₂, the fill density of the halon within the container, and the maximum safe storage temperature shall be part of the material analysis (certification). The pressure shall be reported in ~~bar~~ (preferred) or ~~pound-force per square inch gage~~ (preferred) or ~~bar~~. The fill density shall be reported in ~~kilograms~~ pounds per cubic ~~metre~~ foot at ~~21°C~~ 70°F ~~70°F~~ (preferred) or ~~pounds~~ kilograms per cubic ~~foot~~ metre at ~~70°F~~ 21°C. The maximum safe storage temperature of the Halon 1301 container shall be reported in degrees ~~Celsius~~ Fahrenheit (preferred) or in degrees ~~Fahrenheit~~ Celsius 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. **Warning**—Exposure to concentrations of Halon 1301 in excess of 7.5 % (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 shall be taken from the liquid or vapor phase as appropriate. Samples of halons, taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427 or ASQ Z1.4, 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 during handling, storage, or transport.

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.

6.1.1.1 Prior to sample introduction, a standardization of the GC must be performed using a known bromotrifluoromethane purity standard. The standardization must be performed as described herein.

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 flame ionization detector (FID) and an electronic integrator or suitable computer data gathering and reduction system.

6.1.2.2 *Chromatographic Column*, 7.34 m (24 ft) by 1/8 in. (3.175 mm) outside diameter thin-wall stainless steel tubing packed with Carbowax B, 60 to 80 mesh, containing 1 % SP1000 (also known as Carbowax 20M). (Column is available, prepacked, from several chromatographic supply vendors.)

6.1.2.3 *Sample Introduction*—Take sample from liquid phase and flash into evacuated 125-cc gas dispersion tube equipped with side outlet tube and septum. Bring gas dispersion tube to 1 atmosphere pressure. Using standard gas tight syringe take 1/4 to 1/2 cc sample from gas dispersion tube and introduce into GC.

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

6.1.3 *Reagents*—The carrier gas shall be a chromatographic grade of helium. Column packing shall consist of 1 % SP1000 (Carbowax 20M) weight/weight on Carbopack B, 60 to 80 mesh.

6.1.4 *Procedure for Programmed Temperature Gas Chromatography (PTGC)*:

6.1.4.1 Install the column in the gas chromatograph and adjust the oven temperature to 40°C. Adjust the helium carrier gas flow rate to 30 cc per minute. Adjust injection port temperature to 200°C and the detector to 250°C. Inject the sample and hold the column oven at 40°C for six minutes, then program the temperature to rise 10°C per minute to a maximum of 180°C, then posthold for 10 min before recycling.

6.1.5 *Calculation*:

6.1.5.1 Calculate weight percent and convert to mole percent Halon 1301 as follows:

$$W_i = \frac{A_i \times RRF_i \times 100}{\sum [A_i \times RRF_i]} \quad (3)$$

where:

W_i = weight percent of component i

A_i = peak area of component i

RRF_i = Relative Response Factor for component i

$\sum [A_i \times RRF_i]$ = sum of all component peak areas times their respective relative response factors.

6.1.5.2 For ASTM specification, the results must be converted to and reported in mole percentages. Convert individual impurities from Wt % to Mole % as follows:

$$\text{Mole } \%_i = \frac{\text{Wt } \% \times 148.9}{M} \quad (4)$$

where:

$\text{Wt } \%_i$ = weight percent of component i

149.8 = Molecular weight of Halon 1301

M = Molecular weight of component i

6.2 *Methanol Impurity*—The methanol impurity may be determined using capillary column programmed temperature gas chromatography. The column is 105 meter DB-1701 \times 0.25 mm inside diameter. The presence of methanol may cause inaccurate indication of other contaminants such as R115 and R22, possibly causing artificial enhancement of the R115/R22 contaminant peaks when present.

6.3 *Acidity*—From the Halon 1301 liquid phase, 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 6.3.1.2-6.3.2.3, using a pH indicator or another acceptable laboratory technique providing equivalent results.

6.3.1 *Sodium Hydroxide Titration* :

6.3.1.1 *Reagents*:

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

(2) *Methyl Red Indicator*, 0.1 % solution.

6.3.1.2 *Procedure*—Measure (graduate) 50 mL of ice-cold distilled water (stored in a 34°F refrigerator) in a 250-ml stoppered Erlenmeyer flask. Sparge 50g of bromotrifluoromethane into the cold water. Swirl the flask gently during the addition or, alternatively, add a magnetic stirring bar and stir the solution during the addition. Following the sample addition, pipet 5.0 mL of the water solution into a small glass vial, cap, and save for the Halogen ion test, 6.5. To the remaining water in the flask, add two drops of methyl red indicator, swirl, and if a reddish color remains, titrate to a yellow end point with 0.01 normal sodium hydroxide (NaOH). Run a blank (no Halon 1301) by titrating 45 mL of the cold DI water.

6.3.1.3 *Calculation*— Calculate parts per million acid halide, as HBr, as follows:

$$\text{acid halides, ppm} = \frac{(A - B) \times N \times 80.9 \times 10^3}{\text{grams of sample} \times 0.9} \quad (5)$$

where:

A = mL of NaOH for sample,

B = mL of NaOH for blank,

N = normality of the NaOH titrant

NaOH = sodium hydroxide

80.9×10^3 = equivalent weight of HBr (hydrogen bromide)

10^3 = Factor to convert result to ppm

Acid halides in excess of the amount specified in Table 1 shall constitute test failure by this method. Should no acidity be detected (that is, no color change of the indicator is observed), report result as “ passes test.”

6.3.2 *Acidity by Universal Indicator* :

6.3.2.1 *Apparatus*:

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