

# **ASTM D7673-10 Standard Specification for Halon 1211**

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# Outline

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# Background

- **Need for a standard specification for Halon 1211 prompted by the mid-2009 discovery of contaminated Halon 1211**
- **Handheld extinguishers containing contaminated Halon 1211 installed on European aircraft, some on US carriers**

# Background

- **Halon 1211 Standards prior to ASTM D7673**
  - *ISO 7201-1*
  - *MIL-DTL-38741A*
  - **Both standards lack detailed methodology for purity assay via gas chromatography (GC)**
  - **Presents risk of failure to detect and/or identify impurities in Halon 1211**

# ASTM D7673-10 Standard Specification for Halon 1211: Scope

- Requirements for Halon 1211 as a firefighting medium
- Does not address equipment or hardware
- Does not address handling, transportation or storage
  - *These will be addressed in a new standard “Halon 1211 Standard Practice for Handling, Transportation and Storage of Halon 1211, Bromochlorodifluoromethane (CF<sub>2</sub>BrCl)” – in preparation*

## Referenced Documents

- ASTM D2108 Test Method for Color of Halogenated Organic Solvents and Their Admixtures (Pt-Co Scale)
- AHRI Standard 700-2006, Appendix C (analytical procedures)
- ISO 3427 Gaseous Halogenated Hydrocarbons (Liquefied Gases) – Taking of a Sample
- 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

# Material Requirements

- **Type I – Mixtures of Halon 1211 and Nitrogen**
- Nitrogen partial pressure such that safe working pressure of receiving vessel is not exceeded
- Fill density should not exceed that needed to completely fill the container at the maximum envisaged storage temperature
- Example:
  - US DOT 4W260 cylinder: for a 100 lb/ft<sup>3</sup> fill density, the nitrogen partial pressure should not exceed 129 psig at 70 °F if the maximum envisaged storage temperature is 131 °F
    - *at 131 °F the cylinder is not liquid full and the safe working pressure of the cylinder is not exceeded*

# Material Requirements

- **Type I – Mixtures of Halon 1211 and Nitrogen**
- When a material analysis is required it shall include:
  - Total container pressure
  - Partial pressure of nitrogen
  - Fill density of Halon 1211
  - Maximum safe storage temperature



# Material Requirements

- **Type I – Mixtures of Halon 1211 and Nitrogen**
  - Shall conform to the following requirements

## Table 1: Requirements

Property	Requirement
Halon 1211 purity, % by mol, min	99.0
Acidity, ppm by mass, max	3.0
Water content, ppm by mass, max	20
Nonvolatile residue, % by weight, max	0.02
Suspended matter or sediment	None visible
Color	Report value

# Material Requirements

- **Type II – Halon 1211**
- Shall conform to purity requirements of Type I
- Shall contain no more than 1.5% by volume non-condensable gases in the vapor phase, expressed as air
- By agreement between purchaser & supplier, analysis may be required and limits established for compounds not specified in Table 1

# Sampling

- **Samples shall be taken from liquid or vapor phase as appropriate**
- **Liquid phase sampling**
  - *In accordance with ISO 3427 or*
  - *2008 Appendix C for Analytical Procedures for AHRI Standard 700-2006, Part 7*
- **Sampling vessel shall be capable of resisting the vapor pressure of the sample at the highest temperature that could be encountered during handling, storage, or transport**

# Test Methods

- **Purity:** by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS)
- **Acidity:** 2008 App. C to AHRI 700-2006, part 1
- **Water Content:** 2008 App. C to AHRI 700-2006, part 2
- **Nonvolatile Residue:** 2008 App. C to AHRI 700-2006, part 3
- **Non-condensable gases:** 2008 App. C to AHRI 700-2006, part 4
- **Suspended Matter and Sediment:** Visual observation
  - *Any observed suspended matter or sediment = failure*
- **Color:** ASTM D2108
  - *Results should be used as an indicator of other contamination*
  - *Results inconsistent with the outcome from other required tests will be basis for repeat testing to validate test results of specific properties*

# Purity by Gas Chromatography

- **Standardization of the GC must be performed using a known Halon 1211 purity standard**
- **Gas Chromatography System**
  - Flame ionization detector (FID)
  - Capillary column split injector
  - Sub-ambient (liquid nitrogen) cooling valve
  - Electronic integrator or suitable data gathering/reduction system
- **Gas Chromatographic Column**
  - 210 m column: (a) 105m RTx-1701 plus (b) 105 m RTx-200
  - Columns 0.25 mm ID, film thickness 1 micron

# Purity by Gas Chromatography

- **Sample Introduction**
  - Liquid phase sample
  - Injection using standard gas-tight syringe
- **Chromatographic Grade Helium Carrier Gas**
- **Temperature Programming:**
  - -20 °C for 21 min
  - 15 °C per minute rise to 165 °C
  - Posthold at 165 °C for 20 min before recycling

# Purity by Gas Chromatography

- Section 6, Note 1

**Note 1 – The GC method described in 6.1.2-6.1.5 may not detect certain impurities, for example, impurities which may elute within or near the relatively large Halon 1211 peak. If other tests indicate the presence of unidentified impurities, then the gas chromatography/mass spectrometry (GC/MS) method described in 6.2.2-6.2.5 is recommended.**

## Purity by GC/MS

- **Standardization of the GC/MS must be performed using a known Halon 1211 purity standard**
- **Gas Chromatography/Mass Spectroscopy System**
  - GC/MS System
  - Electronic integrator or suitable data gathering/reduction system
- **Chromatographic Column**
  - 105 m RTx-1 capillary column
  - 0.25 mm ID, film thickness 1 micron



# Purity by GC/MS

- **Sample Introduction**
  - Liquid phase sample
  - Injection using standard gas-tight syringe
- **Chromatographic Grade Helium Carrier Gas**
- **Temperature Programming:**
  - -30 °C for 13 min
  - 10 °C per minute rise to 200 °C
  - Posthold at 200 °C for 10 min before recycling

# Container, Packaging, and Package Marking

- **Containers shall be marked in accordance with 49 CFR Part 172 Subpart D**
- **Shipping name: ASTM D7673 Type II (pure)**  
**Chlorodifluorobromomethane (Refrigerant Gas R 12B1)**  
**UN 1974, Hazard Class 2.2 (nonflammable gas)**
- **Shipping name: ASTM D7673 Type I (N<sub>2</sub> pressurized)**  
**Liquefied Gas, nonflammable charged with nitrogen**  
**UN1058, Hazard Class 2.2 (nonflammable gas)**

# Container, Packaging, and Package Marking

- **In addition to DOT requirements, containers must be marked with the following information as a minimum:**
  - **Supplier's name and address**
  - **Halon 1211 (Bromochlorodifluoromethane)**
  - **Statement that material conforms to Specification D7673**
  - **For storage or transport within the US, a warning label shall be affixed to the container conforming with US EPA requirements, in accordance with Section 6.1 of the Clean Air Act, as amended**

# Container, Packaging, and Package Marking

- **Extreme Elevated Temperature Considerations**
  - **When Type I or Type II Halon 1211 may be exposed to constant temperatures  $\geq 131$  °F (55 °C) during transport or storage, higher container pressures will be encountered that required alternative fill densities compared to those specified in 4.1.1**
  - **A maximum non-condensable gas content of 1.5% is recommended for long-term storage of Type II Halon 1211**

# Conclusion

- **ASTM D7673-10 covers the requirements for Halon 1211 as a fire fighting medium**
- **Purity analysis includes detailed GC and GC/MS methodologies**
- **Adherence to ASTM D7673 will ensure the performance and safety in use of Halon 1211 for suppliers, equipment manufacturers and end-users**
- **Requirements for the handling, transport and storage of Halon 1211 will be specified in a new ASTM standard (in progress)**
- **ASTM D5632-08 Standard Specification for Halon 1301 will be updated based on the new Halon 1211 Standard Specification (in progress)**