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Standard Specification for Halon 1301, Bromotrifluoromethane (CF₃Br)¹

This standard is issued under the fixed designation D5632; 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 D5631.

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

1.4 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are mathematical conversions that are provided 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 ~~4.34.5~~.

2. Referenced Documents

2.1 *ASTM Standards:*²

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

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

2.3 ~~U.S. Industry Standard: AHRI Standards:~~⁴

ASQ Z1.4 Sampling Procedures and Tables for Inspection by Attributes (ANSI Approved, DoD Adopted) 2008 Appendix C for Analytical Procedures for AHRI Standard 700-2006

2.4 *U.S. Government Standards:*⁵

CFR Title 49, Part 172.101 Tables of Hazardous Materials and Special Provisions

CFR Title 49, Part 172 Subpart D Marking Requirements of Packaging for Transportation

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 ~~halon-1301~~ Halon 1301 — The halogenated hydrocarbon compound Bromotrifluoromethane used as a fire extinguishing medium.

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 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, 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.~~

⁴ Available from Air-Conditioning, Heating, and Refrigeration Institute, 2111 Wilson Blvd, Suite 500, Arlington, VA 22201.

⁵ ~~Available from Matheson Co., 430 T Caredean Road, Horsham, PA 19044. Phone 215-674-0686.~~

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20036.

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.

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

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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~~ 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~~ 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, ~~the~~ 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 ~~(yielding 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).

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~~ 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 ~~fixed~~ non-condensable gases in vapor phase, expressed as ~~air~~ nitrogen when tested by the appropriate test method(s) listed in Section 6.

4.3 ~~By~~ 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.

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 ~~halons~~ Halon 1301 shall be taken from the liquid or vapor phase as appropriate. Samples of ~~halons~~ Halon 1301, taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427 or ~~ASQZ1-4; the method specified in 2008 Appendix C Inspection Level S-4. The~~ for Analytical Procedures for AHRI Standard 700-2006, 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.

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
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

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—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.~~

~~6.1.1.1 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

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:~~

~~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 Carbopack 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—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 RTx-1701 (14 % cyanopropylphenyl – 86 % methylpolysiloxane), 0.25 mm, 1µm.~~

~~(2) 105 m RTx-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 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—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. 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:—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 posthold 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.~~

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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.

~~6.1.5.2 For (b) For ASTM specifications, the results must shall be converted to and reported in mole percentages. Convert individual impurities from $W_{wt} \%$ to $M_{mol} \%$ as follows:~~