

# **AEROSPACE MATERIAL SPECIFICATION**

**AMS 1428E** 

**AUG 1997** Issued Revised **JUN 2006** 

Superseding AMS 1428D

Fluid, Aircraft Deicing/Anti-Icing, Non-Newtonian (Pseudoplastic), SAE Types II, III, and IV

### **RATIONALE**

This revision of the document reflects the following changes. Inclusion of Cold Storage Stability test (3.2.2.10) and an alternate procedure for Exposure to Dry Air testing (3.2.2.2.2). Bright yellow was added as the color for SAE Type III fluid (Table 1). Highest Viscosity Dilution (3.2.5.3.1) and Preproduction Tests samples (4.2.3.1 and 4.2.3.2) were clarified with more detail. Annexes A, B, and C were replaced with references to separate pieces of documentation, SAE AS 5901, AS 5900 and AS 5900, respectively. High and Low Water Spray Endurance Time tests (HWSET and LWSET) were removed (formerly 3.2.4.2 and 3.2.4.3). Accelerated Aging Thermal Stability (3.2.2.1), Shear Stability (3.2.2.7.1), and Hydrogen Embrittlement (3.3.2.5) and were changed to be consistent with AMS 1424 test practices POFOFAT

#### 1 SCOPE

### Form

This specification covers three types of deicing/anti-icing material, each in the form of a non-Newtonian fluid.

### Classification

Deicing/anti-icing fluids covered by this specification are classified as follows:

# TABLE 1 - CLASSIFICATION

Type II II High Speed Ramp Test Water white/pale straw Type III III Low Speed Ramp Test Bright Yellow	Fluid	Anti-Icing Performance	Aerodynamic Performance	
Type II II High Speed Ramp Test Water white/pale straw Type III III Low Speed Ramp Test Bright Yellow	SAE	Use Table 3	Use SAE AS 5900	Color
Type III III Low Speed Ramp Test Bright Yellow	Type	(3.2.4.1)	(see Note)	Applicable if Requested
	Type II	II 0/4.	High Speed Ramp Test	Water white/pale straw
Type IV IV Figh Speed Ramp Test Green	Type III	III C	Low Speed Ramp Test	Bright Yellow
Type TV Tright Speed Namp Test Steeli	Type IV	IV	High Speed Ramp Test	Green

As per 3.2.5.2, any fluid can be tested in accordance with the AS5900 high speed ramp test and/or low speed Note: ramp test to demonstrate acceptable aerodynamic performance. The designations in Table 1 are for fluid classification purposes only. Consult the fluid manufacturer for complete aerodynamic test data.

Consult aircraft manufacturer's Aircraft Operations Manual, Aircraft Maintenance Manual and service letters to determine any restrictions relating to the use of deicing/anti-icing fluids meeting this specification for the type and model of aircraft being treated. See also latest edition of ARP4737.

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- 1.1.2 A fluid defined as non-Newtonian exhibits a different apparent viscosity value when tested at the same temperature, using the same viscometer and spindle in a predetermined volume when the only variant is the rotational speed of the spindle. Typically, the non-Newtonian fluid shear stress is not in direct proportion to its rate of flow.
- 1.1.3 A fluid described as pseudoplastic shall exhibit reduced apparent viscosity values as the spindle speed is increased, and revert to original flow behavior when the shear stress is removed. This shall be demonstrable at the temperatures listed in 3.2.3.1.1.

# 1.2 Application

This fluid may be used as follows:

- a. Unheated and undiluted, or diluted for anti-icing
- b. Heated and undiluted for deicing/anti-icing as a one step process
- c. Diluted with water and heated for deicing/anti-icing, as a one step process
- Diluted with water and heated as the deicing stage in a two-step process, usually when used with the unheated and undiluted fluid as step two
- 1.2.1 Consult aircraft manufacturer's Aircraft Operations Manual, Aircraft Maintenance Manual and service letters to determine any restrictions relating to the use of deicing/anti-icing fluids for type and model of aircraft being treated. Refer also to ARP4737.

### 1.3 Precautions

- 1.3.1 The lowest operational use temperature (LOUT) for a SAE Type II, III, and IV fluid shall be determined for the neat (undiluted) fluid or for each intended use dilution and is the lowest temperature at which the fluid has been tested and certified as acceptable in accordance with the appropriate aerodynamic acceptance test (3.2.5) while still maintaining the 7 °C (13 °F) freezing point temperature buffer (see ARP4737). The fluid manufacturers' literature shall clearly state the LOUT for the neat fluid and intended use dilutions.
- 1.3.2 The deicing/anti-icing formulation may be mildly toxic and contact with human skin and eyes should be avoided. Prolonged exposure to concentrations of vapor or windborne mists should be avoided. Consult the fluid manufacturers' Material Safety Data Sheet for further information.
- 1.3.3 Caution should be exercised in the use of glycol-water deicing/anti-icing solutions in and around electrical/electronic circuitry with noble metal coated wiring or terminals which could make contact with the fluid. Exothermic reactions, which may result in fire have been reported. This may occur where defectively insulated wires, switches, or circuit breakers carrying direct current are encountered. Fluids based on glycol shall contain an inhibitor to minimize this potential fire hazard.
- 1.3.4 A fluid meeting this specification is unique to each manufacturer and may be adversely affected by mixing with other aircraft deicing/anti-icing fluids.
- 1.3.5 Slippery conditions may exist on the ground, or equipment following the deicing/anti-icing procedure.

### 1.4 Safety - Hazardous Materials

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

# 2. APPLICABLE DOCUMENTS

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been cancelled and no superseding document has been specified, the last published issue of that document shall apply.

# 2.1 SAE Publications

AMS 2470

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), www.sae.org.

Anodic Treatment of Aluminum Alloys Chrome Acid Process

AIVIS	2470	Allouic Treatment of Aluminum Alloys, Chrome Acid Process
AMS	2475	Protective Treatments, Magnesium Alloys
AMS	2825	Material Safety Data Sheets
AMS	4037	Aluminum Alloy Sheet and Plate, 4.4Cu - 1.5Mg - 0.60Mn (2024T3 Flat Sheet, T351 Plate), Solution Heat Treated
AMS	4041	Aluminum Alloy Alclad Sheet and Plate 4.4Cu - 1.5Mg - 0.60Mn Alclad 2024 and 1-1/2% Alclad 2024, -T3 Flat Sheet; 1-1/2% Alclad 2024-T351 Plate
AMS	4049	Aluminum Alloy, Sheet and Plate, Alclad, 5.6Zn - 2.5Mg - 9.6Cu - 0.23Cr (Alclad 7075; -T6 Sheet, T651 Plate), Solution and Precipitation Heat Treated
AMS	4376	Plate, Magnesium Alloy, 3.0Al - 1.0Zn - 0.20Mn (AZ31B-H26), Cold Rolled and Partially Annealed
AMS	4911	Titanium Alloy, Sheet, Strip, and Plate, 6Al - 4V, Annealed
AMS	4916	Titanium Alloy Sheet, Strip, and Plate, 8AI - 1Mo-1V, Duplex Annealed
AMS	5045	Steel Sheet and Strip, 0.25 Carbon, Maximum, Hard Temper
AMS	5886	Alloy, Corrosion and Heat Resistant, Bars, Forgings, and Rings, 50Ni - 20Cr - 20Co - 5.8Mo - 2.2Ti - 0.45Al, Consumable Electrode or Vacuum Induction Melted 2100 °F (1149 °C) Solution Heat Treated
AMS-	P-83310	Plastic Sheet, Polycarbonate, Transparent
AS 59	900	Standard Test Method for Aerodynamic Acceptance for SAE AMS 1424 and SAE AMS 1428 Aircraft Deicing/Anti-icing Fluids
AS 59	901	Water Spray and High Humidity Endurance Test Methods for SAE AMS 1424 and SAE AMS 1428 Aircraft Deicing/Anti-icing Fluids
ARP 4	4737	Aircraft Deicing/Anti-icing Methods
ARP :		Endurance Time Tests for Aircraft Deicing/Anti-icing Fluids SAE Types II, III and IV
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# 2.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM C 672	Scaling Resistance of Concrete Surfaces Exposed to De-icing Chemicals
ASTM D 93	Flash Point by Pensky-Martens Closed Cup Tester
ASTM D 891	Specific Gravity, Apparent, of Liquid Industrial Chemicals
ASTM D 1177	Freezing Point of Aqueous Engine Coolants
ASTM D 1193	Reagent Water
ASTM D 1331	Surface and Interfacial Tension of Solutions of Surface-Active Agents
ASTM D 1568	Sampling and Chemical Analysis of Alkylbenzene Sulfonates
ASTM D 1747	Refractive Index of Viscous Materials
ASTM D 2196	Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield Type) Viscometer
ASTM D 3278	Flash Point of Liquids by Small Scale Closed Cup Apparatus
ASTM D 4052	Density and Relative Density of Liquids by Digital Density Meter
ASTM D 4177	Automatic Sampling of Petroleum and Petroleum Products
ASTM E 70	pH of Aqueous Solutions with the Glass Electrode
ASTM F 483	Total Immersion Corrosion Test for Aircraft Maintenance Chemicals
ASTM F 484	Stress Crazing of Acrylic Plastics in Contact with Liquid or Semi-Liquid Compounds

Effects of Cleaners on Unpainted Aircraft Surfaces
Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces
Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments
Stress Corrosion of Titanium Alloys by Aircraft Engine Cleaning Materials
Preparing Aircraft Cleaning Compounds, Liquid Type, Temperature-Sensitive, or Solvent-Based,
for Storage Stability Testing
Sandwich Corrosion Test
Corrosion of Low-Embrittling Cadmium Plate by Aircraft Maintenance Chemicals

### 2.3 U.S. Government Publications

Available from the Document Automation and Production Service (DAPS), Building 4/D, 700 Robbins Avenue, Philadelphia, PA 19111-5094, Tel: 215-697-6257, http://assist.daps.dla.mil/quicksearch/.

MIL-PRF-25690 Plastic, Sheets and Formed Parts, Modified Acrylic Base, Monolithic, Crack Propagation Resistant

### 2.4 APHA Publications

Available from American Public Health Association, 1015 Fifteenth Street, NW, Washington, DC 20005 or www.apha.org.

Standard Methods for the Examination of Water and Waste Water

### 2.5 OECD Publications

Available from OECD: 2, rue André Pascal, Cedex 16, 75016 Paris FRANCE or www.oecd.org.

**OECD Guidelines for Testing Chemicals** 

### 3. TECHNICAL REQUIREMENTS

# 3.1 Material

The composition of the fluid shall be optional with the manufacturer and shall be based on freezing point depressants with additives, such that the finished product shall meet the requirements of this specification.

# 3.1.1 Non-Glycol Based Fluid

A fluid based on non-glycol freezing point depressants shall be tested as follows: Two pieces of AMS 5886 bar, 35 mm diameter and 15 mm long shalt have one end of each machined flat. An 8.5 mm drill shall then be used to drill a centered hole 6.5 mm deep in one end to allow the milling of a cup shaped depression. A 12.5 mm bull nosed end mill shall be used to open up the drilled hole to produce a 7 mm deep depression. The cup shall then be finished by improving the surface with 600, 180, and 6 micrometers diamond paste. The cups shall be cleaned and degreased using a suitable solvent and allowed to dry. One cup shall be filled with the candidate test fluid, the other with water conforming to ASTM D 1193 Type IV, both test pieces shall then be placed in an oven at 105 °C  $\pm$  2 (221 °F  $\pm$  4). The fluid shall then be allowed to evaporate by progressive increase in oven temperature at the rate of 10 °C (18 °F) per minute to a final temperature of 250 °C  $\pm$  5 (482 °F  $\pm$  9) where they shall be maintained for 15 minutes  $\pm$  1. Test pieces shall then be transferred to an air furnace set at 1040 °C  $\pm$  10 (1904 °F  $\pm$  18) and maintained for 2 hours  $\pm$  5 minutes.

Test pieces shall be removed and allowed to cool to ambient temperature. Microscopic examination of the polished cups at 500X shall be undertaken. No corrosion worse than that of the control cup shall be evident in the candidate test fluid cup.

The report shall include photographs of both cups after testing and the candidate test fluid cup shall be identified.

## 3.1.2 Toxicity

The user shall ensure that the fluid meets all local, state, and/or federal toxicity regulations. The information to satisfy the federal, state, and provincial requirements shall be provided by the manufacturer, and for local requirements, upon request, from the user.

### 3.1.3 Appearance

The fluid, as received by purchaser, shall be homogeneous, uniform in color (see Table 1), and free from skins, lumps, and foreign materials which would be detrimental to usage of the product. The fluid may be clear or translucent - as described by the manufacturer.

### 3.1.4 Environmental Information

Formulated fluid shall be tested in accordance with APHA "Standard Methods for the Examination of Water and Waste Water". The manufacturer shall provide results for not less than the following:

## 3.1.4.1 Biochemical Oxygen Demand (BOD)

The fluid shall be incubated at 20 °C (68 °F) for 5, 15, 20, or 28 days dependent upon the method chosen, and the BOD determined.

# 3.1.4.2 Total Oxygen Demand (TOD) or Chemical Oxygen Demand (COD)

The TOD or COD of the fluid, expressed in kilograms of oxygen per kilogram of fluid, shall be determined.

# 3.1.4.3 Biodegradability

This characteristic can be approximated by determining the ratio of BOD and TOD or COD. The percent of fluid biodegraded can be calculated by dividing BOD by TOD or COD, and shall be reported for the incubation time periods specified in the chosen test method.

# 3.1.4.4 Aquatic Toxicity

The fluid shall be tested in accordance with EPA 40 CFR 797.1300 and 794.14, revised July 1, 1989, or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals), methods 202 and 203 using test species required by regulatory agencies for permitted discharges. Examples include fathead minnows, daphnia magna and rainbow trout. The LC50 concentration, (the highest concentration at which 50% of the organisms do not survive the test period) shall be stated in milligrams per liter.

# 3.1.5 Trace Contaminants

Report the presence, in percentages by weight or parts per million by weight, of sulfur, halogens, phosphate, nitrate, and heavy metals (lead, chromium, cadmium, and mercury). Report the test method used and detection limits.

# 3.2 Physical Properties

The fluid shall conform to the following requirements; tests shall be performed in accordance with the specified test methods.

# 3.2.1 Fluid as Received in Neat Form

### 3.2.1.1 Flash Point

Shall be not lower than 100 °C (212 °F) determined in accordance with ASTM D 93 or ASTM D 3278. In case of dispute, the flash point determined in accordance with ASTM D 93 shall apply.

## 3.2.1.2 Specific Gravity

Shall be within ±0.015 units of the preproduction value, determined in accordance with ASTM D 891, Method C, or ASTM D 4052.

3.2.1.3 pH

Shall be within ±0.5 units of the preproduction value, determined in accordance with ASTM E 70.

3.2.1.4 Refractive Index

Shall be within ±0.0015 units of the preproduction value, determined in accordance with ASTM D 1747.

3.2.1.5 Surface Tension

Shall be within ±10% of the preproduction value at 20 °C ± 2 (68 °F± 4), determined in accordance with ASTM D 1331.

- 3.2.2 Fluid Stability
- 3.2.2.1 Thermal Stability-Accelerated Aging (to simulate long term heated storage without water loss)

Age sample as in 3.2.2.1.1 and examine as in 3.2.2.1.2.

- 3.2.2.1.1 Transfer  $800 \pm 10$  ml of fluid to a 1 liter borosilicate bottle (e.g., Pyrex® brand or equivalent) fitted with a tight, heat-resistant plastic seal and tightly close. For the reference sample, also keep  $800 \pm 10$  ml of the fluid in an identical bottle and store at room temperature until the completion of 3.2.2.1.2. Transfer the other closed bottle containing the test fluid to a circulating-air oven or heated oil or water bath. Elevate the temperature to  $70 \,^{\circ}\text{C} \pm 2$  (158  $^{\circ}\text{F} \pm 4$ ) and maintain the test sample in this environment for 30 days.
- 3.2.2.1.2 After 30 days, remove the test sample from the heated environment and examine the contents for evidence of separation, precipitation or insoluble deposits. Report any evidence of these factors. Allow the sample to cool to 20 °C ± 2 (68 °F ± 4). Invert the bottle through four complete cycles, then examine for evidence of a significant difference in the amount of separation, precipitation or insoluble deposits of the heat-aged test fluid versus the unheated reference fluid. Report findings. Determine the refractive index of the test and reference fluids as in 3.2.1.4. If the test and reference fluids have a refractive index difference of greater than 0.0020, the test is invalid. If the refractive index is within limits, test for viscosity at 0 °C ± 2 (32 °F ± 4) as in 3.2.3.1.1 and pH as in 3.2.1.3. Compare and record the results of the heat-aged test fluid and the unheated reference fluid. The viscosity shall neither be reduced by more than 20% nor be increased by more than 10%. The pH difference shall not be greater than 1.0 unit. Fluid aged as above shall be tested in accordance with AS 5901 (WSET only) using one set of three plates. Report the result of the test.
- 3.2.2.2 Exposure to Dry Air (to simulate fluid behavior following overnight exposure to dry air)

The fluid shall be tested in accordance with 3.2.2.2.1. If the fluid, after losing 20% of its original weight, exceeds the viscosity limit defined in 3.2.2.2.1, the fluid shall then be tested in accordance with 3.2.2.2.2 to determine the percent weight loss and aerodynamic performance after exposure to a simulated dry air environment. Results from Successive Dryout and Rehydration (3.2.2.4) test should also be reviewed to ensure that any fluid that fails to meet the viscosity limit defined in 3.2.2.2.1 will not result in high levels of dried residue and/or gel formation.

3.2.2.2.1 The fluid, after exposure to a dry air environment which results in weight reduction of 20% ± 1 shall have a viscosity not exceeding 500 mPa·s when measured at 3.0 rpm and with spindle LV1 with sample at 20 °C ± 2 (68 °F ± 4) using the method described in 3.2.3.1.2.

- 3.2.2.2.1.1 Pour approximately 800 ml of fluid into a pre-weighed glass tray with the following approximate dimensions  $200 \times 200 \times 50$  mm. Weigh the tray and contents to the nearest 2 grams, record the weight. Place this tray with its contents into an environment with air temperature at 23 °C ± 3 (73 °F ± 6) and the relative humidity not greater than 50% and preferably in the 30 to 45% range. Periodically weigh the tray and contents. When the weight of the fluid is  $20\% \pm 1$  lower than its initial weight, pour 500 ml ± 10 of the fluid into a 600 ml beaker. Allow to cool to 20 °C ± 2 (68 °F ± 4) and test for viscosity.
- 3.2.2.2.2 The fluid shall be tested to determine the percent weight loss obtained under simulated conditions defined in 3.2.2.2.2.1. Fluid at this percent weight loss shall meet aerodynamic performance requirements in accordance with AS 5900 by performing three runs of the aerodynamic acceptance test (3.2.5.2) at -5 °C ± 2 (23 F ± 4), and speed ramp(s) used for preproduction tests of the Neat fluid.
- 3.2.2.2.2.1 Place a 1 mm layer of fluid into a pre-weighed Petri dish cover with the approximate dimensions of 10 cm diameter × 5 mm height. Record the weight to the nearest 0.02 grams. Place the Petri dish into a 55% ± 2 relative humidity environment with air temperature at 0 C ± 2 (32 F ± 4) and 4 m/s ± 0.5 horizontal air velocity for 12 hours ± 0.2. Equilibrate the dish and its contents to 20 C ± 2 (68 F ± 4) and record the final weight. The percent weight loss shall be reported from an average of at least three Petri dish tests.
- 3.2.2.2.2.2 Additional fluid at the percent weight loss obtained under the simulated conditions (± 1 wt%) in 3.2.2.2.2.1 can be prepared at room temperature in pans under a fume hood to make the quantity required for aerodynamic testing.
- 3.2.2.3 Dry-out by Exposure to Cold Dry Air (to simulate fluid dry-out in cold air on the ramp and on the aircraft including aerodynamically quiet areas)

Test as per 3.2.2.3.1 and 3.2.2.3.2.

- 3.2.2.3.1 Using a cold chamber, position three plates, inclined at an angle of 2 degrees from the horizontal. Pour 100 to 150 ml on each plate. Set the air and plate temperatures at 1 °C ± 1 (34 °F ± 2). Relative humidity less than 40%, and the air moving from the top over the surface towards the bottom of the plates at approximately 2.5 m/s (5.6 mph). Maintain these conditions until the fluid has dried or for 24 hours. Examine the remaining residues. A gel, gum (tacky feel), hard granular solid, or peelable film are not acceptable.
- 3.2.2.3.2 Remove plates from cold chamber and allow them to warm to 5 °C ± 2 (41 °F ± 4) and rinse each plate with 500 ml water, conforming to ASTM D 1193 Type IV, at 15 °C ± 10 (59 °F ± 18) from a 1 liter squeeze bottle. Report appearance of plate after rinsing. Only plates free of residue are acceptable.
- 3.2.2.4 Successive Dryout and Rehydration (to simulate the formation of dried residues and for such to form gels upon rehydration)

Fluid shall be tested in accordance with Appendix A and results reported.

3.2.2.5 Thin Film Thermal Stability (to simulate heated leading edge dryout)

Test as per 3.2.2.5.1, 3.2.2.5.2 and 3.2.2.5.3.

- 3.2.2.5.1 At ambient temperature, 40 to 50 ml of the fluid shall be poured on to each of two unpainted aluminum or aluminum alloy test panels, approximately 152.5 × 50 mm (6 × 2 inches). After 5 minutes, the panels shall be placed, inclined at an angle of 10 degrees from the horizontal, in an oven maintained at 95 °C ± 2 (203 °F ± 4). After 60 minutes ± 1, remove the panels, allow to cool to ambient temperature, and inspect. Report the appearance. A gel, gum (tacky feel), hard granular solid or peelable film are not acceptable.
- 3.2.2.5.2 Rinse each plate with 500 ml water conforming to ASTM D 1193 Type IV at 15 °C ± 10 (59 °F ± 18) from a 1 liter squeeze bottle positioned not closer than 20 cm from the surface. Report the appearance of the plates after rinsing. Only plates free of residue are acceptable.

- 3.2.2.5.3 Repeat test as in 3.2.2.5.1 and 3.2.2.5.2 except that the oven temperature shall be at 48  $^{\circ}$ C  $\pm$  2 (118  $^{\circ}$ F  $\pm$  4). Report results of both tests.
- 3.2.2.6 Storage Stability (to simulate storage in tanks)

Prior to the start of this test, the viscosity shall be determined at 20  $^{\circ}$ C  $\pm$  2 (68  $^{\circ}$ F  $\pm$  4), in accordance with 3.2.3.1.1. The fluid shall be tested in accordance with ASTM F 1105, except that the sample shall be protected from exposure to UV light. Upon completion of the test, the fluid shall be retested for viscosity as before and the result compared to the original values. Both results shall fall within the limits determined in 3.2.3.1.

# 3.2.2.7 Shear Stability

The anti-icing performance tests as in 3.2.4 shall start within two hours after the product has been sheared, but not within the first 20 minutes after shearing using the laboratory method as in 3.2.2.7.1.

3.2.2.7.1 Pour 500 ml ± 5 of fluid at 20 °C ± 2 (68 °F ± 4) into a 600 ml, straight-sided glass vessel, 85 mm ± 0.5 diameter, and position a Brookfield counter rotating mixer (or equivalent device, as demonstrated in testing versus counter rotating mixer), so the bottom of the blade is 25 mm ± 2 from the bottom of the vessel and mix for 5 minutes ± 10 seconds at 3500 rpm ± 100 (calibrated in water prior to each test).

## 3.2.2.8 Hard Water Stability

The fluid, diluted 1:1 with standard hard water made up as in 3.2.2.8.1 and aged as in 3.2.2.8.2, shall show no evidence of insoluble deposit, and the pH shall not vary by more than  $\pm 1.0$  unit from a fresh unaged sample, if after aging the refractive index has increased by more than 0.0020 at 20 °C  $\pm$  2 (68 °F  $\pm$  4) from a fresh unaged sample, the test is invalid as water has been allowed to evaporate during the test. A sample of the fluid, after hard water stability testing, shall be tested in accordance with AS 5901 (WSET only) using one set of three plates, and the results reported.

### 3.2.2.8.1 Composition of Hard Water

Dissolve 400 mg  $\pm$  5 calcium acetate dihydrate (Ca[C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>]<sub>2</sub>·2H<sub>2</sub>O) or 363 mg  $\pm$ 5 calcium acetate monohydrate (Ca(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>·H<sub>2</sub>O) and 280 mg  $\pm$  5 magnesium sulfate heptahydrate (MgSO<sub>4</sub>·7H<sub>2</sub>O), both of analytical reagent quality, in 1 liter of ASTM D 1193, Type IV, water.

3.2.2.8.2 Place 800 ml ± 10 of the diluted fluid into a 1 liter borosilicate bottle (e.g., Pyrex® brand or equivalent) fitted with a tight, heat-resistant plastic seal and tightly close. Place container in an oven or oil or water bath and raise the temperature to 95 °C ± 2 (203 °F ± 4). Allow the sample to remain in this elevated temperature environment for 30 days. Remove and allow to cool to 20 °C ± 2 (68 °F ± 4). Inspect as in 3.2.2.8.

# 3.2.2.9 Tendency to Foam

At the option of the user, the vendor shall demonstrate that fluid as supplied or in intended use dilutions and heated to intended use temperatures, applied through commercial deicing/anti-icing vehicle systems to an inclined flat or curved surface, preferably an aircraft wing or horizontal stabilizer, at pressures and flow rates normal for the intended use, does not cause foam which does not rapidly collapse, and fluid surface shall not have the appearance of snow or slush.

3.2.2.10 Cold Storage Stability (to simulate the stability of the fluid cycling between cold temperature and room temperature)

Prior to the start of this test, the sample shall be visually examined to determine freedom from insoluble deposits. Report any evidence of these factors. Determine the pH in accordance with 3.2.1.3. Determine the refractive index in accordance with 3.2.1.4. Determine the viscosity in accordance with 3.2.3.1.2, ASTM D2196, Method B, except the sample shall not be shaken, using a Brookfield LV viscometer or equivalent, fitted with the guardleg and appropriate spindle for the speed selected. Values shall be taken at a spindle speed of 0.3 rpm at 20 °C  $\pm$  2 (68 °F  $\pm$  4). The report shall clearly state the size and type of spindle used.

3.2.2.10.1 Transfer 2.0 liters of fluid into an appropriate 2.5 liter container (ex: high-density polyethylene-HDPE, glass separatory funnel, etc.) with a well-closed cap. Transfer the closed container containing the sample of fluid to a freezer. Maintain the sample at -20 °C ± 2 (-4 °F ± 4) for 24 hours. After 24 hours, remove the closed container containing the fluid sample from the freezer. Allow the sample to stand at 20 °C ± 2 (68 °F ± 4) for 24 hours. Repeat the cycling between -20 °C and 20 °C for 5 complete cycles. An example of the cycling period is shown in Table 2.

Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	Day 9	Day 10
-20 °C	20 °C								

3.2.2.10.2 Upon completion of the cycling, the fluid shall be visually examined for evidence of separation, precipitation, or insoluble deposits. Report any visual observations. Allow fluid to equilibrate to room temperature. Siphon/extract 1 liter from the top portion of the fluid sample, ensuring that the tip of the siphon hose remains 2 inches beneath the surface of the fluid, and transfer the top portion into a separate container (labeled top). Siphon/extract 1 liter from the bottom portion, ensuring that the tip of the siphon hose remains at the bottom of the container, and transfer the bottom portion into a separate container (labeled bottom). The top and bottom portions of the fluid sample shall be retested as before for pH, refractive index, and viscosity in accordance with 3.2.2.10 and the results compared to the original values.

## 3.2.3 Rheological Properties

The special rheological properties defined in 3.2.3.1 and 3.2.3.1.1 relate to thickened fluids which are classed as non-Newtonian, pseudoplastic as defined in 1.1.2 and 1.1.3 and are specified to ensure the flow of the film when sufficient shear stress is induced. The exposure of a film of the applied fluid to different environmental factors shall not impair this performance (either by buildup of film thickness due to consecutive applications) or by forming a gel), when tested in accordance with 3.2.2.2 and 3.2.2.3. Fluids of all types shall be tested as in 3.2.3.1.1.

# 3.2.3.1 Viscosity

The fluid shall exhibit non-Newtonian flow behavior over the temperature range at which the fluid has been tested and certified as acceptable in accordance with the Aerodynamic Acceptance test (3.2.5). The viscosity of any neat fluid as supplied shall fall within the limits of the high and low viscosity values defined by the pre-production samples (4.2.3) and the viscosity limits shall be reported. For quality control purposes the manufacturer shall specify the typical viscosity range a user can expect to obtain from fluid being delivered for use.

# 3.2.3.1.1 Viscosity Measurement

The sample will not be shaken prior to testing. Test at 20 °C  $\pm$  2 (68 °F  $\pm$  4), 0 °C  $\pm$  2 (32 °F  $\pm$  4) and in 10 °C (18 °F) increments down to the lowest usable temperature identified by the manufacturer. The viscosity shall be measured using a Brookfield LV viscometer at 0.3, 6 and 30 rpm. The viscosity may be measured using the Brookfield small sample adapter or as specified in 3.2.3.1.2. The report shall state if the small sample adapter was used for the equipment in 3.2.3.1.2 and shall detail the spindle size, container size, volume of fluid employed, and the rotation duration. In case of dispute the method described in 3.2.3.1.2 shall prevail.

3.2.3.1.2 Using a Brookfield LV viscometer fitted with the guard leg and using a sample of approximately 500 ml or sufficient quantity to accommodate the selected spindle and guard leg, contained in an 85 mm diameter, straight sided 600 ml beaker. The test shall be run using the appropriate spindle in accordance with ASTM D 2196, Method B, except the samples shall not be shaken. The reporting requirements are the same as in 3.2.3.1.1.

#### 3.2.4 Anti-Icing Performance

#### 3.2.4.1 Water Spray Endurance Test (WSET), and High Humidity Endurance Test (HHET)

When tested in accordance with AS 5901, the neat fluid, sheared as in 3.2.2.7, shall form a film which will protect against the formation of frozen deposits within the failure zone depicted in Figure 1 of AS 5901 for the times listed in Table 3. Similarly, if the fluid is intended to be diluted with water, it shall be diluted by volume to ratios of 75:25 and 50:50 with water conforming to 3.2.2.8.1 and sheared as in 3.2.2.7, and shall protect as in Table 3. Confirmation shall be obtained from six panels, three panels from each of two successive test runs.

17.1522 0 11.11.11.11.11.11.11.11.11.11.11.11.11.					
Fluid - SAE Type	WSET	HHET			
& Dilution	Time - Minutes	Time - Hours			
II 100:00	30	4			
75:25	20	2			
50:50	5	0.5			
III 100:00	20	2			
75:25	Determine and report	Determine and report			
50:50	Determine and report	Determine and report			
IV 100:00	80	8			
75:25	20	2			
50:50	5	0.5			
	<u> </u>				
nic Acceptance	0,				
to Shear or Not to Shear the Fluid					
to Shear or Not to Shear th	ne Fluid				
n accordance with 3.2.2.7	7.1 the apparent viscosity	increases by more than 15			

TABLE 3 - MINIMUM ANTI-ICING PERFORMANCE

#### 3.2.5 Aerodynamic Acceptance

#### Criterion to Shear or Not to Shear the Fluid 3.2.5.1

If after shearing in accordance with 3.2.2.7.1 the apparent viscosity increases by more than 15% when tested in accordance with 3.2.3.1.1 at 0 °C ± 2 (32 °F ± 4) at spindle speeds of 6, and 30 rpm, the fluid shall be sheared prior to testing. If the apparent viscosity increased by less than or equal to 15% or decreased the fluid shall be tested in the unsheared state.

#### 3.2.5.2 Aerodynamic Test

The fluid shall demonstrate acceptable aerodynamic performance when tested in accordance with AS 5900 High Speed Ramp Test and/or Low Speed Ramp Test. The High Speed Ramp Test is the approved aerodynamic test for fluids used on large transport type aircraft. The Low Speed Ramp Test is the approved aerodynamic test for fluids used on lower takeoff rotation speed commuter aircraft. Also see 1.2.1. A fluid and its volume/volume dilutions (defined in Table 3) shall be tested at temperatures ranging from 0 °C ± 2 (32 °F ± 4) down to the lowest temperature requested by the fluid manufacturer, in approximately 10 °C (18 °F) intervals. For each fluid or dilution, a minimum of three tests shall be performed at each temperature.

#### 3.2.5.3 Test with the Highest Viscosity Dilution

For the preproduction fluid, the dilution ratios which produce high viscosity at different temperature intervals shall be identified and tested for aerodynamic performance as described in 3.2.5.3.1. The report shall include viscosity measurements and highlight aerodynamic performance data at the respective dilution ratios and temperature intervals.

Prepare dilutions of the fluid in 5 wt% increments using water conforming to ASTM D 1193 Type IV. The 3.2.5.3.1 viscosity shall be measured using a Brookfield LV viscometer at 6 and 30 rpm using the small sample adapter and appropriate spindle at 0 °C  $\pm$  2 (32 °F  $\pm$  4), -10 °C  $\pm$  2 (14 °F  $\pm$  4) and -20 °C  $\pm$  2 (-4 °F  $\pm$  4). Measurements shall be made on dilutions with increasing water content until three consecutive decreasing viscosity measurements are obtained. Each dilution which exhibits a viscosity greater than that of the Neat, 75:25 or 50:50 dilution, by more than 15% at each temperature interval, shall be tested in accordance with AS5900 by performing a single aerodynamic acceptance test (3.2.5.2) at the respective temperature interval, and speed ramp(s) used for preproduction tests of the Neat fluid.

### 3.2.5.4 Fluid Elimination

Fluid elimination shall be tested and calculated in accordance with AS 5900 and shall be based on an initial thickness of 2 mm of fluid on the test duct floor. The elimination shall be not less than 74% at all tested temperatures for the High Speed Ramp Test. It shall not be less than 57% at all tested temperatures for the Low Speed Ramp Test.

### 3.3 Fluid Tested Both Neat and as a Diluted Solution

Tests shall be conducted using the neat fluid and using a solution comprised of the neat fluid diluted 1:1 by weight with ASTM D 1193, Type IV, water.

# 3.3.1 Freezing Point

For the neat fluid, the freezing point shall be not higher than -32 °C (-26 °F). When diluted at a ratio of 1:1 by weight with ASTM D 1193, Type IV, water, the freezing point shall be not higher than -10 °C (+14 °F), determined in accordance with ASTM D 1177. Report freezing points for both the neat and 1:1 diluted fluid.

### 3.3.2 Effect on Aircraft Materials

### 3.3.2.1 Sandwich Corrosion

After testing in accordance with ASTM F1110, the test specimens shall not show corrosion worse than controls, when control panels are tested using water conforming to ASTM D 1193.

### 3.3.2.2 Total Immersion Corrosion

The fluid, tested in accordance with ASTM F 483, shall neither produce evidence of corrosion of test panels nor cause a weight change of any test panel greater than shown in Table 4.

TABLE 4 - TOTAL IMMERSION CORROSION

Cilick	Weight Change mg/cm <sup>2</sup> per 24
Test Panel	Hours
AMS 4037 Aluminum Alloy, anodized as in AMS 2470	0.3
AMS 4041 Aluminum Alloy	0.3
AMS 4049 Aluminum Alloy	0.3
AMS 4376 Magnesium Alloy, dichromate treated as in AMS 2475	0.2
AMS 4911 or MAM 4911 Titanium Alloy	0.1
AMS 5045 Carbon Steel	0.8

# 3.3.2.3 Low-Embrittling Cadmium Plate

Test panels, coated with low embrittling cadmium plate, shall not show a weight change greater than 0.3 mg/cm<sup>2</sup> per 24 hours, determined in accordance with ASTM F 1111.

### 3.3.2.4 Stress-Corrosion Resistance

The fluid shall not cause cracks in AMS 4911 titanium specimens when tested in accordance with ASTM F 945, Method A.

### 3.3.2.4.1 Stress Corrosion Resistance

The fluid shall be tested in accordance with ASTM F 945, Method A, using AMS 4916 specimens. The test report shall detail the effects of the fluid and of the control solution.

## 3.3.2.5 Hydrogen Embrittlement

The fluid shall be non-embrittling determined in accordance with ASTM F 519, Types 1a, or 1c or 2a. In case of dispute, the Type 1c bar shall be used. Galvanic reactions must not be allowed to occur; this may be accomplished by confining the test sample to the test specimen using a chemically inert cup.

### 3.3.2.6 Effect on Transparent Plastics

When heated to 65 °C ± 2 (149 °F ± 4), the fluid shall not craze, stain, or discolor MIL-PRF-25690 stretched acrylic plastic, determined in accordance with ASTM F 484.

3.3.2.6.1 Similarly fluid shall not craze, stain, nor discolor AMS-P-83310 polycarbonate plastic, determined in accordance with procedures in ASTM F 484 except that the specimens shall be stressed for 30 minutes ± 1 to an outer fiber stress level of 13.8 MPa (2000 psi).

### 3.3.3 Effect on Painted Surfaces

When heated to 65 °C  $\pm$  2 (149 °F  $\pm$  4) and applied to a painted surface having an initial surface temperature of 22 °C  $\pm$  2 (72 °F  $\pm$  4), the fluid shall not produce any streaking, discoloration, or blistering of the paint film, and shall not decrease paint film hardness by more than two pencil hardness numbers, determined in accordance with ASTM F 502.

# 3.3.4 Effect on Unpainted Surfaces

The fluid tested in accordance with ASTM F 485, shall neither produce streaking nor leave any stains, which require polishing to remove.

## 3.3.5 Pavement Compatibility

# 3.3.5.1 Runway Concrete Scaling Resistance

The condition of the runway concrete surface shall have a rating not greater than 1 for 50 freeze-thaw cycles, determined in accordance with ASTM C 672, except that the concrete shall be air-entrained with an air content as in ASTM C 672, have a minimum cement content of 302 kg/m $^3$  ± 4.5 (510 lb/yd $^3$  ± 10) and a slump, 38 mm ± 13 (1.5 inches ± 0.5). A 25% ± 1 by volume solution of the neat fluid prepared using tap water shall be substituted for the specified calcium chloride. Performing more than one freeze- thaw cycle per day is acceptable.

# 4. QUALITY ASSURANCE PROVISIONS

### 4.1 Responsibility for Inspection

The vendor of the fluid shall supply all samples for conformance testing and shall be responsible for obtaining independent laboratory confirmation of conformance to the requirements of this specification. Each fluid sample container shall be clearly identified with the vendor's name, fluid name or code number and lot number, and manufacturing site address. The purchaser or user reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the fluid conforms to the requirements of this specification.

### 4.2 Classification of Tests

### 4.2.1 Lot Acceptance Tests

pH (3.2.1.3), refractive index (3.2.1.4), and viscosity at 20 °C ± 2 (68 °F ± 4) and 0 °C ± 2 (32 °F ± 4 °F) at 0.3, 6, and 30 rpm are acceptance tests and shall be performed on each lot. Any Certificate of Analysis for a production lot shall contain these data.

# 4.2.2 Periodic Tests

Anti-icing performance (3.2.4), and aerodynamic performance (3.2.5) are periodic tests, and shall be performed on, or just prior to the second anniversary of initial testing, thereafter every two calendar years. Aerodynamic performance (3.2.5) shall be performed by an approved and autonomous test facility. Anti-icing performance (3.2.4) shall be performed by an autonomous facility, unless another facility is agreed upon between the purchaser or user and the vendor.

### 4.2.3 Preproduction Tests

Two preproduction samples shall be submitted and tested in accordance with 4.2.3.1 and 4.2.3.2. Tests shall be performed prior to or upon the initial shipment of the fluid to a purchaser (except for Storage Stability (3.2.2.6) which may be waived by the purchaser to permit entry of a new product), when a change in any ingredient or production method requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

# 4.2.3.1 High Viscosity Preproduction Sample

Tests for all technical requirements shall be performed on the High Viscosity sample. The High Viscosity sample shall define the maximum viscosity that complies with the aerodynamic acceptance test (3.2.5). This viscosity value will be reported as the maximum on-wing viscosity of the fluid.

# 4.2.3.2 Low Viscosity Preproduction Sample

The Low Viscosity sample shall have a viscosity value less than the High Viscosity sample and greater than or equal to the viscosity of the sample submitted for Endurance Time testing (ARR 5485). The following tests shall be conducted on the Low Viscosity sample:

- a. Viscosity shall be measured at 20 °C  $\pm$  2 (68 °F  $\pm$  4) and 0 °C  $\pm$  2 (32 °F  $\pm$  4), at 0.3, 6, and 30 rpm in accordance with 3.2.3.1.1.
- b. WSET in accordance with 3.2.4.1.
- c. Aerodynamic Performance in accordance with AS 5900. The test shall consist of one data point, three runs, using the neat fluid. The test shall repeat the lowest temperature ±1 °C (2 °F), at which the High Viscosity sample met the Aerodynamic Performance requirements.
- 4.2.3.3 For direct U.S. Military procurement, substantiating test data and, when requested, preproduction fluid shall be submitted to the cognizant agency as directed by the procuring activity, contracting officer, or request for procurement.
- 4.3 Sampling and Testing

Shall be in accordance with 4.3.1 or 4.3.2, as applicable. A lot shall be all fluid produced in one continuous manufacturing process using materials from the same batches of raw materials and presented for vendor's inspection at one time. Sufficient fluid from a single production lot shall be taken to perform all required tests.

### 4.3.1 Bulk Shipments

In accordance with ASTM D 4177.

# 4.3.2 Drum Shipments

In accordance with ASTM D 1568.

4.3.3 When a statistical sampling plan has been agreed upon by the purchaser and the vendor, sampling shall be in accordance with such plan in lieu of sampling as in 4.3.1 or 4.3.2, and the report of 4.5 shall state that such a plan was used.

# Approval

- 4.4.1 A sample fluid shall be approved by the purchaser before fluid for production use is supplied, unless such approval is waived by purchaser. Results of tests on production fluid shall be essentially equivalent to those on the approved sample.
- 4.4.2 The vendor shall use ingredients, manufacturing processes, and methods of inspection for production fluid which are essentially the same as those used to produce the fluid which is presented for qualification to this specification. If it is necessary to make any change in ingredient or the manufacturing process, the vendor shall submit such fluid for reapproval. Production fluid made by the revised procedure shall not be shipped to the purchaser until full testing and approval has been received.
- 4.4.3 Whenever a fluid is to be produced at multiple locations, or by a licensee or subcontractor, all testing shall be required on fluid produced at each site, prior to initial shipment, as if the fluid were being initially qualified, unless the production method, materials, and handling are the same as the originally qualified vendor's production materials and methods. In any case, the fluid so produced shall initially be confirmed by the aerodynamic acceptance test (3.2.5), and by a single test run of three panels under WSET (3.24.1). POFOFO

#### 4.5 Reports

#### 4.5.1 Preproduction and Periodic Test Reports

Before the initial shipment, the vendor of fluid shall furnish a report showing the results of tests to determine conformance to all the technical requirements of this specification. These tests shall be performed by an approved and/or independent testing facility/facilities (see 4.2.2). The aerodynamic acceptance test facility shall determine and report the following properties from the sample of fluid submitted for testing: lick to vie

- a. Viscosity (3.2.3.1.1)
- Refractive Index (3.2.1.4)
- pH (3.2.1.3) C.
- Surface Tension (3.2.1.5)
- 4.5.1.1 The reports shall include the quantity, lot number, AMS 1428 (latest revision) and manufacturer's product identification, date of manufacture, and manufacturing location.
- Subsequent reports of the results of periodic recertifications shall compare the results obtained to the original 4.5.1.2 fluid certification documents.
- A material safety data sheet conforming to AMS 2825, or equivalent, shall be supplied to each purchaser prior to 4.5.2 or concurrent with the report of preproduction test results or, if the preproduction test be waived by purchaser, concurrent with the first shipment of fluid for production use. Each request for modification of the fluid formulation or change in the reportable status of any of the raw materials used shall be accompanied by a revised material data sheet for the proposed formulation.

#### 4.6 Resampling and Retesting

If any sample used in the above tests fails to meet the specified requirements, disposition of the fluid may be based on the results of testing three additional samples for each original non-conforming sample. Failure of any retest sample to meet the specified requirements shall be cause for rejection of the fluid represented. Results of all tests shall be reported.

# 5. PREPARATION FOR DELIVERY

- Packaging and Identification 5.1
- 5.1.1 The fluid shall be packaged in containers of a size and type acceptable to purchaser or shall be delivered in bulk.