

Ice Undercutting Test Method for Runways and Taxiways Deicing/Anti-icing Chemicals

RATIONALE

This test method, for liquid and solid deicing/anti-icing chemicals, offers a quantitative procedure to evaluate the ice undercut as a function of the time and temperature by such deicing/anti-icing chemical and is based on the SHRP H-205.6- MODIFIED FOR AIRPORT APPLICATION (draft April 17/02) Test Method for Ice Undercutting by Liquid Deicing Chemicals (or Solids).

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1. SCOPE

This test method provides stakeholders (runway deicing chemical manufacturers, deicing/anti-icing chemical operators and airport authorities) with relative ice undercutting capacity of runway deicing chemicals, by measuring the area of ice undercut pattern as a function of time. Such runway deicing chemicals are often also used on taxiways.

This test method does not quantitatively measure the theoretical or extended time of ice undercutting capability of ready-to-use runway deicing/anti-icing chemicals in liquid or solid form.

1.1 Minimum Requirements

This method sets the minimum requirements for the determination of ice undercutting capabilities of SAE AMS 1431 and 1435 runway deicing/anti-icing chemicals in liquid and solid form as a function of time and temperature under controlled laboratory conditions.

1.2 Hazardous Materials

This test may involve the use of hazardous materials, operations, and equipment. This standard does not address the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 Standard Units

The values stated in SI units are to be regarded as the standard.

2. APPLICABLE DOCUMENTS

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and 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.

2.1 SAE Publications

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.

AMS1431 Compound, Solid Runway and Taxiway Deicing/Anti-icing

AMS1435 Fluid, Generic, Deicing/Anti-icing, Runways and Taxiways

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 D 1193 Specification for Reagent Water

ASTM D 1747 Refractive Index of Viscous Materials

2.3 ISO Publications

Available from International Organization for Standardization, 1, rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland, Tel: +41-22-749-01-11, www.iso.org.

ISO 9001-2008 Quality Management Systems – Requirements

2.4 Other Reference Documents

Handbook of Test Methods for Evaluating Chemical Deicers, Strategic Highway Research Program, National Research Council Washington , DC 1992, SHRP-H-332

3. SUMMARY OF TEST METHOD

3.1 Introduction

This test utilizes small cylindrical cavities in a sheet of ice of uniform thickness frozen in a flat circular modified polystyrene petri dish having an average standardized surface roughness of 120 grit (see Figure 1). The bottoms of the cavities are essentially free of ice. After equilibration to the desired temperature, a known volume of dyed deicing chemical is placed in the cavities, and undercutting commences (see figure 1). Undercutting is evidenced by the formation of essentially circular undercut patterns. At specified time intervals, the dimensions of the observed undercut patterns are measured. The undercut pattern reflects the net result of melting on the walls of the ice cavity and melting at the ice/substrate interface. The undercut is relatively thick near the center and relatively thin at the extremities. The undercut area is defined as the total area of the circular undercut pattern minus the area of the original cavity. Testing temperatures shall be within 1 °C (2 °F) of the stated values and tests shall be performed within a freezer or a cold chamber.



FIGURE 1 - ADAPTED POLYSTYRENE PETRI DISH WITH DYED CHEMICAL

3.2 Significance and Use

This test method provides a means to evaluate and compare the rate of ice undercutting capabilities of runway deicing/anti-icing chemicals in liquid or solid form at the ice/substrate interface over limited, defined time intervals at specified temperatures.

3.3 Test Equipment and Materials

The following test procedure may be performed on deicing/anti-icing chemicals in liquid or solid form.

3.3.1 Material

- a. Polystyrene petri dish, 150 mm (6 in) diameter & 15 mm (0.6 in) deep (or equivalent). e.g.e BD Falcon disposable petri dishes, sterile, Fisher Scientific cat. # 08-757-148.

- b. Silicon carbide abrasive paper, 120 Grit
 - PSA Backed paper
 - 8" (20.3 cm) diameter
 - Pace Technologies (www.metallographic.com)
 - Cat. No. : SiC-120P12-100 (Box of 100)
- c. Pipettor (single or multichannel pipettor) able to deliver a volume of 25 μ L
- d. Plastic syringe with needle (capacity 5 mL)
- e. Graduated cylinder
- f. Aluminum rod, 3 mm (0.12 in) diameter
- g. Digital thermometer (or equivalent)
- h. Digital timer (or equivalent)
- i. Heating plate
- j. Refrigerator or room to store the material at $4\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($39\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$)
- k. Micrometer
- l. Rhodamine or Fluorescein 0.1% (w/v) solution (other dyes can be used provided that it is clearly specified)

3.3.2 Adapted Polystyrene Petri Test Dish

A circular polystyrene petri dish, 150 mm (6 in) diameter and 15 mm (0.6 in) deep, is used to perform ice undercutting tests. The bottom of the petri dish is modified by means a silicon carbide abrasive paper of 120 grit surface roughness used as a standardized test surface. The ice surface area corresponds to 0.018 m^2 (see Figure 1). The dimensions of the test dish may be adjusted to accommodate space limitations; however, any changes in the ice surface area may necessitate proportional changes in deicer test sample size and/or volume of water used to grow the ice.

3.3.3 Petri Dish Special Surface Considerations

The cleanliness or purity of standardized surface specimens is known to affect the nature of bonding between ice and substrates. Care should be taken to protect surface specimens from exposure to dusts, laboratory chemicals, and finger contact.

3.3.4 Standard Measuring Devices

All temperature sensors, electronic balances, and timing devices shall be maintained in a known state of calibration by means of a Quality Management System recognized by an international standards organization such as ISO 9001-2008 (or equivalent).

3.3.5 Ice Preparation

The adapted petri dishes and material required for the ice preparation (water, graduated cylinder, etc ...) have to be kept at $4\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($39\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$) for a minimum period of $8\text{ h} \pm 1\text{ h}$ prior to preparation of the ice. Sixty milliliters of water corresponds to an ice thickness of 3.4 mm in the test dish. This pre-cooled quantity of ASTM D 1193, Type IV water is poured in the dish and swirled or stirred to distribute water over the surface. The dish is then placed on a level surface in a freezer or cold room at $-10\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($14\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$) within 5 minutes after filling the test dish with water. The uncovered petri dish with the water is kept in the freezer or cold chamber until the water is completely frozen (minimum 8 hours). The ice formation period may be prolonged until the water is completely frozen. During the ice formation, it is recommended to maintain ventilation as low as possible to obtain a smoother ice surface. The adapted petri dish containing the ice sheet is stored at the intended test temperature at least $10\text{ h} \pm 1\text{ h}$ prior to the test.

3.3.6 Cavity Fabrication

Cavities in the ice (3.4 mm depth down to the substrate surface) are fabricated using a warm aluminum rod and using a plastic syringe with needle, melted ice is extracted from the pre-made cylindrical cavities. The following process is repeated for each cavity. A rod is fabricated by machining aluminum rod to a nominal diameter of 3 mm (0.12 in). Approximately 100 mL of water in a 250-mL beaker is warmed to 65 °C to 70 °C (149 °F to 158 °F). The aluminum rod is placed in the water and allowed to warm for 30 seconds. The warm rod is taken out of the water, wiped with a tissue, placed vertically on the ice surface, and pressed downward with moderate pressure for 3 to 4 seconds. The rod is then removed, the syringe tip placed in the cavity, and melted water drawn into the syringe. The syringe is held in position 2 to 3 seconds, then withdrawn and water ejected. If visual inspection indicates a film of ice on the bottom of the cavity, the process with rod and syringe can be repeated, taking care not to enlarge the diameter of the cavity. The cavity-forming process should be conducted in the cold room at the test temperature. Use one petri dish per set of five cavities about 4 cm (1.5 in.) apart. The adapted petri dish containing the ice sheet shall be kept at the intended test temperature at least 2 hours prior to the test.

3.4 General Test Procedures

3.4.1 Deicing/anti-icing Chemical Form

The following test procedure can be performed on deicing/anti-icing chemical in liquid or solid form.

3.4.1.1 Liquid Runway Deicing/anti-icing Chemical

Liquid runway deicing/anti-icing chemical shall be evaluated as supplied. No dilution or preparation is needed prior to the test.

3.4.1.2 Solid Runway Deicing/anti-icing Chemical

Solid runway deicing/anti-icing chemical must be diluted to give a 25% w/w solution. To obtain this 25 % w/w solution, dissolve "x" g of solid runway deicer in "3x" g of demineralized water.

3.4.1.3 Reference Control Solution

A reference control solution shall be tested along with the deicing/anti-icing chemical. The results are used as a validation test and allows to manufacturer an optional comparative test. The reference solution consists of a potassium acetate (reagent grade ACS > 99%) solution 50% w/w in ASTM D 1193, Type IV water.

The 50% w/w reference control solution is prepared by mixing x g of potassium acetate and x g of demineralized water and is calculated according to Equation 1:

$$\% w / w = \left(\frac{x \text{ g CH}_3\text{CO}_2\text{K}}{x \text{ g CH}_3\text{CO}_2\text{K} + x \text{ g H}_2\text{O}} \right) * 100 \quad (\text{Eq. 1})$$

where:

CH₃CO₂K: potassium acetate (Reagent grade ACS > 99%)

H₂O: water (ASTM D 1193, Type IV)

3.4.2 Incorporation of Dye

If the liquid deicing/anti-icing chemical or the 25% w/w dilution of solid deicing/anti-icing chemical do not have a vivid enough (strength) to accurately report undercutting capability, a dye shall be prepared and added as follows;

- a. Two dyes are recommended to perform the test; the Rhodamine B (CAS # 81-88-9) and the Fluorescein (CAS # 518-47-8). Both dyes can be used however; the Rhodamine B should be prioritized since it shows the better contrast and facilitates the ice undercutting measurement. The dye should be chosen so that there are no compatibility issues with the different runway deicer chemistries. The dye used for the test should be specified and agreed accordingly.
- b. The dye shall be diluted to 0.1 percent by volume (0.1% w/v) in ASTM D 1193, Type IV water. To obtain this 0.1 % w/v solution, dissolve 0.025 g of dye in 25 mL of demineralized water.

- c. Fill a 1.5 mL micro centrifuge tube (vial) with the deicer to be tested. Add 2 drops of the dye (0.1% w/v) solution to the deicing/anti-icing chemical sample, close the vial and shake vigorously.
- d. Keep sample at test temperature for a minimum of 8 hours prior to the test.

3.4.3 Deicing/anti-icing Chemical Addition to Cavities

Use a micro-pipette to place a 25 μL aliquot of deicing/anti-icing chemical into each of five cavities. Make sure that the pipettor is accurately calibrated before using. Undercutting is measured (using a digital micrometer) and recorded according to specified time intervals (currently 5, 10 and 30 minutes). A timer is essential for monitoring elapsed time. A thermometer or temperature sensor serves as a means to observe temperature and helps achieve constant test temperature.

3.5 Temperature Regulated Test Enclosures

An enclosure, which can be regulated to within $\pm 1.0\text{ }^{\circ}\text{C}$ ($\pm 2.0\text{ }^{\circ}\text{F}$) of the designated test temperature, $-2\text{ }^{\circ}\text{C}$ to $-10\text{ }^{\circ}\text{C}$ ($28\text{ }^{\circ}\text{F}$ to $14\text{ }^{\circ}\text{F}$), is required for two purposes: (1) to equilibrate test specimens to the designated temperature, and (2) to maintain temperature during actual tests.

The enclosure should have a temperature capability ranging from $-18\text{ }^{\circ}\text{C}$ ($0\text{ }^{\circ}\text{F}$) to at least $-1\text{ }^{\circ}\text{C}$ ($30\text{ }^{\circ}\text{F}$)

3.6 Number of Tests

A minimum of five test shall be performed (five cavities used) for each test temperature. Test temperatures shall be $-10\text{ }^{\circ}\text{C}$ ($14\text{ }^{\circ}\text{F}$) and $-2\text{ }^{\circ}\text{C}$ ($28\text{ }^{\circ}\text{F}$) within $0.5\text{ }^{\circ}\text{C}$ ($1\text{ }^{\circ}\text{F}$). The ice undercutting test can be performed at any other temperature upon agreement between purchaser and vendor.

3.7 Data Recording and Reporting

Circular-shaped ice undercuttings, in millimeters, shall be recorded for each temperature for the time intervals of 5, 10 and 30 minutes and reported, for example see Table 1 (data for additional time intervals can be gathered upon request). All data is measured in millimeters (mm) within 0.1 mm. The recorded value for each temperature/time observation shall be the average of the five cavities where the measurement of each cavity is the two longest perpendicular axial dimensions (vertical and horizontal). The undercut area is defined as the total area of the circular undercut pattern minus the area of the original cavity. The ice undercutting is calculated according to equations (2) (3) and (4). Present the graph of the ice undercutting area (mm^2) as a function of time (see Figure 2).

The Ice undercutting area for a given deicer is defined as: $IU = IU_e - A_s$ (Eq. 2)

where :

IU : Ice undercutting area for a given deicer (mm^2)

IU_e : Total area of circular undercut pattern at specified time interval (mm^2)

A_s : Initial area of original cavity (mm^2)