



## Standard Specification for Halon 1301, Bromotrifluoromethane (CF<sub>3</sub>Br)<sup>1</sup>

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 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, hand-helds, 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 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:

D 5631 Practice for Handling, Transportation, and Storage of Halon 1301, Bromotrifluoromethane (CF<sub>3</sub>Br)<sup>2</sup>

#### 2.2 ISO Standards:<sup>3</sup>

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

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

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

#### 2.3 U.S. Industry Standard:<sup>4</sup>

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

<sup>1</sup> 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.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.05.

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

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

### 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 =

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

For example,

$$\text{bromotrifluoromethane} - \text{CF}_3\text{Br} - \text{Halon 1301} \quad (2)$$

### 4. Material Requirements

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

4.1.1 The nitrogen (N<sub>2</sub>) 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 at 21°C (161 psi at 70°F) for a 1121-kg/m<sup>3</sup> (70 lb/ft<sup>3</sup>) 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.

**TABLE 1 Requirements**

Property	Requirement
Halon 1301 purity, %, mol/mol, min	99.6 (exclusive of any N <sub>2</sub> 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

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<sub>2</sub>, 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. 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. **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.

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 Carbowax 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 × 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

$80.9 \times 10^3$  = 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.

(2) *Neoprene Connecting Tubing*.

(3) *Wet Test Meter*, 0.1 ft<sup>3</sup> revolutions.

(4) *Needle Valve Control*, No. 55-660, Matheson Co<sup>5</sup>, or equivalent.

6.3.2.2 *Reagent Universal Indicator*,<sup>6</sup> with color chart, or equivalent.

6.3.2.3 *Procedure*—Prepare neutralized distilled water by adding 0.4 ml of universal indicator solution to 100 ml of distilled water and titrate to a pH of 7.0 with 0.01 N sodium hydroxide (if acidic) or with 0.01N HCl (if alkali) solution until the water reads 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 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 litres (66 gm) 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. Also transfer 10 ml of the water solution to a small glass vial, cap, and save for the Halogen Ion test, 6.5. 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 ppm.

6.4 *Water Content*—Moisture determination shall be performed by Coulometric Karl Fischer titration or suitable alternative methodology. Water content greater than specified in Table 1 shall constitute failure of this test.

6.5 *Qualitative Test for Halogen Ion*—Test a sample for the presence of halogen ions in accordance with 6.5.1-6.5.4 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.

#### 6.5.1 Reagents:

6.5.2 *3M silver Nitrate solution*—Dissolve 5.1 g of anhydrous silver nitrate in DI water to give a total volume of 10.0 mL. Store in a brown glass bottle away from light.

6.5.3 *Nitric Acid*, 1:1 v/v in water:

6.5.4 *Procedure*—To the water scrubber solution saved during the acidity test (from 6.3.2.3), add 1 drop of 1:1 HNO<sub>3</sub>, swirl to mix. Then add 3 to 4 drops of 3M silver nitrate solution and swirl to mix. The appearance of any haze or turbidity 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 *Suspended Matter and Sediment*—During the Halon 1301 evaporation step of the nonvolatile residue test (see 6.6), examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

6.8 *Fixed or Nonabsorbable Gases (NAG) in the vapor phase*—Test Halon 1301 for air in the vapor phase using isothermal gas chromatography or another accepted laboratory technique providing equivalent results.

6.8.1 Prior to sample introduction, calibration of the GC using a 15 % air or nitrogen calibration standard in helium must be performed as described for determination of the NAG in a Halon 1301 sample.

6.8.1.1 Percent NAG may be calculated using isothermal gas chromatography as follows:

$$RF = N_1/A_1 \quad (6)$$

$$C_2 = A_2 \times RF$$

$$C_1 = \frac{C_2 \times P_2 \times 2.397}{T_2 (^\circ R)} \quad (7)$$

<sup>5</sup> Available from Matheson Co., 430-T Caredean Road, Horsham, PA 19044. Phone 215-674-0686.

<sup>6</sup> Available from Fisher Scientific Co., Mid-Atlantic Region, 585 A Drive, Pittsburgh, PA 15238. Phone 1-800-766-7000.

where:

- $RF$  = Response factor for NAG  
 $N_1$  = Volume % nitrogen in standard  
 $A_1$  = Peak area of nitrogen determination  
 $A_2$  = Peak area of NAG in sample  
 $C_1$  = NAG volume % @ 75°F  
 $C_2$  = NAG volume % @ sampling temperature  
 $P_2$  = Vapor pressure in psia of bromotrifluoromethane at the sampling temperature in degrees F  
 $T_2$  = Sampling temperature in degrees Rankine (°R)  
 2.397 = Pressure/temp ratio for CF<sub>3</sub>Br at 75°F

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

NOTE 1—The 1.5 % NAG limit is for Type II material only.

6.8.1.2 *Gas chromatograph*, equipped with a thermal conductivity detector (TCD) and an integrator, 1-mV recorder, or other output recording device.

6.8.1.3 *Chromatographic Column*, 2 m (6 ft) by 1/8 in. (3.175-mm) outside diameter thin-wall stainless steel tubing packed with Porapak Q, 80 to 100 mesh, or equivalent. (Column is available prepacked from several chromatographic supply vendors).

6.8.1.4 *Reagents*—The carrier gas shall be a chromatographic grade of helium. Column packing shall consist of Porapak Q, 80 to 100 mesh, or equivalent.

6.8.1.5 Install the column in the gas chromatograph and adjust the temperature of the column oven to 100°C (isothermal analysis), injection port to 100°C, and detector block to 125°C.

6.8.1.6 Adjust the carrier gas flow rate to 30 cc helium/min.

6.8.1.7 Use autoinjection method with a 1cc loop. The sample is taken from the gas phase of the container.

## 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 Code of Federal Regulations (CFR) Title 49, Part 172 Subpart D “Marking Requirements of Packaging for Transportation”. The proper shipping name for ASTM D 5632 Type II (pure) Halon 1301 is “Liquefied Gas, n.o.s.” UN 1009 (49 CFR 172.101). The proper shipping name for nitrogen superpressurized Halon 1301 is “Liquefied Gas, nonflammable charged with nitrogen”, UN1058 (49 CFR 172.101). In addition to DOT requirements, containers must be marked 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<sub>3</sub>Br; fire fighting; firefighting agent; fire protection; fire suppressant; fluorobrominated hydrocarbon; Halon 1301

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