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Standard Specification for Halon 1301, Bromotrifluoromethane (CF₃Br)¹

This standard is issued under the fixed designation D5632/D5632M; 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 U.S. 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 D5631.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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 4.5.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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.

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2. Referenced Documents

2.1 ASTM Standards:²

D2108 Test Method for Color of Halogenated Organic Solvents and Their Admixtures (Platinum-Cobalt Scale)

D5631 Practice for Handling, Transportation, and Storage of Halon 1301, Bromotrifluoromethane (CF₃Br)

2.2 ISO Standard:³

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

2.3 AHRI Standard:⁴

2008 Appendix C for Analytical Procedures for AHRI Standard 700-2014

2.4 U.S. Government Standard:⁵

CFR Title 49, Part 172 U.S. DOT Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements

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

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* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁴ Available from Air-Conditioning, Heating, and Refrigeration Institute (AHRI), 2111 Wilson Blvd, Suite 500, Arlington, VA 22201, <http://www.ahrinet.org>.

⁵ Available from U.S. Government Publishing Office (GPO), 732 N. Capitol Street, NW, Washington, DC 20401-0001, <http://www.gpo.gov>.

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.

$$\text{number of hydrogen atoms} = \quad (1)$$

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

For example, (2)

bromotrifluoromethane – CF₃Br – Halon 1301

3.1.2 *non-condensable gases*—non-condensable gases consist primarily of air (nitrogen, oxygen) accumulated in the vapor phase of a liquefied compressed gas where the solubility of air in the liquid phase is extremely low and air is not significant as a liquid phase contaminant. Non-condensable gases are in some instances termed *non-absorbable gases*.

4. Material Requirements

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

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 1301 within a container should not exceed that needed to achieve complete filling of the container at the maximum envisaged storage temperature. For example, for a U.S. DOT 4BA 500 cylinder, the nitrogen partial pressure shall not exceed 161 psi at 70 °F [12.1 bar at 21 °C] for a 70 lb/ft³ [1121 kg/m³] fill density, which yields a total pressure of 360 psig at 70 °F [25.8 bar at 21 °C]. For this example, the safe working

pressure of the 4BA500 cylinder is not exceeded for temperatures below 130 °F [54 °C]. (See Practice D5631.) (See Fig. 1.)

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 1301 within the container, and the maximum safe storage temperature shall be part of the material analysis (certification). The pressure shall be reported in pound-force per square inch gauge (preferred) or bar. The fill density shall be reported in pounds per cubic foot at 70 °F (preferred) or kilograms per cubic metre at 21 °C. The maximum safe storage temperature of the Halon 1301 container shall be reported in degrees Fahrenheit (preferred) or in degrees 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 non-condensable gases in the 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.

4.4 Unless otherwise specified, Type I is assumed.

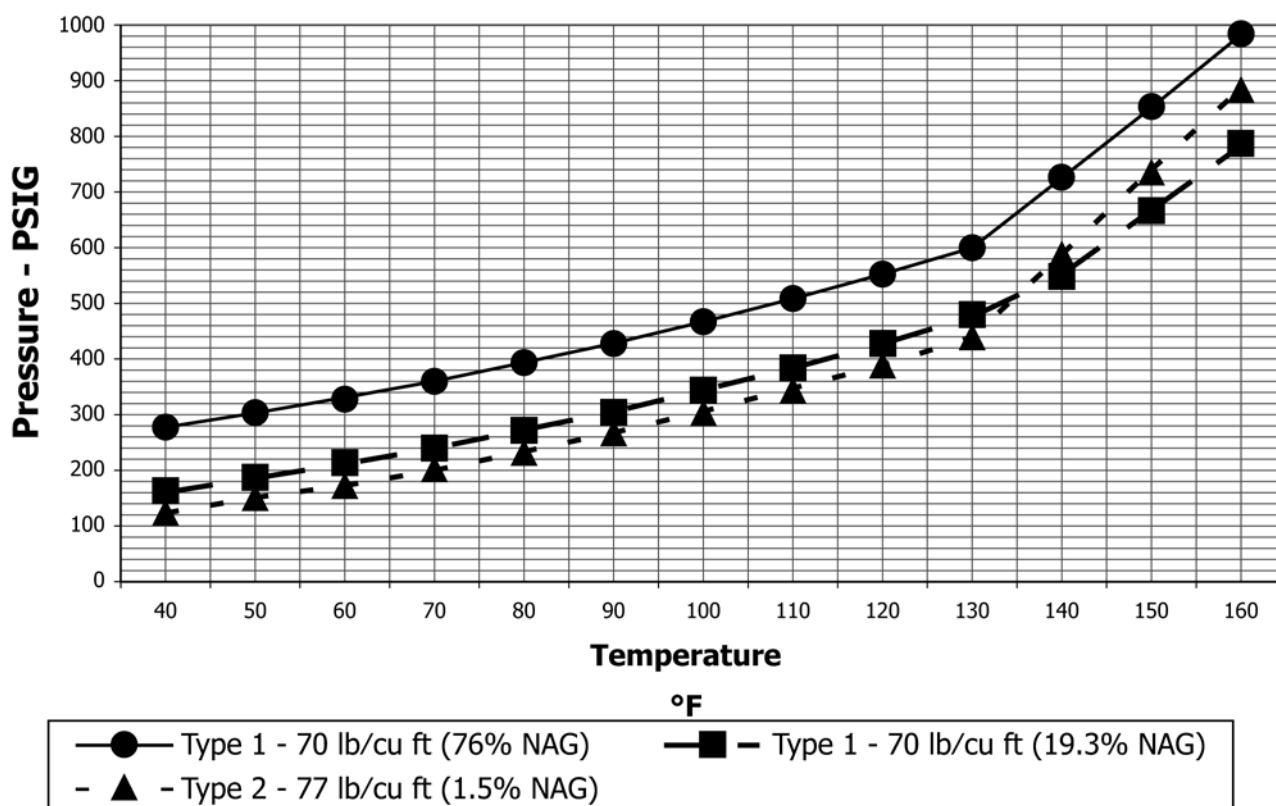


FIG. 1 Isometric Diagram for Bromotrifluoromethane



TABLE 1 Requirements

Property	Requirement
Halon 1301 purity, %, mol/mol, min	99.6 (exclusive of any N ₂ present)
Acidity, ppm by mass, as Hbr, 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
Color	Report value

4.5 Warning—Exposure to concentrations of Halon 1301 in excess of 7.5 % by volume in air during periods of elevated adrenaline could produce cardiac arrhythmia in some personnel.

5. Sampling

5.1 Samples of Halon 1301 shall be taken from the liquid or vapor phase as appropriate. Samples of Halon 1301, taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427 or the method specified in 2008 Appendix C for Analytical Procedures for AHRI Standard 700-2014, Part 7.

5.1.1 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. (See Practice D5631.)

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**—Determine the purity by gas chromatography in accordance with the technique described in 6.1.1 – 6.1.1.6 or by gas chromatography/mass spectrometry (GC/MS) in accordance with 6.1.2 – 6.1.2.5.

6.1.1 Purity by GC:

6.1.1.1 Prior to sample introduction, a standardization of the GC shall be performed using a known bromotrifluoromethane purity standard.

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

(a) **Gas Chromatograph System (GC System)**, equipped with a flame ionization detector (FID), capillary column split injector, subambient (liquid nitrogen) cooling valve, and an electronic integrator or suitable computer data gathering and reduction system.

(b) **210 m Chromatographic Column**—Connect the two columns below together with the first column end attached to the injection port:

(1) 105 m Rtx⁶-1701 (14 % cyanopropylphenyl – 86 % methylpolysiloxane), 0.25 mm, 1µm.

(2) 105 m Rtx⁶-200 (5 % diphenyl – 95 % dimethyl polysiloxane), 0.25 mm, 1µm.

(3) The two columns are attached together, with the first (Column 1) attached to the injection port of the GC system. Columns are available from several chromatographic supply vendors.

6.1.1.3 **Sample Introduction**—Take sample from the liquid phase employing the procedure described in 5.1 of this specification. Introduce and transfer the sample into the GC using a standard gas-tight syringe.

6.1.1.4 **Reagents**—The carrier gas shall be a chromatographic grade of helium.

6.1.1.5 Temperature Programming:

(a) Install the column in the gas chromatograph and adjust the oven temperature to -20 °C. Inject the sample and hold the column oven at -20 °C for 21 min, then program the temperature to rise 15 °C/min to a maximum of 165 °C, then post hold for 20 min before recycling.

6.1.1.6 Calculation:

(a) Calculate weight percent of Halon 1301 as follows, and convert it to mole percent in 6.1.1.6(b) if necessary.

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

where:

W_i = weight percent of component i ,
 A_i = peak area of component i ,
 RRF_i = relative response factor for component i ,
 and
 $\sum [A_i \times RRF_i]$ = sum of all component peak areas times their respective relative response factors.

(b) For ASTM specifications, the results shall be converted to and reported in mole percentages. Convert individual impurities from wt % to mol % as follows:

$$\text{mol } \%_i = 100 \times (W_i/M_i) / \sum [W_i/M_i] \quad (4)$$

where:

$\text{mol } \%_i$ = mol percent of component i ,
 W_i = weight percent of component i ,
 M_i = molecular weight of component i , and
 $\sum [W_i / M_i]$ = sum of all component weights divided by their respective molecular weights.

NOTE 1—The GC method described in 6.1.1 – 6.1.1.6 may not detect certain impurities, for example, impurities that may elute within or near the relatively large Halon 1301 peak. If other tests indicate the presence of unidentified impurities, then the gas chromatography/mass spectrometry (GC/MS) method described in 6.1.2 is recommended.

6.1.2 Purity by GC/MS:

6.1.2.1 Prior to sample introduction, a standardization of the GC/MS shall be performed using a known bromotrifluoromethane purity standard.

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

(a) **Gas Chromatography/Mass Spectrometry System (GC/MS System)** and an electronic integrator or suitable computer data-gathering and reduction system.

(b) **Chromatographic Column**, 344 ft [105 m] Rtx⁶-1 capillary column, 0.01 in. [0.25 mm] internal diameter, stationary film thickness of approximately 1 µm. (Column is available from several chromatographic supply vendors.)

⁶ Rtx is a registered trademark of Restek Corporation.

(c) *Sample Introduction*—Take sample from the liquid phase employing the procedure described in 5.1. Using a standard gas-tight syringe introduce the sample into the GC/MS.

6.1.2.3 *Reagents*—The carrier gas shall be a chromatographic grade of helium.

6.1.2.4 *Temperature Programming*:

(a) Install the column in the gas chromatograph/mass spectrometer and adjust the oven temperature to -30 °C. Inject the sample and hold the column oven at -30 °C for 13 min, then program the temperature to rise 10 °C/min to a maximum of 200 °C, then posthold for 10 min before recycling.

6.1.2.5 *Calculation*:

(a) Calculate weight percent of Halon 1301 as follows, and convert it to mol percent in 6.1.2.5(b) if necessary:

$$W_i = \frac{A_i \times RRF_i \times 100}{\sum [A_i \times RRF_i]} \quad (5)$$

where:

W_i = weight percent of component i ,
 A_i = peak area of component i ,
 RRF_i = relative response factor for component i , and
 $\sum [A_i \times RRF_i]$ = sum of all component peak areas times their respective relative response factors.

(b) For ASTM specifications, the results shall be converted to and reported in mole percentages. Convert individual impurities from wt % to mol % as follows:

$$\text{mol } \%_i = 100 \times (W_i/M_i) / \sum [W_i/M_i] \quad (6)$$

where:

$\text{mol } \%_i$ = mole percent of component i ,
 W_i = weight percent of component i ,
 M_i = molecular weight of component i , and
 $\sum [W_i / M_i]$ = sum of all component weights divided by their respective molecular weights.

6.2 *Acidity*—Determine acidity using the method specified in 2008 Appendix C to AHRI Standard 700-2014, Part 1.

6.3 *Water Content*—Determine water content using the method specified in 2008 Appendix C to AHRI Standard 700-2014, Part 2.

6.4 *Qualitative Test for Halogen Ion*—Test a sample for the presence of halogen ions in accordance with 6.4.1 – 6.4.2 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 *Reagents*:

6.4.1.1 *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.

6.4.1.2 *Nitric Acid*, 1:1 v/v in water.

6.4.2 *Procedure*—To the water scrubber solution saved during the acidity test (6.2), add 1 drop of 1:1 HNO₃, 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.

6.5 *Nonvolatile Residue*—Determine the nonvolatile residue in accordance with the gravimetric method specified in 2008 Appendix C to AHRI Standard 700-2014, Part 3.

6.6 *Non-Condensable Gases*—Determine non-condensable gases using the method specified in 2008 Appendix C to AHRI Standard 700-2014, Part 4.

6.7 *Suspended Matter and Sediment*—While performing the nonvolatile residue analysis, examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

6.8 Determine color with the method specified in Test Method D2108. Results should be used as an indicator of other contamination, and must have a description of what is observed as well as the value recorded on the final report. Results from the color test that are inconsistent with outcome from other required tests will be the basis for repeat testing to validate the test results of specific properties.

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 D5632/D5632M Type II (pure) Halon 1301 is “Bromotrifluoromethane (Refrigerant Gas R 13B1),” UN 1009, Hazard Class 2.2 (nonflammable gas). The proper shipping name for nitrogen superpressurized Halon 1301 is “Liquefied Gas, nonflammable charged with nitrogen,” UN1058, Hazard Class 2.2. In addition to DOT requirements, containers shall be marked with the following information as a minimum:

7.1.1 Supplier’s name and address,

7.1.2 Halon 1301, Bromotrifluoromethane,

7.1.3 Statement that material conforms to Specification D5632/D5632M, 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.

7.2 *Extreme Elevated Temperature Considerations*—When Type I or Type II mixtures of Halon 1301 and N₂ may be exposed to constant temperatures at or greater than 131 °F [55 °C] during transportation or storage, then higher container pressures will be encountered that require alternative fill in the container to that specified in 4.1.1. In this instance, limit container fill density to a maximum of 62.5 lb/ft³ [1001 kg/m³]. A maximum fill density of 62.5 lb/ft³ [1001 kg/m³] and a maximum NAG content of 19.3 % should be observed if it is desired to prevent a liquid full condition up to 149 °F [65 °C]. The isometric diagram for nitrogen-superpressurized Bromotrifluoromethane is shown in Fig. 1.

8. Keywords

8.1 bromotrifluoromethane; CF₃Br; fire fighting; firefighting agent; fire protection; fire suppressant; fluorobrominated hydrocarbon; Halon 1301



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