[METRIC]
MIL-DTL-38741A
21 December 1998
SUPERSEDING
MIL-B-8741(USAF)
9 April 1984

DETAIL SPECIFICATION

BROMOCHLORODIFLUOROMETHANE, TECHNICAL

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers one technical grade of bromochlorodifluoromethane for use as a fire-extinguishing agent. This material is also referred to as Halon-1211.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Defense Supply Center Richmond (DSCR), ATTN: DSCR-VBD, 8000 Jefferson Davis Highway, Richmond, VA 23297-5610 by using Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A FSC 6830

2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 1209 - Standard Test Method for Color of Clear Liquids (Platinum-Cobalt Scale).

ASTM D 2108 - Standard Test Method for Color of Halogenated Organic Solvents and Their Admixtures (Platinum-Cobalt Scale).

(Applications for copies of ASTM documents should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3. Material. The material shall be a technically pure grade of bromochlorodifluoromethane and shall conform to the requirements of table I.

TABLE I. Requirements for bromochlorodifluoromethane.

Characteristic	Requirement	Test
Assay as bromochlorodifluoromethane, percent by weight, minimum	99.0	4.4.1
Acid halides, ppm by weight, maximum	3.0	4.4.2
Free halogens, ppm by weight, maximum	3.0	4.4.3
Nonvolatile residue, grams/100 mL, maximum	0.02	4.4.4
Suspended matter or sediment	None	4.4.5
Color (Platinum Cobalt Color Standard)	Equal to or less than #15	4.4.6
Moisture, mg/kg, maximum	20.0	4.4.7

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as conformance inspection (see 4.3).

4.2 Sampling for tests. Sampling may be used for lot acceptance. The material not used during testing may be returned to the lot after acceptance (see 6.2).

4.3 Conformance inspection. Conformance inspection shall be performed in accordance with inspection provisions set forth in table I. The characteristics required by table I, when tested in accordance with 4.4, shall constitute minimum inspections to be performed by the supplier prior to Government acceptance or rejection by lot. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies, submitted to the Government for acceptance, comply with all requirements of the contract.

4.4 Test methods.

4.4.1 Assay. The percentage of bromochlorodifluoromethane shall be determined by programmed temperature gas chromatography using a packed column and flame ionization detector (FID). Component peak areas are integrated electronically and quantified by the area normalization-response factor method. Manufacturers may use the test method of 4.4.1.1 through 4.4.1.7 or another method determined acceptable by the procuring agency.

4.4.1.1 Apparatus. The following apparatus is required to determine the percentage of bromochlorodifluoromethane:

a. Temperature programmable gas chromatograph with dual flame ionization detectors, 500 μL sample loop, and packed column capability. One detector is used for gas analysis; the other is used as a baseline reference.

b. Electronic integrator or suitable data acquisition system and chromatography software.

c. Packed column, 24 feet by 1/8-inch OD stainless steel, packed with either 1% AT-1000 on Carbograph 1 [Alltech] or 1% SP-1000 on Carbopak B [Supelco], 60/80 mesh.

d. Sampling apparatus for vaporizing liquid refrigerant samples. The apparatus shown in figure 1 may be employed and comprises a gas sampling bulb (either 250 mL or 500 mL) wrapped with fiberglass tape, a vacuum gauge, appropriate valves, and an optional septum.

4.4.1.2 Reagents.

a. The chromatography gases used are UHP helium (carrier gas), UHP hydrogen (detector fuel), and zero grade air (oxidizer).

b. Small quantities of the substances listed in table II are used to prepare the calibration standard. Calibration standards may be purchased commercially or prepared in accordance with 4.4.1.4. The purity of each compound listed in table II must be predetermined using the chromatographic operating conditions in 4.4.1.3.

Table II. Calibration substances.

Common Name	IUPAC Convention Name	CAS Number
CFC-12	dichlorodifluoromethane	75-71-8
CFC-13	chlorotrifluoromethane	75-72-9
Halon 1201	bromodifluoromethane	1511-62-2
Halon 1202	dibromodifluoromethane	75-61-6
Halon 1211	bromochlorodifluoromethane	353-59-3
Halon 1301	bromotrifluoromethane	75-63-8
HCFC-22	chlorodifluoromethane	75-45-6
HFC-23	trifluoromethane	75-46-7

4.4.1.3 Operating conditions.

a. Detector	FID
b. Carrier Gas	Helium; 30 mL $min.^{-1}$
c. Initial Column Temp.	40 °C
d. Initial Hold	12 min.
e. Program	20 K $min.^{-1}$
f. Final Column Temp.	160 °C
g. Post Hold	15 min.
h. Sample Loop	1 mL
i. Detector Temp.	200 °C
j. Injection Port Temp.	150 °C

4.4.1.4 Calibration standard preparation.

a. Evacuate a clean; dry; 1,000 mL; aluminum, carbon steel, or stainless steel; sample cylinder to an absolute pressure of not more than 25 Pa. Weigh the evacuated cylinder to the nearest 0.1 milligram. Using a vacuum manifold to avoid the introduction of air, add 11 grams of bromochlorodifluoromethane vapor to the cylinder. Close the cylinder and reweigh, recording the amount of bromochlorodifluoromethane added to the nearest 0.1 milligram.

> CAUTION - Do not fill the sample cylinder to more than 12 grams per liter or conduct any laboratory work in an ambient temperature below 15 °C. Failure to observe these precautions will cause the calibration standard to condense and fractionate, resulting in sporadic data.

b. Attach a septum to the cylinder and chill the base of the cylinder in liquid nitrogen for 15 minutes. Do not purge the cylinder when attaching the septum as this would alter the relationship between the bromochlorodifluoromethane and the contaminants. Instead, the small amount of air behind the septum is permitted to enter the standard when the valve is opened.

c. After verifying that the septum is still attached securely, open the valve. Keep the cylinder in liquid nitrogen.

d. Using appropriately sized syringes, individually and in turn add the gaseous contaminants listed in table III in the amount indicated. The indicated vapor densities are based on a laboratory temperature of 20.0 °C and a barometric pressure of 100.0 kPa. These data must be adjusted to reflect actual laboratory conditions.

e. Using an appropriately sized syringe, add the liquid contaminant listed in table III in the amount indicated. For best accuracy, this contaminant and its syringe should be pre-chilled in a freezer (approximately -20 °C) and promptly transferred. The indicated liquid density is based on available manufacturer data and is approximate due to the absence of practical temperature control.

TABLE III. Calibration standard impurities.

Component	Vapor Density, mg/mL	Volume Added, mL	mg Added[1]	mg/kg Added[2]	Total mg/kg Present[3]
HFC-23	2.895	3.9	11.3		
CFC-13	4.331	2.6	11.3		
HCFC-22	3.606	3.2	11.5		
Halon 1301	6.288	1.8	11.3		
Halon 1201	5.355	2.1	11.2		
CFC-12	5.077	2.2	11.2		
Component	Liquid Density, mg/μL	Volume Added, μL	mg Added[1]	mg/kg Added[2]	Total mg/kg Present[3]
Halon 1202	2.463	4.6	11.3		

[1] If necessary, correct mg added for the purity of the calibration component.
[2] Values are determined during preparation of standard.
[3] Column to be filled in, (see 4.4.1.5), after determining ppm present in stock components (see 6.3).

f. Close the valve and allow the calibration standard cylinder to return to ambient temperature. Set the cylinder aside for a minimum of 12 hours to allow all the cylinder's contents to equilibrate.

4.4.1.5 Calibration standard analysis.

a. Analyze the standard in triplicate using the chromatographic operating conditions of 4.4.1.3. Adjust the standard's recorded component and contaminant levels to account for any significant impurities encountered in the starting materials using the method of standards addition (see 6.3). A sample chromatogram of a calibration standard showing the order and positioning of compound elution appears in figure 2.

b. Determine the weight percentage to the nearest 0.01 percent of each compound, including the bromochlorodifluoromethane and each contaminant, in the calibration standard using the formula:

$$W_N = \frac{100 \times M_N}{M_T}$$

Where W_N = Weight percentage of compound being measured.
M_N = Mass of compound in calibration standard.
M_T = Total mass in calibration standard.

c. The new standard can be used for a period of approximately six months, provided that sufficient material remains in the container. The gas chromatograph should be recalibrated using the standard as often as necessary to maintain confidence in the analytical results.

4.4.1.6 Determination of contaminant response factors.

a. Determine and record each compound's absolute response factor as follows:

$$ARF_N = \frac{W_N}{A_N}$$

Where ARF_N = Absolute response factor of compound N.
W_N = Weight percentage of compound N.
A_N = Peak area of compound N (average of 3 runs).

b. Using the bromochlorodifluoromethane as the reference peak, determine and record each contaminant's relative response factor as follows:

$$RRF_N = \frac{ARF_N}{ARF_{BROMOCHLORODIFLUOROMETHANE}}$$

Where RRF_N = Relative response factor of contaminant N (compute to four significant figures).
ARF_N = Absolute response factor of contaminant N.

4.4.1.7 Specimen analysis. The specimen bromochlorodifluoromethane is analyzed using the operating conditions listed in 4.4.1.3. The analysis shall be performed as follows:

a. Load the specimen as illustrated in figure 1 by flashing liquid into a suitable apparatus or bag. The vaporized sample may have a very slight positive pressure with respect to atmosphere, but should not exceed 10 psig. To assure the analytical integrity of the result, the entire aliquot of bromochlorodifluoromethane must be vaporized.

b. Using a vacuum manifold, evacuate the sample loop and interconnecting tubing to the sample apparatus valve. Close the valve to the vacuum pump and load the sample loop with the specimen bromochlorodifluoromethane.

c. Analyze the specimen bromochlorodifluoromethane. The most frequently encountered impurities in bromochlorodifluoromethane are listed in table III and will be available in the calibration standard. Use component spiking and/or GC/MS to identify and quantify any unknown peaks which appear in the analysis.

d. The weight percentage of bromochlorodifluoromethane is calculated as follows:

$$W_B = \frac{100 \times A_B \times RRF_B}{\sum_{1\ to\ i}(A_i \times RRF_i)}$$

Where W_B = Weight percentage of bromochlorodifluoromethane.
A_B = Peak area of bromochlorodifluoromethane.
RRF_B = Relative response factor for bromochlorodifluoromethane.
$\Sigma_{1\ to\ i}$ = Sum of all component peak areas times their respective response factors.

4.4.2 Acid halides. A large sample of the specimen bromochlorodifluoromethane shall be evaporated in the presence of a small amount of crushed ice-distilled water slurry. The solution is titrated for acid halides with standardized sodium hydroxides.

4.4.2.1 Reagents.

a. Sodium hydroxide, 0.01*N* solution: Dissolve 0.40 grams of carbonate-free sodium hydroxide in recently boiled distilled water in a 1,000 mL volumetric flask. Cool and dilute to the mark. This solution shall be standardized against reagent grade potassium acid phthalate.

b. Methyl red indicator, 0.1% solution.

CAUTION - Perform this procedure in a hood.

4.4.2.2 Procedure. Place 10 mL of a crushed ice-distilled water slurry in a 250 mL glass stoppered Erlenmeyer flask and add 50 grams of bromochlorodifluoromethane to the slurry. Place the stopper in the flask loosely and swirl the flask gently from time to time until the ice is completely melted. Add one drop of methyl red indicator and if a reddish color remains, titrate to a yellow endpoint with 0.01*N* sodium hydroxide solution. The ppm acid halides, as HBr, shall be calculated as follows:

$$\text{ppm Acid halides (as HBr)} = \frac{A \times N \times \left(0.0809 \times 10^{6}\right)}{W}$$

where: A = Amount of NaOH solution in milliliters.
N = Normality of NaOH solution.
W = Weight of bromochlorodifluoromethane sample in grams.

4.4.3 Free halogen. The free halogen shall be treated with an excess of potassium iodide and the iodide liberated titrated with sodium thiosulfate solution.

4.4.3.1 Reagents.

a. Sodium thiosulfate, 0.01*N* solution: Prepare a 0.1*N* solution by dissolving 25 grams of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) and 0.5 gram of sodium carbonate in 1 liter of distilled water. Standardize against 0.1*N* potassium dichromate solution. From this 0.1*N* solution of sodium thiosulfate, prepare a 0.01*N* solution as follows: Pipet a 10 mL aliquot of the standard 0.1*N* sodium thiosulfate solution into a 100 mL volumetric flask and dilute to the mark with distilled water and mix. Prepare the 0.01*N* sodium thiosulfate solution fresh daily.

b. Sulfuric acid, 1:4 solution in water.

c. Potassium iodide, 10 percent solution in water.

d. Starch indicator.

4.4.3.2 Procedure. Pour 100 mL of 10 percent potassium iodide solution into a 250 mL Erlenmeyer flask. Add 10 mL 1:4 sulfuric acid and 1 mL of starch indicator solution. Bubble 100 grams of bromochlorodifluoromethane vapor through the potassium iodide solution. Titrate any liberated iodine with standard 0.01*N* sodium thiosulfate solution. Run a reagent blank along with the sample. Calculate the free halogen, as bromine, as follows:

$$\text{ppm Free halogen (as Br)} = \frac{(A - B) \times N \times \left(0.0799 \times 10^{6}\right)}{W}$$

Where: A = Milliliters of sodium thiosulfate solution required for titration of the sample.
B = Milliliters of sodium thiosulfate solution required for titration of the blank.
N = Normality of sodium thiosulfate solution.
W = Weight of bromochlorodifluoromethane sample used in grams.

4.4.4 Nonvolatile residue. Add 100 mL of bromochlorodifluoromethane to an evaporating dish which has been weighed to nearest milligram. Allow to evaporate to dryness in a hood. After evaporation is complete, dry for 15 minutes in a drying oven at 105 °C. Cool the evaporating dish in a desiccator and reweigh to nearest milligram. Calculate the non-volatile residue as follows:

Nonvolatile residue in grams per 100 mL = $B - A$

where: B = Weight of dish and residue after process.
A = Weight of dish before process.

4.4.5 Suspended matter or sediment. Examine visually for any suspended matter or sediment.

4.4.6 Color. A sample of the bromochlorodifluoromethane shall be determined in accordance with ASTM D 1209 or ASTM D 2108.

4.4.7 Moisture. The analysis may be conducted by the phosphorous pentoxide method, infrared absorption, by an electrolytic moisture analysis, or by a piezoelectric analyzer. The accuracy of the results shall be by orthodox Karl Fischer method.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. The bromochlorodifluoromethane covered by this specification is intended for use as a fire extinguishing agent.

6.1.1 Military unique rationale. Because bromochlorodifluoromethane is no longer produced, all requisitions must be for reprocessed material. The reprocessed bromochlorodifluoromethane covered by this specification is military unique because it is used in military aircraft and the parameters called out may not be required for use of the product in commercial applications. In addition, the military specification is used as a basis for the national specification for reprocessed bromochlorodifluoromethane.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of this specification.

b. Quantity of bromochlorodifluoromethane desired.
c. Sampling plan to be used, if required (see 4.2).
d. Specification for desired container.
e. Issue of DoDISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.2).
f. Packaging requirements (see 5.1).

6.3 Impurities in bromochlorodifluoromethane. The purest bromochlorodifluoromethane will contain some of the contaminants listed in table II in low concentrations. During chromatographic analysis of the calibration standard, individual impurity peak areas are increased by the peak areas corresponding to the mass of the impurity in the stock bromochlorodifluoromethane. The actual mass of each contaminant is thereby determined by the method of standards addition. The mass of each contaminant present in the stock bromochlorodifluoromethane is combined with the mass added to give the total mass of each contaminant of the calibration standard.

6.4 Inspection lot. Bromochlorodifluoromethane manufactured as one batch and offered for delivery at one time shall be considered a lot for purposes of inspection and test.

6.5 Subject term (key word) listings.

Fire extinguishing
Fire fighting
Halon

6.6 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodian:
Air Force - 68

Preparing activity:
DLA - GS

(Project 6830-1024)

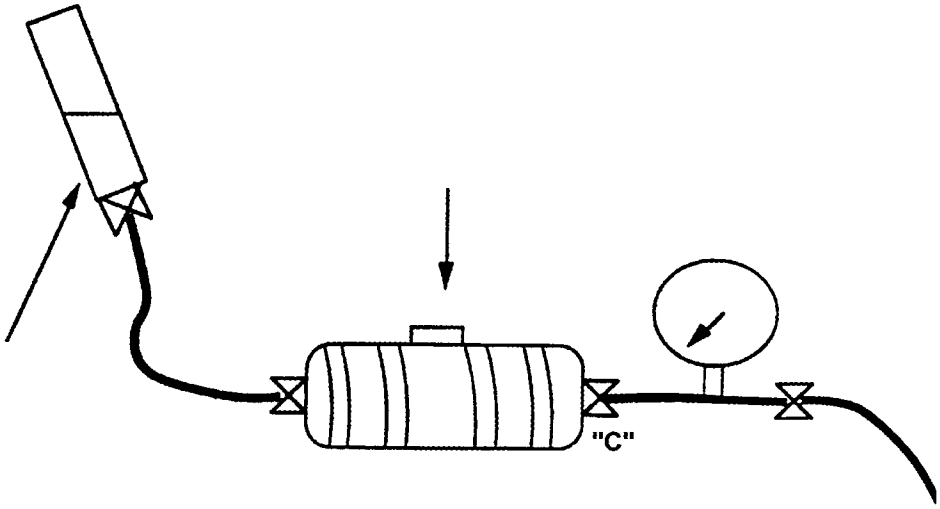
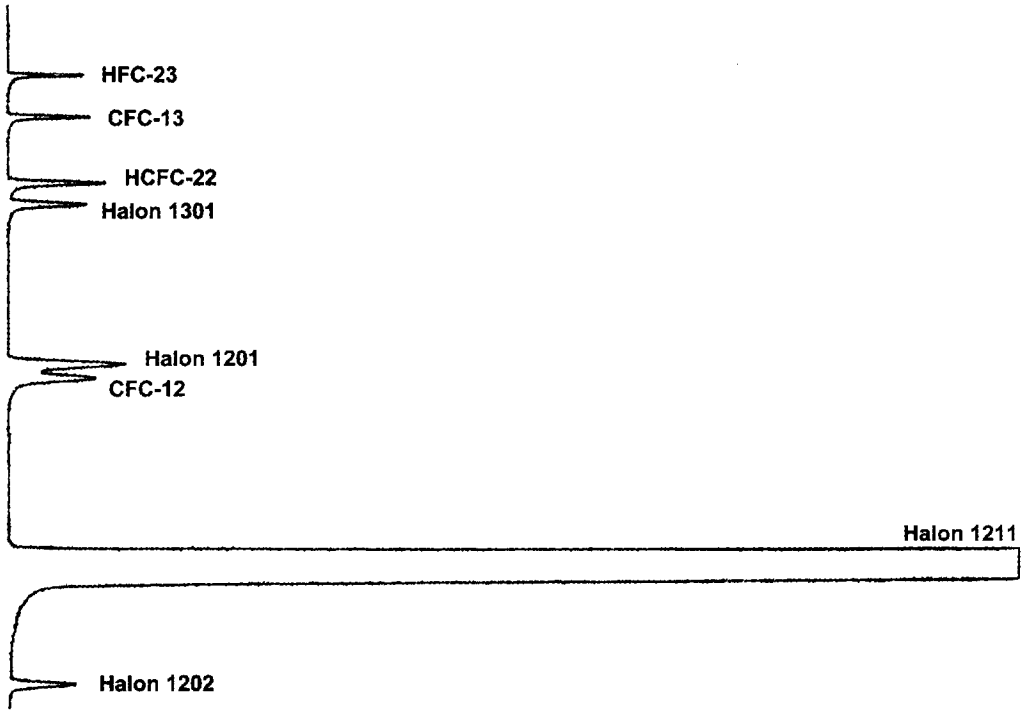


FIGURE I - APPARATUS USED FOR SAMPLING FOR ASSAY IN (4.4.1)



STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.

2. The submitter of this form must complete blocks 4, 5, 6, and 7.

3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	**1. DOCUMENT NUMBER** MIL-DTL-38741A	**2. DOCUMENT DATE (YYMMDD)** 21 DECEMBER 1998

3. DOCUMENT TITLE BROMOCHLORODIFUOROMETHANE, TECHNICAL

4. NATURE OF CHANGE *(Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME *(Last, First, Middle Initial)*	b. ORGANIZATION	
c. ADDRESS *(Include Zip Code)*	d. TELEPHONE (Include Area Code) (1) Commercial (2) AUTOVON *(if applicable)*	**7. DATE SUBMITTED** (YYMMDD)

8. PREPARING ACTIVITY

a. NAME DEFENSE SUPPLY CENTER RICHMOND	b. TELEPHONE *Include Area Code)* (1) Commercial (2) AUTOVON
c. ADDRESS *(Include Zip Code)* ATTN: DSCR-VBD 8000 JEFFERSON DAVIS HIGHWAY RICHMOND, VA 23297-5610	**IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT:** DEFENSE QUALITY AND STANDARDIZATION OFFICE 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22401-3466 Telephone (703) 756-2340 AUTOVON 289-2340