

Compound, Solid Runway and Taxiway Deicing/Anti-Icing

RATIONALE

This specification covers a deicing and anti-icing compound in the form of a solid. In addition, runway and taxiway deicing/anti-icing compounds have been found to catalytically oxidize aircraft carbon brakes and to corrode cadmium plate to varying extents. There is a need for users to understand the effect of these compounds on carbon brakes and cadmium plate. This specification requires testing and reporting of such.

1. SCOPE

1.1 Form

This specification covers a deicing and anti-icing compound in the form of a solid. Unless otherwise stated, all specifications referenced herein are latest (current) revision.

1.2 Application

These compounds have been used typically at airports on aircraft maneuvering areas, such as aprons, runways, and taxiways, for the prevention and removal of frozen deposits of snow, frost, and ice, but usage is not limited to such applications.

1.3 Precautions

1.3.1 Material Compatibilities

While this specification covers technical requirements for solid deicing/anti-icing compounds, it does not address the compatibility issue of combining deicers/anti-icers during the operational phase. Compounds meeting this specification are unique to each manufacturer and may be adversely affected by combining with other deicing/anti-icing products. It is the user's responsibility to become familiar with the safe and proper use of applying multiple deicers/anti-icers.

1.3.2 Pavement Friction Evaluation

Airport authorities should ascertain the friction coefficient of the runway after the application of a deicing/anti-icing compound prior to aircraft landing operations.

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## 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 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

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](http://www.sae.org).

AMS2470	Anodic Treatment of Aluminum Alloys, Chromic Acid Process
AMS2475	Protective Treatments, Magnesium Alloys
AMS2825	Material Safety Data Sheets
AMS4037	Aluminum Alloy, Sheet and Plate, 4.4Cu - 1.5Mg - 0.60Mn (2024; -T3 Flat Sheet, -T351 Plate), Solution Heat Treated
AMS4041	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
AMS4049	Aluminum Alloy, Sheet and Plate, Alclad, 5.6Zn - 2.5Mg - 1.6Cu - 0.23Cr (Alclad 7075; -T6 Sheet -T651 Plate), Solution and Precipitation Heat Treated
AMS4376	Plate, Magnesium Alloy, 3.0Al - 1.0Zn - 0.20Mn (AZ31B-H26), Cold Rolled and Partially Annealed
AMS4911	Titanium Alloy, Sheet, Strip, and Plate, 6Al - 4V, Annealed
AMS4916	Titanium Alloy Sheet, Strip, and Plate, 8Al - 1Mo - 1V, Duplex Annealed
AMS5045	Steel, Sheet and Strip, 0.25 Carbon, Maximum, Hard Temper
AIR5567	Test Method for Catalytic Carbon Brake Disk Oxidation
AIR6130	Cadmium Plate Cyclic Corrosion Test

## 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](http://www.astm.org).

- ASTM C 672 Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
- ASTM D 56 Standard Test Method for Flash Point by Tag Closed Tester
- ASTM D 1193 Reagent Water
- ASTM D 1568 Sampling and Chemical Analysis of Alkylbenzene Sulfonates
- ASTM E 70 pH of Aqueous Solutions with the Glass Electrode
- ASTM E 203 Water Using Karl Fischer Reagent
- ASTM F 483 Total Immersion Corrosion Test for Aircraft Maintenance Chemicals
- ASTM F 484 Stress Cracking of Acrylic Plastics in Contact with Liquid or Semi-Liquid Compounds
- ASTM F 485 Effects of Cleaners on Unpainted Aircraft Surfaces
- ASTM F 502 Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces
- ASTM F 519 Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals
- ASTM F 945 Stress Corrosion of Titanium Alloys by Aircraft Engine Cleaning Materials
- ASTM F 1104 Preparing Aircraft Cleaning Compounds, Liquid Type, Water Base, for Storage Stability Testing
- ASTM F 1110 Sandwich Corrosion Test
- ASTM F 1111 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, Sheet and Formed Parts, Modified Acrylic Base, Monolithic, Crack Propagation Resistant
- MIL-P-83310 Plastic Sheet, Polycarbonate, Transparent
- MIL-STD-2073-1 DoD Material Procedures for Development and Packaging Requirements

## 2.4 APHA Publications

Available from American Public Health Association, 800 1 Street, NW, Washington, DC2001-3710, Tel: 202-777-APHA, [www.apha.org](http://www.apha.org).

Standard Methods for the Examination of Water and Waste Water, Method 112A

## 2.5 United States Environmental Protection Agency

Available from U.S. Government Printing Office, Superintendent of Documents, Mail Stop: SSOP, Washington, DC 20402-932.

EPA Methods, US Title 40 Code of Federal Regulations (CFR) Parts 797.1300, Daphnid Acute Toxicity Test, and 797.1400, Fish Acute Toxicity Test

## 2.6 Organization for Economic Cooperation and Development

Available from Organization for Economic Co-operation and Development, 2 Rue Andre Pascal, 75775 Paris Cedex 16, France, Tel: +33 1 45 24 82 00, [www.oecd.org](http://www.oecd.org).

OECD Guidelines for Testing of Chemicals, Methods 202 and 203

## 2.7 LFV Test Method 2-98 Publications

Available from Swedish Civil Aviation Administration, LFV Teknik, Box 53, SE-190 45 Stockholm-Arlanda, Sweden.

## 2.8 International Organization for Standardization (ISO)

Available from International Organization for Standardization (ISO), 1 ch. de la Voie-Creuse, Case postale 56, CH-1211 Geneva 20, Switzerland, Tel. +41 22 919 02 11, [www.standardsinfo.net](http://www.standardsinfo.net).

ISO 5725 Accuracy (trueness and precision) of measurement methods and results

## 2.9 Federal Supply Classification (FSC) of Federal Catalog System

Available from US Government via [www.DATA.gov](http://www.DATA.gov).

FSC 6850 Miscellaneous Chemical Specialties

# 3. TECHNICAL REQUIREMENTS

## 3.1 Material

The composition of the compound may contain additives, such as corrosion inhibitors, coating agents, etc., as required to produce a product meeting the requirements of this specification.

### 3.1.1 Environmental Information

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

- 3.1.1.1 Biochemical Oxygen Demand (BOD) of the compound for 5-, 15-, and 20-day incubation periods. The test solutions shall be incubated at 68 °F (20 °C).
- 3.1.1.2 Total Oxygen Demand (TOD) or Chemical Oxygen Demand (COD) of the compound, expressed in kilograms of oxygen per kilograms of compound.
- 3.1.1.3 Percent biodegradation of compound for 5-, 15-, and 20-day incubation periods. Percent biodegradation can be approximated by dividing BODx100 by either TOD or COD.

#### 3.1.1.4 Aquatic Toxicity

Formulated compound shall be tested in accordance with EPA (40 Code of Federal Regulations (CFR) Parts 797.1300 and 797.1400) or OECD (Organization for Economic Cooperation and Development Guidelines for Testing of Chemicals, Methods 202 and 203) procedures 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 test species survive, shall be given in milligrams per liter.

#### 3.1.2 Trace Contaminants

Report the presence, in percentage by weight, of sulfur, halogens, phosphate, nitrate, and heavy metals (lead, chromium, cadmium, and mercury).

3.1.3 Vendor shall report the product chemical analysis, determined in accordance with a recognized method acceptable to purchaser; and total water content shall be determined in accordance with ASTM E 203.

3.1.4 Vendor shall provide a phase diagram relating product dilution to freezing point. Delivered product shall be within +7 °F (+4 °C) of the preproduction value.

#### 3.1.5 Appearance

The compound, as received by purchaser, shall be uniform, free-flowing, and free from foreign material detrimental to usage of the compound.

### 3.2 Physical Properties

The compound, as supplied by vendor, shall conform to the following requirements: tests shall be performed in accordance with specified test on the product as delivered by vendor, unless otherwise specified herein.

#### 3.2.1 pH

The compound, diluted with ASTM D 1193, Type IV water, to 15% by weight of solids taking into account water contained in the compound, shall be within  $\pm 0.5$  of the preproduction value established in 4.2.3, determined in accordance with ASTM E 70.

#### 3.2.2 Flash Point

The compound on a dry basis shall be not lower than 200 °F (93 °C), determined in accordance with ASTM D 56.

#### 3.2.3 Chloride Content

The level of soluble chloride on a dry basis shall not exceed 250 ppm, determined in accordance with APHA Standard Methods for the Examination of Water and Waste Water, Method 112A.

#### 3.2.4 Storage Stability

The compound, when stored in a closed container for at least one year in accordance with ASTM F 1104 shall not deliquesce or otherwise deteriorate.

#### 3.2.5 Effect on Transparent Plastics

3.2.5.1 The compound, diluted with ASTM D 1193, Type IV, water to 15% by weight of solids taking into account water contained in the compound, shall not craze, stain, or discolor Type C stretched acrylic plastic conforming to MIL-P-25690, determined in accordance with ASTM F 484.

3.2.5.2 The compound, diluted with ASTM D 1193, Type IV, water to 15% by weight of solids taking into account water contained in the compound, shall not craze, stain, or discolor MIL-P-83310 polycarbonate plastic, determined in accordance with ASTM F 484, except that the specimens shall be stressed for 30 minutes  $\pm$  2 to an outer fiber stress of 2000 psi (13.8 MPa).

### 3.2.6 Effect on Painted Surfaces

Compound, diluted with ASTM D 1193, Type IV, water to 15% by weight of solids taking into account water contained in the compound, shall neither decrease the paint film hardness by more than two pencil hardness levels nor shall it produce any streaking, discoloration, or blistering of the paint film, determined in accordance with ASTM F 502.

### 3.2.7 Effect on Unpainted Surfaces

Compound, diluted with ASTM D 1193, Type IV, water to 15% by weight of solids taking into account water contained in the compound, shall neither produce streaking nor leave stains which require polishing to remove, determined in accordance with ASTM F 485.

### 3.2.8 Effect on Runway Pavements

#### 3.2.8.1 Runway Concrete Surface 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 concrete shall:

Be air-entrained with an air content as specified in ASTM C 672. Have a minimum cement content of 510 pound/cubic yard  $\pm$  10 (302 kg/m<sup>3</sup>  $\pm$  6). Have a slump, 1.5 inches  $\pm$  0.5 (38 mm  $\pm$  13).

A 25% by volume solution of the deicer/anti-icer fluid as supplied by the manufacturer in commercial concentration in tap water shall be substituted for calcium chloride. Performing more than one freeze-thaw cycle per day is acceptable.

#### 3.2.8.2 Asphalt Concrete Degradation Resistance (Appendix A, valid for deicer/anti-icer products used in Europe)

Compound, diluted with tap water to 50% or regular highest concentration by weight of solid, shall be tested in accordance with LFV Method 2-98 (See Appendix A). The reduction in adhesion value of the runway asphalt concrete surface shall not be more than 50% of the adhesion value of the specimens not stored in deicing diluted compound. Adhesion values shall be determined and documented. The following test parameters shall be used:

1. Marshall test specimens with paving grade bitumen 160/220 [penetration value at 77 °F (25 °C) of 180 mm<sup>-1</sup>  $\pm$  10 and softening point 102 °F  $\pm$  1 (39 °C  $\pm$  1)] shall be used,
2. Binder content 5.7% by mass,
3. Maximum aggregate size 0.625 inch (16 mm), and distribution as shown in Figure 1:

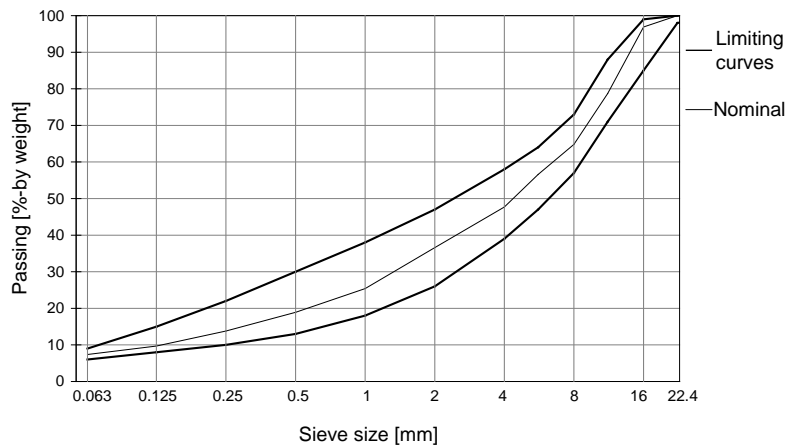


FIGURE 1 - AGGREGATE SIZE DISTRIBUTION

4. The aggregate used shall be characterized regarding variety of stone, origin and petrographical analysis. The aggregate shall be of good mechanical stability,
5. Air voids 7% ± 1 by volume,
6. Specimens are sawed to approximately 1.125 inch (30 mm) thickness.

3.2.9 Effect on Aircraft Metals

Compound, diluted with ASTM D 1193, Type IV, water to 5% and 15% by weight solids taking into account water contained in the compound, shall meet the following requirements:

3.2.9.1 Sandwich Corrosion

Specimens, after testing in accordance with ASTM F 1110, shall show corrosion not worse than control panels run using ASTM D 1193, Type IV water.

3.2.9.2 Total Immersion Corrosion

The compound, tested in accordance with ASTM F 483, except that panels shall be AMS4376 tested for 24 hours, shall neither cause corrosion of test panels nor a weight change of any test panel greater than shown in Table 1.

TABLE 1 - TOTAL IMMERSION CORROSION

Test Panel	Weight Change mg/cm <sup>2</sup> per 24 hours
AMS4037 Aluminum Alloy, anodized as in AMS2470	0.3
AMS4041 Aluminum Alloy	0.3
AMS4049 Aluminum Alloy	0.3
AMS4376 Magnesium Alloy, dichromate treated as in AMS2475	0.2
AMS4911 Titanium Alloy	0.1
AMS5045 Carbon Steel	0.8

### 3.2.9.3 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.

3.2.9.3.1 The compound shall be tested for cyclic immersion corrosion of cadmium plate in accordance with AIR6130 and the results reported as specified in Section 4 of AIR6130.

### 3.2.9.4 Hydrogen Embrittlement

The diluted compound shall be non-embrittling, determined in accordance with ASTM F 519, Type 1a, 1c, or 2a.

### 3.2.9.5 Stress Corrosion Resistance

The diluted compound shall not cause cracks in AMS4911 titanium alloy specimens, determined in accordance with ASTM F 945, Method A.

#### 3.2.9.5.1 Stress Corrosion Resistance

The diluted compound shall be tested in accordance with ASTM F 945, Method A using AMS4916 specimens. The results obtained from AMS4916 shall be reported for informational purposes only.

### 3.2.10 Performance

The compound, used in accordance with manufacturer's recommendation, shall remove accumulated frozen deposits of frost and ice from aircraft maneuvering areas, such as airport aprons, runways, and taxiways. The compound shall be tested in accordance with AIR6170 for ice melting effectiveness, with AIR6172 for ice undercutting effectiveness, and with AIR6211 for ice penetration effectiveness. Acceptance criteria shall be agreed upon by purchaser and vendor.

### 3.2.11 Effect on Carbon-Brake Systems

The compound shall be tested for catalytic oxidation of carbon in accordance with AIR5567 and the results shall be reported as shown in 4.2 of AIR5567. The results shall be reported for informational purposes only.

## 4. QUALITY ASSURANCE PROVISIONS

### 4.1 Responsibility for Inspection

The vendor of the compound shall supply all samples for conformance testing and shall be responsible for obtaining independent laboratory confirmation of conformance to the requirements of this specification. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the compound conform to specified requirements.

### 4.2 Classification of Tests

#### 4.2.1 Acceptance Tests

Total water content (3.1.3), pH (3.2.1), flash point (3.2.2), and chloride content (3.2.3) are acceptance tests and shall be performed on each lot.

#### 4.2.2 Periodic Tests

Effect on transparent plastics (3.2.5), effect on painted surfaces (3.2.6), effect on unpainted surfaces (3.2.7), runway concrete scaling resistance (3.2.8.1), asphalt concrete degradation resistance (3.2.8.2, Appendix A, valid for deicer/anti-icer products used in Europe) and effect on aircraft metals (3.2.9) are periodic tests and shall be performed at a frequency selected by the vendor unless frequency of testing is specified by purchaser, but in no case less than once every 2 years.



#### 4.2.3 Preproduction Tests

All technical requirements are preproduction tests and shall be performed prior to or on the initial shipment of the compound to a purchaser, when a change in ingredients and/or manufacturing procedures requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

#### 4.3 Sampling and Testing

Shall be in accordance with applicable requirements of ASTM D 1568; a lot shall be all compound produced in one continuous manufacturing operation from the same batches of raw materials and presented for vendor's inspection at one time.

4.3.1 When a statistical sampling plan has been agreed upon by purchaser and vendor, sampling shall be in accordance with such plan in lieu of sampling as in 4.3 and the report of 4.5 shall state that such plan was used.

#### 4.4 Approval

4.4.1 Sample compound shall be approved by purchaser before compound for production use is supplied, unless such approval be waived by purchaser. Results of tests on production compound shall be essentially equivalent to those on the approved sample.

4.4.2 Vendor shall use ingredients, manufacturing procedures, and methods of inspection on production compound which are essentially the same as those used on the approved sample compound. If necessary to make any change in ingredients or in manufacturing procedures, vendor shall submit for reapproval a statement of the proposed changes in ingredients and/or manufacturing procedures and, when requested, sample compound. Production compound made by the revised procedure shall not be shipped prior to receipt of approval.

#### 4.5 Reports

The vendor of compound shall furnish with each shipment a report showing the results of tests to determine conformance to all technical requirements. These tests shall be carried out by an independent facility. This report shall include the manufacturer's product identification, lot number, AMS1431D, purchase order number, and quantity.

4.5.1 A material safety data sheet conforming to ANSI Z400 (superseding AMS2825), or equivalent, shall be supplied to each purchaser prior to, or concurrent with, the report of preproduction test results or, if preproduction testing be waived by purchaser, concurrent with the first shipment of compound for production use. Each request for modification of compound formulation shall be accompanied by a revised material safety data sheet.

#### 4.6 Resampling and Retesting

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

### 5. PREPARATION FOR DELIVERY

#### 5.1 Packaging and Identification

5.1.1 Compound shall be packaged in clean containers of a type and size acceptable to purchaser and vendor.

5.1.2 Each container of compound shall be legibly marked with not less than AMS1431D, the phrase "FOR AIRFIELD USE", manufacturer's product identification, purchase order number, lot number, and quantity.

5.1.3 Labeling requirements shall meet all federal, state, and local laws. In the United States of America there are states whose Right to Know Regulations relate to labeling. Product manufactured, stored, or used in those states is subject to those regulations.

5.1.4 Containers of compound shall be prepared for shipment in accordance with commercial practice and in compliance with applicable rules and regulations pertaining to the handling, packaging, and transportation of the compound to ensure carrier acceptance and safe delivery.

## 6. ACKNOWLEDGMENT

A vendor shall mention this specification number and its revision letter in all quotations and when acknowledging purchase orders.

## 7. REJECTIONS

Compound not conforming to this specification, or to modifications authorized by purchaser, will be subject to rejection.

## 8. NOTES

8.1 A change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications, nor in documents that contain editorial changes only.

8.2 Dimensions and properties in inch/pound units and the Fahrenheit temperatures are primary; dimensions and properties in SI units and the Celsius temperatures are shown as the approximate equivalents of the primary units and are presented only for information.

8.3 Purchase documents should contain the following:

AMS1431D

Type of solid required or limitations (if applicable)

Size and type of containers desired (See 5.1.4)

Quantity desired

Packaging requirements (See 5.1.5).

8.4 Compound meeting the requirements of this specification has been classified under Federal Supply Classification (FSC) 6850.

PREPARED BY AMS COMMITTEE "G-12"

## APPENDIX A

This Appendix is valid for deicer/anti-icer products used in Europe. LFV Method 2-98 is a European standard method within CEN (European Committee for Standardization) named EN 12697-41.

LFV METHOD 2-98  
EFFECT OF DE-ICING FLUID ON THE SURFACE TENSILE STRENGTH  
OF ASPHALT CONCRETE FOR AIRFIELDS - ADHESION TEST

## A.1 INTRODUCTION

The purpose of the test is to determine the effect of storage in de-icing fluid on the surface tensile strength of asphalt concrete. The surface strength is the force in N/mm<sup>2</sup> required for failure to occur in the upper surface of the asphalt concrete under perpendicular "pull off" tension with an increase in tensile force of 200 N/s.

The test is performed largely in the same way as the method used for testing the adhesion of road markings to a road pavement and/or the adhesion of bridge deck waterproofing to an underlying concrete or steel surface.

## A.2 TEST METHODS

## A.2.1 Principle

Testing shall be performed on a sawn cylindrical test specimen on which a well-defined test surface has been carefully drilled out in the asphalt concrete to a depth of about 5 mm. A steel plate shall be bonded to the test surface. The specimen with test plate shall then be stored in de-icing fluid. During testing, the plate is pulled off with an increase in tensile force of 200 N/s, the force being applied perpendicularly to the test surface. The surface strength upon failure and the type of failure shall be recorded.

The results are compared with those for specimens which have not been stored in de-icing fluid.

## A.2.2 Apparatus and Materials

- a. Vessel with tight-fitting lid for storing specimens in de-icing fluid.
- b. Vacuum exsiccator.
- c. Vacuum pump for evacuation of the exsiccator. The pump shall be capable of achieving a pressure of 6.7 kPa within 10 minutes and maintaining this pressure (within  $\pm 0.3$  kPa) throughout the vacuum treatment.
- d. Manometer for measuring absolute pressure in the exsiccator.
- e. Approved equipment for laboratory mixing of bituminous asphalt mixture.
- f. Approved equipment for compaction of Marshall specimens or other approved laboratory compaction equipment such as gyratory compaction machine, roller or vibrating hammer.
- g. Circular steel plates with a diameter of 50 mm and a tolerance of 0.5 mm. The steel plate shall be attached by suitable means (e.g., screwed) to the tensile test machine. Minimum thickness of steel plate shall be 10 mm from bottom of steel plate to bottom of screw hole.
- h. Suitable adhesive (e.g., two part epoxy resin) for bonding the steel plates to the test specimen.
- i. Base and holder for fixing the specimen prior to testing (See Figure A1).
- j. Tensile test machine, with force increasing rate control and automatic load recording, fitted with suitable clampings and base to ensure that the tensile force can be applied without momentum perpendicular to the test specimen.

- k. Equipment for drilling out a test surface.
- l. Conditioning device giving a temperature of  $(23 \pm 1)$  °C.
- m. Circular saw capable of cutting asphalt with finish that has no imperfections discernible by touch.
- n. Heating cabinet giving a temperature of  $(40 \pm 2)$  °C for heated storage of specimens.
- o. Exsiccator grease.

#### A.2.3 Preparation of Test Specimens

Produce a number of specimens by compaction according to Marshall or other laboratory compaction method. The specimens should have a diameter of  $(100 \pm 5)$  mm and a height of  $(60 \pm 10)$  mm. The asphalt mix may be produced in an asphalt mixing plant or in the laboratory.

#### A.2.4 Determination of Dry Weight and Bulk Volume

Allow the specimens to reach room temperature. Mark them with a waterproof marking. Store the specimens overnight in room temperature on a flat surface. The next day, determine the bulk density for each specimen according to EN 12697-6. Divide the specimens into two equal groups (a wet and a dry group) with regard to bulk density. The mean bulk density must not differ by more than  $30 \text{ kg/m}^3$  between the groups.

#### A.2.5 Preparation of Test Surface

Saw the specimens in half and carefully drill a test surface with a diameter of 50 mm and a depth of about 5 mm approximately in the centre of the sawn surface of the specimen. Allow the specimens to dry on a flat surface at room temperature for at least three days. Bond the test plate to the test surface by carefully applying a thin layer of epoxy adhesive. Allow the specimen to cure at room temperature until the following day. Prepare the test surfaces of specimens from both groups.

#### A.2.6 Storage in De-icing Fluid

Store the specimens with bonded test plate from the wet group in de-icing liquid at  $(40 \pm 2)$  °C and perform the test at  $(23 \pm 1)$  °C.

Store the specimens in de-icing fluid, first for 3 hours  $\pm$  5 minutes under vacuum and room temperature, and then for a further 70 days  $\pm$  1 hour at normal pressure and specified storage temperature. Four specimens are normally stored for testing.

Place the specimens with the test plate upwards in the exsiccator. Pour de-icing fluid at room temperature into the exsiccator to a level 2 to 3 cm above the top of the asphalt concrete surface.

Evacuate to an absolute pressure of  $6.7 \text{ kPa} \pm 0.3$  within 10 minutes  $\pm$  1. Adjust the evacuation rate and pressure with a valve or rubber hose with clamp.

Keep the absolute pressure at  $(6.7 \pm 0.3)$  kPa for 3 hours. Turn off the pump and carefully admit air into the exsiccator until atmospheric pressure is reached.

Continue storage in a vessel at  $(40 \pm 2)$  °C for a further 70 days  $\pm$  1 hour. Here again, the specimens must be placed with the test plate upwards immersed in de-icing fluid to a level 2 to 3 cm above the top of the asphalt concrete specimen surface. During storage, the vessel shall be covered with a tight-fitting lid. At the same time, the group of dry specimens is stored on a flat surface at room temperature.

After storage, condition the specimens to test temperature in the de-icing fluid not longer than until the next day.

### A.2.7 Procedure

Take the specimen out of the de-icing fluid. Directly fix the specimen in the tensile test machine and the test plate attached to the machine. Apply the tensile force perpendicularly to the test surface and perform the test with an increase in tensile force of 200 N/s until failure occurs.

Record the tensile force together with the mode of failure. The following general modes of failure may occur:

- a. in the asphalt concrete, 5 mm or deeper;
- b. superficially in the asphalt concrete surface;
- c. adhesive failure.

The test is carried out at  $(23 \pm 1)$  °C.

At least three valid tests shall be carried out. The mean surface strength shall be calculated from a minimum of three accepted test results. Test results for specimens stored in de-icing fluid are compared to test results for not stored specimens.

### A.2.8 Expression of Results

#### A.2.8.1 Method of Calculation

The surface strength shall be calculated, to the nearest  $0.1 \text{ N/mm}^2$ , as the stress at maximum force by the following equation:

$$\sigma_{\max} = \frac{F_{\max}}{A} \quad (\text{Eq. A1})$$

where:

$\sigma_{\max}$  = surface strength at failure, in  $\text{N/mm}^2$

$F_{\max}$  = recorded maximum force, in N

$A$  = test area, in  $\text{mm}^2$

The mean value of the three test results shall be calculated.

#### A.2.8.2 Precision of the Test Method

Reproducibility and repeatability of the test method have been determined in accordance with ISO 5725 for seven laboratories using different equipment. The experiment was done on gyratory compacted test specimens with maximum particle size 16 mm and void content 7%. The origin of the aggregate was Skärlanda in Östergötland, the binder was a Laguna 160/220 and the nominal binder content was 5.7% by weight. Five different storage agents were used.

Results relating to  $\sigma_{\max}$  (6 laboratories, 1 excluded by statistical tests):

repeatability, standard deviation:  $s_r = 130$  N

repeatability, critical range at 95% confidence level when testing three specimens:  $CR_r = 430$  N

reproducibility, standard deviation:  $s_R = 220$  N

#### A.2.9 Test Report

The test report shall include at least the following information:

- a. all details necessary to identify the de-icing product tested (such as type, product name, density, pH value and concentration);
- b. a reference to this method and any deviation from it;
- c. information on preparation of test specimens in accordance with A.2.3, type of asphalt, including aggregate and bitumen designation;
- d. bulk density of all specimens and mean and SD for each group according to A.2.4;
- e. information on storage according to A.2.6;
- f. the test results and failure mode according to A.2.7 for each individual test, mean values;
- g. the dates of delivery and preparation of specimens;
- h. the date of tests.

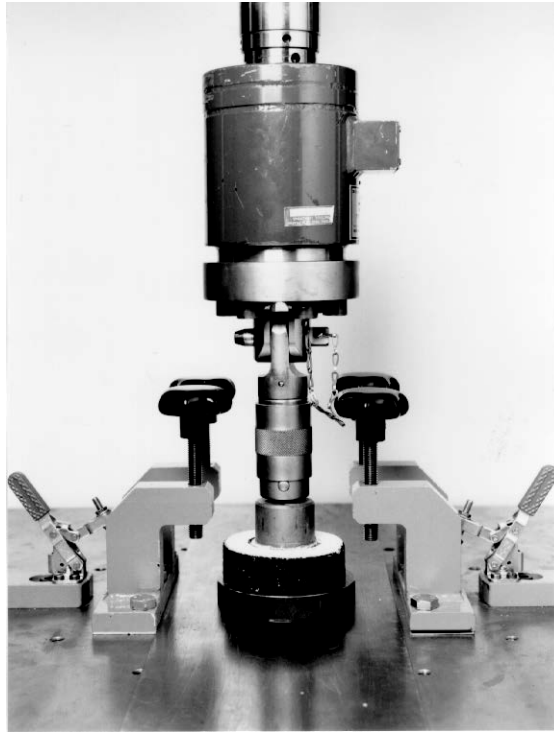


FIGURE A1 - EXAMPLE OF BASE, TEST SPECIMEN AND EQUIPMENT FOR ADHESION TESTING (ROAD MARKINGS)