

MIL-D-4540B
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MILITARY SPECIFICATION

DIBROMODIFLUOROMETHANE, TECHNICAL

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

1. SCOPE

1.1 This specification covers a technical grade of dibromodifluoromethane intended for use as fire extinguisher fluid also known as DB.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

MIL-T-27730 Tape, Antiseize, Tetrafluoroethylene, with Dispenser

MIL-C-45662 Calibration Systems Requirements

STANDARDS

MIL-STD-101 - Color Code for Pipelines and for Compressed - Gas Cylinders

MIL-STD-129 - Marking for Shipment and Storage

MIL-STD-147 - Palletized and Containerized Unit Loads, 40" X 48" Pallets, Skids, Runners, or Pallet-Type Base

(Copies of Military Specifications and Standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

FSC 6830

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Department of Transportation (DOT)

49CFR171-190 - Code of Federal Regulations.

(The Code of Federal Regulations (CFR) and the Federal Register (FR) are for sale on a subscription basis by the Superintendent of Documents, US Government Printing Office, Wash DC 20402. When indicated, reprints of certain regulations may be obtained from the federal agency responsible for issuance thereof.)

American Society for Testing and Materials Publications

ASTM Standards, Parts 17 and 30

(Copies of ASTM publications may be obtained upon application to the American Society for Testing and Materials, 1916 Race Street, Philadelphia PA 19103.)

Compressed Gas Association

V-1 American-Canadian Standard Compressed Gas Cylinder Valve Outlet and Inlet Connection; ASA-B 57.1; CSA - B96

(Copies of CGA publications may be obtained from Compressed Gas Association, Inc., 500 Fifth Avenue, New York NY 10036.)

2.2.1 Technical Society and Technical Association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using federal agencies.

3. REQUIREMENTS

3.1 Material. The dibromodifluoromethane shall conform to the requirements specified in Table I, when tested in accordance with the applicable test method.

3.2 Limiting Values. The following applies to all specified limits of this specification. For purposes of determining conformance with these requirements, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limitation value, in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E29-67.)

3.3 Cylinder Maintenance. Government- and supplier-furnished cylinders that require maintenance shall be processed by the supplier for serviceability to meet the requirements of this specification and 49CFR171-190 (6.2.)

3.3.1 Treatment and Painting. Government-furnished cylinders for dibromodifluoromethane requiring repainting shall be cleaned, treated, and painted. Each cylinder shall be color coded and marked in accordance with MIL-STD-101.

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TABLE I

Physical and Chemical Requirements

Characteristic	Properties	Test Paragraph
Appearance Sediment Suspended matter	None None	4.6.1
Color	Colorless	4.6.1
Odor Initial Residual	Characteristic None	4.6.2
Specific gravity, 15°/15° C	2.270 to 2.310	4.6.3
Index of refraction, 15° C	1.3960 to 1.4040	4.6.4
Distillation range at 760 mm pressure, °C (max)	21.0 to 32.0 (95% by vol)	4.6.5
Nonvolatile residue, grams per 100 ml (max)	0.02	4.6.6
Halogen Ion	Pass test	4.6.7
Free Bromine	Pass test	4.6.8
Cloud point, (max)	0° C	4.6.9

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3.3.2 Valves. Unless otherwise specified (6.2), replacement valve for defective valves in Government-furnished cylinder shall be supplier-furnished and shall be in accordance with Compressed Gas Association Connection No. 660. Each valve shall be equipped with a siphon tube to permit charging with nitrogen (6.1).

3.3.2.1 Thread Compound. When valves are installed, or removed and reinstalled, tape conforming to MIL-T-27730 shall be used. It shall be applied only to the male threads, except for the first two threads.

3.4 Capacity. Unless otherwise specified (6.2), cylinders shall be filled to the rated capacity. The weight of dibromodifluoromethane supplied shall be the difference between the filled weight and the unfilled tare weight of the cylinder.

3.5 Leakage. Cylinders and valves shall not leak after being filled.

3.6 Rejected Cylinders. Government-owned cylinders considered unsatisfactory for filling or refilling shall be processed as specified in the contract or purchase order (6.2). Other cylinders shall be disposed of by the contractor.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements (6.2).

4.2 Classification of Tests. The inspection and testing of the product shall be classified as quality conformance tests.

4.3 Inspection of the Product.

4.3.1 Lotting. A lot shall consist of one of the following:

4.3.1.1 The product produced in not more than 24 consecutive hours from a continuous process which is used to fill shipping cylinders directly from the process output. A continuous process shall be the production of product by continuous input of raw materials and output of finished product by one manufacturer in one plant with no change in manufacturing conditions or materials.

4.3.1.2 The product from individual runs of a batch process which is used to fill shipping cylinders directly from the process output. A batch process shall be the production of product by runs from single additions of raw materials which are mixed, reacted, and purified as necessary forming the product.

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4.3.1.3 The product from either or both the continuous and batch processes which is held in a single storage tank and subsequently withdrawn to fill shipping cylinders. The product shall be mixed until homogeneous at the time of withdrawal and shall not be added to while being withdrawn. After each addition to the storage tank, the contents shall constitute a separate lot.

4.3.2 Sampling. Unless otherwise specified herein, a sufficient quantity of dibromodifluoromethane to adequately perform all of the tests described under 4.6 shall be taken from filled cylinders in accordance to sampling plan in 4.3.3. The sample shall be kept cool (about 59°F) until delivered to the laboratory (6.2).

4.3.3 Sampling Plan. Unless otherwise specified herein, sampling for tests shall be from filled cylinders and shall be in accordance with 4.3.3.1.

4.3.3.1 Cylinders. The number of cylinders selected for sampling from each lot shall be in accordance with Table II. Also, the first and last cylinders to be filled within a given lot shall be sampled. Other samples shall be selected at random. The product from each cylinder sampled shall constitute a separate sample.

TABLE II
Sampling Plan for Test

Number of Cylinders in Lot	Number of Cylinders To Be Sampled
1-9	1
10-49	2
50-149	3
150 and over	4

4.4 Rejection. When any samples of the product tested as specified herein fails to conform to the requirements specified herein, the entire lot represented by the sample shall be rejected.

4.5 Test Conditions. The test conditions herein specified shall apply to the test methods in 4.6.

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4.5.1 Chemicals. Unless otherwise specified, all chemical tests shall be made with ACS specification reagent grade chemicals and distilled water.

4.5.2 Calibration. Test equipment shall be calibrated as applicable in accordance with MIL-C-45662.

4.5.3 Laboratory Conditions. The inspection laboratory shall be free from drafts and have a temperature no higher than 75°F (24°C). The dew point of the room shall be 54°F (12°C) or less.

4.6 Test Method.

4.6.1 Appearance and Color. Fill a 25- by 150-millimeter (mm) test tube to within 1-inch of the top with some of the thoroughly-mixed sample. Examine by transmitted light for evidence of suspended matter, sediment, and color.

4.6.2 Odor. Examine some of the sample for the characteristic odor of dibromodifluoromethane. Dip strips of heavy filter paper into the sample and allow to dry for 15 minutes. Examine for residual odor.

4.6.3 Specific Gravity. Determine the specific gravity by any suitable method accurate to within 0.002.

4.6.4 Index of Refraction. Determine the index of refraction at 59°F (15°C) with a standardized Abbe refractometer or other equally accurate instrument.

4.6.5 Distillation Range. The distillation shall be in accordance with ASTM D86 with the following variations. The neck of the Engler distillation flask shall be wrapped from the bulb to the top with rubber tubing which will serve as a cooling jacket. The tubing may be held in place with gummed tape. The American Society for Testing and Materials solvents distillation thermometer designation 37C-52 shall be used. The flask shall be heated with a micro burner. The graduate shall be tightly fitted to the condenser by means of a cork stopper of approximately the same outside diameter as the graduate. The cork shall be held in place on top of the graduate with tightly fitting Gooch flat style rubber tubing and fitted with a drying tube. When the flask is charged and the apparatus assembled, cool water shall be run through the rubber tubing jacket until a temperature of 59°F (15°C) is reached. The flask shall then be heated but the flow of cooling water continued throughout the distillation. If less than 96 milliliters (ml) are recovered, the distillation shall be repeated. The barometric pressure shall be ascertained and the distillation range corrected for variation of barometric pressure from the standard by the Sydney Young equation using the constant of 0.000125.

4.6.6 Nonvolatile Residue. Fill a tared 100-ml Goetz centrifuge tube to the 100-ml mark with the sample. Place the tube in a water bath at 86° to 90° F (30° to 32° C) until only 2 or 3 ml remain. Increase the bath temperature to 125° to 130°F (52° to 54°C) for 30 minutes. Dry the outside of the tube in a stream of dry air and weigh. Report the difference in weight as nonvolatile residue.

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4.6.7 Halogen Ion.

4.6.7.1 Preparation of Test Reagent.

4.6.7.1.1 Silver nitrate reagent - Dissolve 500 mg of ACS reagent grade silver nitrate weighed to nearest 10 mg in five ml of distilled water in a 100 ml volumetric flask. Make to volume with acetone.

4.6.7.1.2 Potassium bromide standard solution. Dissolve .119 grams of ACS reagent grade potassium bromide in a 100-ml volumetric with water and make to volume, 0.1 ml of the solution is equivalent to 80 micrograms of bromide.

4.6.7.2 Halide Test. Transfer 5 ml of the silver nitrate reagent to each of clean, clear, glass-stoppered test tubes or cylinders which are about 20 mm in diameter. Cool these 2 tubes and the dibromodifluoromethane sample to 15°C or lower. Add 5 ml (11.5 grams) of the dibromodifluoromethane sample to one tube, and add 0.1 ml of potassium bromide standard solution and 5 ml of acetone to the other tube. Shake to insure good mixing. Return both tubes to the cold bath. After 5 minutes, examine. The precipitate, opacity, or turbidity of the sample tube shall be less than that of the standard. This indicates less than 7 ppm of halide ion (as bromide) in the sample.

NOTE: A slight turbidity may develop from separation of water droplets. If there is any doubt, add 5 ml of acetone and shake well. A turbidity caused by water will disappear.

4.6.8 Free Bromine.

4.6.8.1 Preparation of Test Reagents.

4.6.8.1.1 Potassium Iodide Solution. Dissolve 4 grams of ACS reagent grade potassium iodide crystals in 100 ml of freshly boiled, distilled water. Special care must be taken to insure that the water used in the test is free of oxygen.

4.6.8.1.2 Sulfuric Acid, 1-4 Solution. Carefully add 5 ml of ACS reagent grade sulfuric acid to 20 ml of distilled water.

4.6.8.1.3 Starch Indicator Solution. Mix one gram of ACS reagent grade starch and 5 milligrams of mercuric iodide with enough cold, distilled water to make a thin paste; add 500 ml of boiling, distilled water. Boil for several minutes while stirring. Cool before use. Store in a glass-stoppered bottle.

4.6.8.2 Test Procedure. Transfer 10 ml (23 grams) of cooled (15°C or below) dibromodifluoromethane to clean, glass-stoppered test tube (20 mm diameter of about 50 ml capacity - glass stoppered, 50 ml, graduated cylinder or 50 ml, glass-stoppered, conical bottom centrifuge tube could be used). Add 5 ml of cooled potassium iodide solution. Shake vigorously to insure good mixing of the two layers. Add five drops of sulfuric acid solution

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and one ml of starch indicator. Allow the sample to stand for five minutes at 15°C. The water layer (upper) should have no orange or brown coloration present.

NOTE: A slight, yellow cast is equivalent to one part per million of free bromine and indicates a satisfactorily low concentration of free bromide.

4.6.9 Cloud Point. Add 35 to 40 ml of the sample to a 25-mm outside diameter by 150-mm long pyrex test tube fitted with a 2-hole stopper containing a glass stirrer and a cloud and pour thermometer (ASTM 5C). Immerse in a dry ice-cooling bath or equivalent, cooling at a rate not in excess of 2°C per minute, and stir gently as the liquid cools. Record the temperature at which the liquid becomes cloudy or separation of water begins.

4.7 Inspection of Preparation for Delivery. The preservation, packing, packaging, and marking for shipment and storage of the material shall be inspected to determine compliance with the requirements of Section 5 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Packing

5.1.1 Level A. Unless otherwise specified, the dibromodifluoromethane shall be contained in government-furnished cylinders in accordance with 49 CFR 171-190 and equipped with valves with outlet connection No. 660 in accordance with Compressed Gas Association Publication V-1. When specified (6.2), cylinders shall be furnished by the supplier and shall be in accordance with DOT regulations and shall be equipped with valve with outlet connection No. 660. Cylinders shall be standard gas cylinders conforming with DOT Specification 3A300, 3B300, 4B300, or 4BA300.

5.1.2 Level C. Dibromodifluoromethane shall be contained in standard gas cylinders conforming with DOT Specifications 3B300, 4B300, or 4BA300.

5.2 Palletization. When specified (6.2) cylinders shall be palletized in accordance with MIL-STD-147.

5.3 Cylinders Inspection and Cleaning. Prior to filling with dibromodifluoromethane each cylinder shall be inspected. The cylinder interior shall be clean and free of contaminants. (6.2)

5.4 Marking. The cylinders shall be marked in accordance with 49CFR171-190.

5.4.1 Military Agencies. Unless otherwise specified, in addition to the marking indicated in 5.4, the cylinders shall be marked in accordance with MIL-STD-129 and color code in MIL-STD-101 (6.2).

5.5 Identification Tag. Unless otherwise specified in the contract or purchase order, an identification tag impervious to climatic conditions

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shall be wired to the outlet port of each cylinder and shall contain the following information: product name, specification number with revision letter, federal stock number, quantity, name of manufacturer, name of contractor (if different from manufacturer), date of manufacture and lot identification number.

6. NOTES

6.1 Intended Use. The dibromodifluoromethane covered by this specification is intended for use as a fire extinguisher fluid. When used to charge fire extinguishers, gas cylinders will be pressurized with nitrogen to 300 pounds per square inch.

6.2 Ordering Data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

6.2.1 Title, number, and date of this specification.

6.2.2 Method of shipment, type and capacity of cylinders (3.4 and 5.1).

6.2.3 Quantity by weight in pounds (avoirdupois) (6.4).

6.2.4 When inspection requirements are to be performed by other than the supplier (4.1).

6.2.5 When sampling is other than specified (4.3.2).

6.2.6 When cleaning and repair schedule is required for leased or government-owned cylinders (3.3, 5.3 and 6.3)

6.2.7 When instructions for disposition of rejected cylinders is required (3.6).

6.2.8 When palletization of cylinders is required (5.2).

6.2.9 When identification tag is to be other than as specified (5.5).

6.2.10 When valves are to be other than as specified (3.3.2).

6.2.11 When supplier-furnished cylinders are required (5.1).

6.2.12 When the cylinder color code is to be waived (5.4.1).

6.3 Performance of Services. The gas supplier should furnish at no additional cost all services which are required at each and every filling of a cylinder to comply with applicable regulations and normal standard practice. Such services would include, but not be limited to, all inspection, testing, evacuation, and handling required for the gas supplied.

6.3.1 A schedule of allowable fees should be specified by the purchaser for the gas supplier's performance of services such as the replacement of valves, valve parts, and cylinder caps, hydrostatic testing, cleaning,

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painting, color coding, marking, and handling of unserviceable cylinders as required. All materials and components for these services may be furnished by the gas supplier.

6.4 Basis of Purchase. Dibromodifluoromethane should be purchased by weight in DOT approved cylinders in the liquid phase at 70°F. (6.2)

6.5 Pollution Control. US Public Laws dictate increased effort to improve air, land, and water pollution control of toxic propellant vapor, leaks, spills, and disposal during all phases of manufacture, transfer, storage, and transportation operations. The manufacturer/supplier is enjoined to approach the appropriate pollution control districts to mutually resolve all problem areas, and to develop adequate control and disposal methods for situations which are likely to develop in any of the phases.

MILITARY INTEREST:

Custodians:

Army - MU

Navy - AS

Air Force - 68

Preparing Activity:

Air Force - 68

Project No. 6830-0070

Review Activity:

Army - ME

Navy - AS

DGSC - GS

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