

Submitted for recognition as an American National Standard

Anti-Icing and Deicing-Defrosting Fluids

FSC 6850

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1. SCOPE:

1.1 Scope:

This specification covers the requirements for two types of anti-icing and deicing-defrosting fluids for use on aircraft.

1.2 Classification:

The anti-icing and deicing-defrosting fluids covered by this specification shall be of the following types, as specified (see 6.2.1):

Type I - Propylene Glycol base fluid with Inhibitor

Type II - Ethylene Glycol base fluid with Inhibitor

2. APPLICABLE DOCUMENTS:

2.1 Government documents:

- 2.1.1 Specifications and standards: Unless otherwise specified, the following specifications and standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

QQ-A-250/4	-	Aluminum Alloy 2024, Plate and Sheet.
QQ-A-250/5	-	Aluminum Alloy Alclad 2024, Plate and Sheet.
QQ-A-250/13	-	Aluminum Alloy Alclad 7075, Plate and Sheet.
QQ-M-44	-	Magnesium Alloy Plate and Sheet (AZ31B).
QQ-P-416	-	Plating, Cadmium (Electrodeposited).
PPP-D-729	-	Drum, Shipping and Storage, Steel, 55-Gallon (208 Liters).
PPP-P-704	-	Pail, Metal, (Shipping, Steel, 1 Through 12 Gallon).

MILITARY

MIL-M-3171	-	Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion on.
MIL-S-7952	-	Steel, Sheet and Strip, Uncoated, Carbon (1020 and 1025) (Aircraft Quality).
MIL-A-8625	-	Anodic Coatings, for Aluminum and Aluminum Alloys.
MIL-T-9046	-	Titanium and Titanium Alloy, Sheet, Strip and Plate.
MIL-D-43703	-	Drums, Shipping and Storage, Molded Polyethylene.

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2.1.1 (Continued):

STANDARDS

FEDERAL

FED-STD-313 - Material Safety Data Sheets, Preparation and Submission of.

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-129 - Marking for Shipment and Storage.
MIL-STD-147 - Palletized Unit Loads.
MIL-STD-290 - Packaging of Petroleum and Related Products

2.1.2 Other Government documents, drawings, and publications: The following other Government documents, drawings, and publications form a part of this specification to the extent specified herein.

PUBLICATIONS

CODE OF FEDERAL REGULATIONS

49 CFR - Transportation.

(Application for copies should be addressed to the superintendent of Documents, Government Printing Office, Washington, DC 20402.)

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the acquisition 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. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM D 92 - Flash and Fire Points by Cleveland Open Cup.
ASTM D 97 - Pour Point of Petroleum Oils.
ASTM D 1122 - Specific Gravity of Engine Antifreezes by the Hydrometer.
ASTM D 1123 - Water in Engine Coolant Concentrate by the Iodine Reagent Method.
ASTM D 1176 - Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes.

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2.2 (Continued):

- ASTM D 1287 - pH of Engine Antifreezes, Antirusts and Coolants.
- ASTM D 3951 - Commercial Packaging.
- ASTM D 4057 - Manual Sampling of Petroleum and Petroleum Products.
- ASTM D 4177 - Automatic Sampling of Petroleum and Petroleum Products.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI)

- ANSI Z129.1 - American National Standard for the Precautionary Labeling of Hazardous Industrial Chemicals.

(Application for copies should be addressed to the American National Standards Institute, 1430 Broadway, New York, NY 10018.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence:

In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS:

3.1 Material:

The material shall be a buffered blend of 1,2 glycols, (a wetting agent and a dehydrolysis inhibitor. The fluid shall be homogenous and completely miscible with water.

3.2 Composition:

This specification covers two different fluid compositions with nearly identical deicing abilities. Type I shall be formulated as specified in Table I, using only propylene glycol to achieve the required 1,2-glycol content. Type II shall be formulated as specified in Table I, using a mixture of 1,2-glycols containing three parts of ethylene glycol to one part propylene glycol (by volume).

3.3 Chemical and physical properties:

The chemical and physical properties of the finished anti-icing and deicing-defrosting fluid shall conform to the requirements specified in Tables I and II when tested as specified in 4.5.

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3.4 Material safety data sheets:

Material safety data sheets shall be prepared and submitted in accordance with FED-STD-313. The anti-icing and deicing-defrosting fluids shall have no adverse effect on the health of personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the contracting activity to the appropriate departmental medical service who will act as an advisor to the contracting agency (see 4.6 and 6.2.1h).

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for inspection:

Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor 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.

4.2 Certificate of compliance:

Where certificates of compliance are submitted, the Government reserves the right to check test such items to determine the validity of the certification.

4.3 Inspection conditions:

Unless otherwise specified, all inspections shall be performed in accordance with the test conditions specified in the applicable paragraphs in this specification.

4.4 Quality conformance inspection:

Quality conformance inspection shall consist of the tests specified in Table III, and the examinations specified in 4.4.4.

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4.4.1 Reduced testing and examination: Unless otherwise specified (see 6.2.1), reduced testing and examination may be performed on the lot if the contractor has previously fully tested and furnished satisfactory material under this specification. The contractor shall certify that the lot of material furnished is manufactured under the same conditions using the same formulation and raw materials as previously tested and conforms to all requirements of this specification. The following minimum tests and examination shall be performed:

Visual examination	4.4.4
Water	4.5.2
Corrosiveness	4.5.8
Pour point	4.5.10 (undiluted material only)
Specific gravity	4.5.11

If during reduced testing, a lot of material fails to meet any requirement of the specification, normal testing shall be reinstated and continued until two successive lots meet all requirements of the specification, after which reduced testing may be resumed.

4.4.2 Lot information:

4.4.2.1 Bulk lot: A bulk lot is an indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container; or manufactured in a single plant run (not exceeding 24 hours) through the same processing equipment, with no change in ingredient material.

4.4.2.2 Packaged lot: A packaged lot is an indefinite number of unit containers of identical size and type, offered for acceptance, and filled with homogenous mixture of material from one isolated container; or filled with a homogenous mixture of material manufactured in a single plant run (not exceeding 24 hours) through the same processing equipment, with no change in ingredient material.

4.4.3 Sampling:

4.4.3.1 For examination of filled containers: Random samples of filled unit containers and a sample of shipping containers fully prepared for delivery shall be selected from each lot of fluid in accordance with MIL-STD-105, Inspection Level II and an Acceptable Quality Level (AQL) of 2.5 percent defective.

4.4.3.2 For tests: Samples for testing shall be randomly selected in accordance with Table IV. A 2 liter sample for tests shall be selected from each lot in accordance with ASTM D 4057 or ASTM D 4177. The sample shall be subjected to all the applicable quality conformance inspections. The lot shall be unacceptable if the sample fails to comply with any of the requirements for quality conformance inspections.

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4.4.4 Examination of filled containers: Samples taken in accordance with 4.4.3.1 shall be examined for compliance with MIL-STD-290 with regard to fill, closure, sealing, leakage, packaging, packing and marking requirements. Any container having one or more defects or under the required fill shall be rejected. If the number of defective or under filled containers exceeds the acceptance number for the appropriate plan of MIL-STD-105, the lot represented by the samples shall be rejected.

4.5 Test methods:

4.5.1 1, 2-glycol content:

4.5.1.1 Reagents: The following reagents shall be used to determine the 1, 2-glycol content of the anti-icing and deicing-defrosting fluid:

- a. 0.4N sodium periodate solution (prepared by weighing exactly 10.6 grams of metasodium periodate (NaIO_4) and dissolving in sufficient distilled water to make 1 liter of solution).
- b. 0.2N sodium thiosulfate solution.
- c. 30 percent potassium iodide solution.
- d. Starch solution.
- e. 20 percent sulfuric acid solution.

4.5.1.2 Procedure: A quantity of 3.5 to 4.0 grams of the anti-icing and deicing-defrosting fluid shall be weighed by difference in a 250 milliliter (ml) volumetric flask. The mass shall be determined to the nearest 0.1 milligram (mg). The flask shall then be filled to the 250 ml mark with distilled water and the solution mixed thoroughly. A 10 ml aliquot of the solution shall be transferred to a 500 ml iodine flask. Fifty ml of the 0.4N sodium periodate solution shall then be added, the flask stoppered and the mixture allowed to react. Two blanks containing the reagents only shall be treated in the same manner as the sample aliquot. After 1 hour, 150 ml of distilled water shall be added to the sample aliquot mixture, followed by the addition of 20 ml of the 20 percent sulfuric acid solution and 40 ml of the 30 percent potassium iodide solution. (The potassium iodide solution should be added by pouring it around the stopper thus forming a liquid seal between the stopper and the neck of the flask to absorb any liberated iodine vapors.) The sample mixture shall be allowed to react for 10 minutes, agitating frequently, after which the neck and sides of the flask shall be rinsed with distilled water from a wash bottle. The sample and the blanks shall then be titrated to a pale yellow iodine color with standardized 0.2N sodium thiosulfate. Two ml of starch solution shall be added and the solutions titrated to the disappearance of the blue color. The blanks should each require 95 to 100 ml of the thiosulfate and the sample mixtures 78 to 90 ml. The 1, 2-glycol content shall be computed as follows:

$$\text{Percent 1, 2-glycol content} = \frac{M \times 25 \times (B - S) \times N}{W} \times 100$$

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4.5.1.2 (Continued):

where:

B = ml thiosulfate solution required by the blank (average of two blank determinations).

S = ml of thiosulfate solution required by the sample aliquot.

N = normality of the thiosulfate solution.

W = mass of the sample in grams.

M (for Type I) = 0.0381

M (for Type II) = 0.0325

4.5.2 Water content: Water content shall be determined in accordance with ASTM D 1123.

4.5.3 Phosphate content:

4.5.3.1 Preparation of reagent: Fifty grams of crystalline magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and 100 grams of ammonium chloride shall be dissolved in 500 ml of water. Approximately 10 ml of concentrated aqueous ammonia shall be added and the resultant solution allowed to stand overnight. It shall be filtered if a precipitate appears. The solution shall then be made slightly acid with hydrochloric acid after which it shall be diluted to 1 liter and placed in a glass-stoppered bottle.

4.5.3.2 Procedure: A 20 gram sample of the anti-icing and deicing-defrosting fluid, weighed to the nearest 0.01 gram, shall be diluted with 100 ml of distilled water. Ten ml of a mixture of equal parts by volume of concentrated hydrochloric acid and water and 30 ml of the reagent prepared in accordance with 4.5.3.1 shall be added to the diluted sample. Concentrated aqueous ammonia shall then be added until the solution is neutral to methyl red indicator. The solution shall be stirred for 5 minutes and as stirring is continued, an additional 15 ml of aqueous ammonia shall be added. The solution shall then be allowed to stand for 4 hours or longer after which it shall be filtered and the magnesium pyrophosphate precipitate ignited in a weighed platinum crucible at 1000°C to 1050°C (1832°F to 1922°F) until constant weight is reached. The dibasic potassium phosphate content shall be computed as follows:

$$\text{Percent dibasic potassium phosphate content} = \frac{1.565 \times M}{W} \times 100$$

where:

M = mass of the magnesium pyrophosphate precipitate in grams.

W = mass of the sample in grams.

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4.5.4 Sodium di-(2-ethylhexyl) sulfosuccinate content:

4.5.4.1 Reagents: The following reagents shall be used in the determination of the sodium di-(2-ethylhexyl) sulfosuccinate content of the anti-icing and deicing-defrosting fluid:

- a. Standard sodium di-(2-ethylhexyl) sulfosuccinate solution prepared by transferring 0.8 gram of pure sodium di-(2-ethylhexyl) sulfosuccinate, weighed to the nearest 0.1 mg, to a 250 ml glass-stoppered volumetric flask and diluting to the mark with distilled water. This solution shall be agitated (a mechanical shaker may be used) until complete solution is effected. Solutions over 2 weeks old shall be discarded.
- b. Methylene blue chloride solution prepared by dissolving 0.050 ± 0.005 gram of chemically pure (CP) methylene blue chloride in 1 liter of distilled water. Carefully add 10 ml of concentrated CP sulfuric acid and 50 grams of anhydrous sodium sulfate. Mix thoroughly to effect complete solution.
- c. 0.005N cetyl pyridinium bromide prepared by dissolving 2.5 grams of cetyl pyridinium bromide in 1 liter of distilled water and filtering the solution.

4.5.4.2 Procedure: One hundred and fifty grams of anti-icing and deicing-defrosting fluid shall be weighed by difference in a 250 ml volumetric flask. The mass shall be determined to the nearest 0.1 gram. The flask shall be filled to the 250 ml mark with distilled water and the solution mixed thoroughly. A 10 ml aliquot of the anti-icing fluid solution and a 10 ml aliquot of the standard sodium di-(2-ethylhexyl) sulfosuccinate solution shall be transferred to separate 100 ml oil sample bottles. Twenty ml of the methylene blue chloride solution and 25 ml of CP chloroform shall be added to each bottle. The bottles shall be stoppered and shaken vigorously for several seconds. The contents of each bottle shall then be titrated with 0.005N cetyl pyridinium bromide in 1.0 ml portions, with vigorous shaking of the bottle after each addition, until the blue color in the chloroform (lower) layer begins to migrate to the upper layer. The titration shall then be continued drop by drop until the color intensities of both layers match exactly when viewed in transmitted light. The sodium di-(2-ethylhexyl) sulfosuccinate content shall be computed as follows:

$$\text{Percent sodium di-(2-ethylhexyl) sulfosuccinate content} = \frac{W_1 \times T_2}{W_2 \times T_1} \times 100$$

where:

W_1 = mass of sodium di-(2-ethylhexyl) sulfosuccinate used for standard solution.

W_2 = mass of anti-icing and deicing-defrosting fluid used for solution.

T_1 = ml of cetyl pyridinium bromide solution required by the standard solution aliquot.

T_2 = ml of cetyl pyridinium bromide solution required by the anti-icing fluid solution.

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- 4.5.5 Sodium salt of tolyltriazole content: A contractor's notarized certificate of compliance signed by a responsible official of the company is acceptable for the content of the sodium salt of tolyltriazole (see 4.2).
- 4.5.6 Alkaline earths: Ten ml of the anti-icing and deicing-defrosting fluid shall be placed in a 30 ml test tube. The fluid shall be made alkaline to litmus with ammonium hydroxide solution after which shall be added 5 ml of reagent grade ethyl alcohol (95 percent). Five ml of 0.5N ammonium oxalate shall then be added and the solution visually examined for evidence of precipitation. No immediate precipitate shall form.
- 4.5.7 Chlorides: Place 10 ml of the anti-icing and deicing-defrosting fluid in a 30 ml test tube. Add 10 ml of distilled water and mix. Add 10 drops of concentrated nitric acid, agitate the mixture, and add 2 ml of a 3 percent aqueous solution of silver nitrate. Filter and discard the filtrate. Wash the residue with concentrated ammonium hydroxide, recover the filtrate (washings) and acidify with concentrated nitric acid. No precipitate indicates the absence of chlorides. A slight turbidity is allowed.
- 4.5.8 Corrosiveness:
- 4.5.8.1 Test panels: Test specimens shall consist of 1 by 6 inch panels of the following metals: aluminum alloy conforming to QQ-A-250/4 and anodized to conform to MIL-A-8625, Type I or II; clad aluminum alloy conforming to QQ-A-250/13; clad aluminum alloy conforming to QQ-A-250/5; magnesium alloy conforming to QQ-M-44, cleaned and treated in accordance with MIL-M-3171; Type III titanium alloy 6Al-4V, conforming to MIL-T-9046; polished aluminum alloy conforming to QQ-A-250/4; and steel conforming to MIL-S-7952 cadmium plated to conform to QQ-P-416. The panels, prior to being subjected to test, shall be cleaned by boiling for 1 minute in CP isopropyl alcohol and 1 minute in CP mineral spirits.
- 4.5.8.2 Procedure: Five hundred ml of a 20 percent by volume solution of the anti-icing and deicing-defrosting fluid in distilled water shall be placed in a 1000 ml tall-form beaker. The panels described above shall then be placed on end in the solution, without touching each other, and allowed to stand for 24 hours at a temperature of $25^{\circ} \pm 5^{\circ}\text{C}$ ($77^{\circ} \pm 9^{\circ}\text{F}$). Wetting of the upper portion of the panels shall be avoided. After the 24 hour immersion, the panels shall be removed and allowed to air dry in an upright position for 3 hours. The panels shall then be re-immersed in the solution for 1 minute, removed and allowed to air dry for 24 hours. The panels shall be rinsed and then visually examined for corrosion by comparing the unexposed surfaces of the panels with the surfaces previously immersed.
- 4.5.9 Flash point: The flash point shall be determined in accordance with ASTM D 92, except that the fire point need not be determined.
- 4.5.10 Pour point: The pour point of the fluid and of a 70 percent aqueous solution of the fluid, prepared in accordance with ASTM D 1176, shall be determined in accordance with ASTM D 97, except that the cloud point need not be determined.

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- 4.5.11 Specific gravity: The specific gravity of the fluid shall be determined in accordance with ASTM D 1122.
- 4.5.12 Effect on acrylics: The fluid shall be tested for compatibility with Type A and Type C acrylic specimens in accordance with ASTM F484 with the following exceptions. Type A specimens shall be stressed to an outer fiber stress of 2000 psi (13790 KPa), and Type C specimens shall be stressed to an outer fiber stress of 3000 psi (20685 KPa).
- 4.5.13 pH value: A 50 percent aqueous solution of the fluid shall be prepared in accordance with ASTM D 1176. The pH value of this solution shall be determined using the apparatus and procedure specified in ASTM D 1287.
- 4.6 Submission of material safety data sheets:
- The contractor shall furnish to the contracting activity the toxicological data and formulations required to evaluate the safety of the material for the proposed use through the submission of the material safety data sheets detailed in FED-STD-313.
5. PACKAGING:
- 5.1 Preservation:
- Preservation shall be Level A or Level C, as specified (see 6.2.1).
- 5.1.1 Level A: The anti-icing and deicing-defrosting fluid shall be furnished in 5-gallon or 55-gallon containers as specified (see 6.2.1). The 5-gallon container shall conform to Type I, Class 3 (screw cap closure) of PPP-P-704, or when specified (see 6.2.1), to MIL-D-43703, Size 1, Molded Polyethylene Container. The 55-gallon container shall be a tight-head drum conforming to Type I or Type II of PPP-D-729 or, when specified (see 6.2.1), to MIL-D-43703, Size 4, Molded Polyethylene Container.
- 5.1.2 Level C: The anti-icing and deicing-defrosting fluid shall be preserved in specified quantities in accordance with ASTM D 3951, utilizing containers required by the Code of Federal Regulations, Title 49.
- 5.2 Packing:
- Packing shall be Level A, B, or C, as specified (see 6.2.1).
- 5.2.1 Levels A and B: The anti-icing and deicing-defrosting fluid, preserved in accordance with 5.1.1, does not require any overpacking. Fluid preserved in 5-gallon containers in accordance with 5.1.1 shall be palletized in accordance with MIL-STD-147.
- 5.2.2 Level C: The anti-icing and deicing-defrosting fluid, preserved as specified in 5.1.2, shall be packed in accordance with ASTM D 3951. The containers shall conform to the rules and regulations of the Code of Federal Regulations, Title 49.

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5.3 Marking:

Marking shall be in accordance with MIL-STD-129 and shall include the additional special markings requirements as specified by the acquiring activity. Unit and intermediate containers, including unit containers that serve as shipping containers, shall be marked with the applicable precautionary marking detailed in ANSI Z129.1. In addition to any special markings required by the contract, each container shall be marked in accordance with the following:

CAUTION

AVOID CONTACT WITH SKIN AND EYES.
DO NOT TAKE INTERNALLY.
AVOID BREATHING VAPOR.
KEEP AWAY FROM FIRE, SPARKS AND
POWERFUL OXIDIZING MATERIALS.
STORE IN A COOL PLACE.
KEEP CONTAINER CLOSED.
MINIMUM FLASH POINT 101.7°C (215°F).

6. NOTES:

6.1 Intended use:

The anti-icing and deicing-defrosting fluids covered by this specification are intended for use in removing frost and ice from the surface of parked aircraft and to prevent the formation of ice and frost on such surfaces. Types I and II are nearly identical in deicing ability, however, type I has been shown to be less toxic to laboratory animals.

6.2 Ordering data:

6.2.1 Acquisition requirements: Acquisition documents should specify the following:

- a. Title, number and date of this specification.
- b. Type required (see 1.2).
- c. Type and size of container (see 5.1.1).
- d. Quantity in gallons.
- e. Level of preservation and packing required (see 5.1 and 5.2).
- f. Reduced testing and examination when specified (see 4.4.1).
- g. When a MIL-D-43073 container is required.
- h. Specify DAR Clauses 7-104.98 and 1-323.2.