
**Paints and varnishes — Coating
systems for wind-turbine rotor
blades —**

Part 6:
**Determination and evaluation of ice
adhesion using centrifuge**

*Peintures et vernis — Matériaux de revêtement pour pales de turbines
éoliennes —*

*Partie 6: Détermination et évaluation de l'adhésion de la glace à
l'aide d'une centrifugeuse*

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Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Apparatus	2
6 Sampling	2
7 Test panels	3
7.1 Substrate.....	3
7.2 Preparation.....	3
7.3 Conditioning.....	3
7.4 Coating thickness.....	3
7.5 Roughness of coating.....	4
8 Procedure	4
8.1 Number of determinations.....	4
8.2 Examination before exposure.....	4
8.3 Sample preparation (ice formation).....	4
8.4 Ice adhesion testing.....	5
8.5 Examination after testing.....	5
9 Evaluation	5
10 Precision	6
11 Test report	6
Annex A (informative) Example of parameters for ice formation processes	7
Bibliography	8

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

A list of all parts in the ISO 19392 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Ice accretion on rotor blade surfaces of wind turbines can decrease the efficiency and limit the performance of wind turbines in cold, humid environments. Ice formation can also lead to damage of the rotor blade and can be hazardous, if ice falls off the blade. Icephobic coatings (“icephobics”) can be applied to rotor blade surfaces to reduce or prevent the adhesion of ice by removing ice prior to reaching a critical ice mass for the rotor blades. They can also increase the efficiency of thermal ice protection systems.

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Paints and varnishes — Coating systems for wind-turbine rotor blades —

Part 6: Determination and evaluation of ice adhesion using centrifuge

1 Scope

This document describes a method to measure ice adhesion from artificial ice on test substrates by using a centrifuge. Basic ice types are defined and test parameters for the ice removal are described to achieve reproducibility of test results for ice adhesion measurements for rotor blade coatings. This document does not intend to provide fixed test parameter to account for the diversity of relevant icing scenarios in this field of application.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 4618, *Paints and varnishes — Vocabulary*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

SAE ARP 5905, *Calibration and Acceptance of Icing Wind Tunnels*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

static ice

ice created at a surface from liquid water with no dynamic effects

3.2

impact ice

ice that forms on surfaces from impacting supercooled water droplets (velocity >40 m/s)

3.3

icephobic coating

icephobic

coating or material that reduces or prevents ice formations and/or ice adhesion

4 Principle

Artificial ice is formed under controlled conditions on flat test surfaces to create a convenient ice mass for the ice adhesion strength measurement. The following ice types shall be used:

- a) static ice (produced by pouring deionized water into a mould to subsequently freeze the ice onto the test surface under controlled conditions);
- b) impact ice (created onto the test surface in an ice wind tunnel by supercooled water droplets at relevant velocity).

After ice formation, prepared test samples are transferred to a centrifuge, preferably situated in the same cold chamber to avoid any change in climatic conditions (e.g. temperature, humidity). The centrifuge with a specified acceleration rate uses centripetal forces to shear the ice from the test surface. The test is finished as soon as detachment of ice is detected (ice hits the centrifuge wall). The visual inspection of the de-iced test sample is mandatory to identify the apparent failure mode, which can be:

- adhesive, meaning there are no ice residues at the test surface (quantitative result); or
- cohesive, meaning there are ice residues at the test surface (qualitative result).

5 Apparatus

Ordinary laboratory apparatus, together with the following:

5.1 Climatic chamber for freezing conditions

Temperature controlled room, allowing storage, handling, and testing of equipment and test specimen at test temperature with a maximum allowable tolerance of ± 1 °C.

5.2 Ice wind tunnel (for “impact” ice formation only)

Open or closed loop facility, calibrated in accordance with SAE ARP 5905 and modified in order to create impact ice on a suitable test specimen for subsequent ice adhesion testing in the centrifuge.

5.3 Centrifuge

Custom-built device to perform the test under relevant temperature conditions and appropriate test parameters as follows:

- adjustable counterweight for relevant ice masses (ideally known prior testing) to prevent imbalance of the rotor;
- piezoelectric cell, vibration sensor, sound recordings or alike to detect the ice impact at the centrifuge wall.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

Examine and prepare each sample for testing, as described in ISO 1513.

7 Test panels

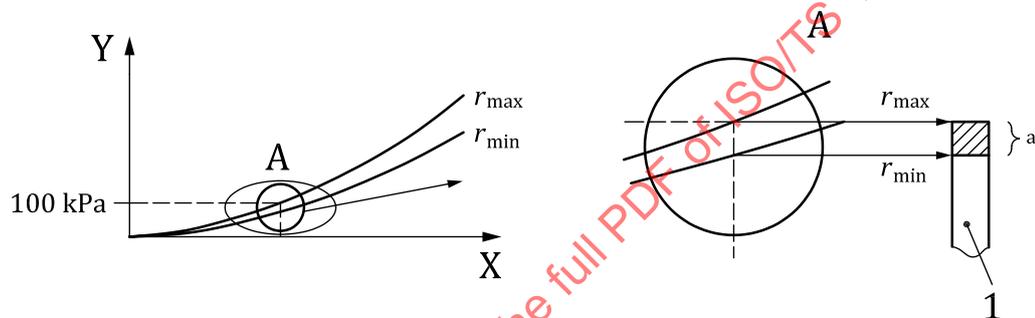
7.1 Substrate

Glass fibre composites or aluminium alloys are preferred.

Prepare and coat glass fibre test specimen similar to the production process with the coating system to be tested.

To account for effects of substrate properties on ice adhesion test results, specify the used substrate material and ensure comparability within the test series.

Sample size shall be adequate for the used centrifuge and allow an iced area, which guarantees reasonable result evaluation. Typically, areas range from minimum 5 cm² to maximum 12 cm². It shall also consider the minimization of non-uniform stress distribution during centrifuge test (see [Figure 1](#): the difference between the calculated adhesive strength 100 kPa at the maximum radius of the ice covered area should be ≤ 30 % if compared to the minimum radius of the ice covered area).



Key

X centrifuge speed

Y ice adhesion strength

r_{max} max. radius

r_{min} min. radius

1 test sample

A iced area

^a Iced area with ≤ 30 % difference for adhesive strength between min. and max. radius (at 100 kPa for the max radius).

Figure 1 — Maximum allowable difference of the adhesion strength within the iced area of the test sample

7.2 Preparation

Prepare and coat test specimen in accordance with the coating supplier's instructions.

7.3 Conditioning

Condition the coated test specimen in accordance with the coating supplier's instructions.

7.4 Coating thickness

If applicable, the thickness of the coating shall be specified.

7.5 Roughness of coating

The roughness of the coating/test surface shall be determined. In case of directional texture, consider direction of ice removal at the test surface.

8 Procedure

8.1 Number of determinations

Test at least three replicates of each material at each of the specified conditions.

For assessment of repeatability of de-icing on the same test surface, at least threefold icing/de-icing cycles are required. To address ice-phobic durability multiple icing/de-icing cycles are required.

8.2 Examination before exposure

Before sample preparation, carry out the following measurements:

- visual examination of the area to be exposed (no defects nor irregularities);
- surface roughness, in accordance with ISO 21920-2; incl. Ra, Rz;
- other surface topography parameters may also be measured.

8.3 Sample preparation (ice formation)

Prior to ice accretion, each surface shall be cleaned by using appropriate solvent (e.g. Isopropanol) and a soft tissue. For cleaning procedure follow the coating manufacturer instructions.

The conditioning and preparation of the test surfaces shall be dependent on the intended ice type, as follows:

- a) Static ice — use specified volume of deionized water to fill the mould / test specimen assembly at test temperature (see 8.4). Assure tight contact of the mould to avoid water spill. Place the assembly at specified test temperature and wait for thorough freezing. Record the resulting ice mass and freezing time.

Freezing time can affect test results and should be kept constant for the test series.

- b) Impact ice — cool down test specimen and relevant equipment to the specified test temperature. Start ice wind tunnel facility, including water injection using deionized water. Insert the pre-conditioned test surface in the running ice wind tunnel and collect impacting supercooled water droplets for a pre-defined time. Remove test specimen from the ice wind tunnel.

After ice formation, remove all additional equipment (e.g. mould, icing mask) from the test specimen, necessary for ice formation processes. No stress insertion shall occur to avoid preliminary ice breakage.

To minimize stress, moulds etc. may not be removed prior to testing. In this case, mould should not affect the ice adhesion measurement and shall be considered in the ice mass determination.

With regard to impact ice, prior to ice formation, determine the mass of each test specimen with an accuracy of $\pm 0,1$ g and record it in the test protocol. After ice formation, repeat this process and calculate the ice mass accordingly.

Handle the prepared samples with care and allow a conditioning time of minimum 15 min, but not longer than five hours prior to testing. Conditions for ice formation shall be reported. Examples are given in [Table A.1](#).

8.4 Ice adhesion testing

The prepared test specimen shall be handled with care, avoiding any bending or impacts that have negative effects on ice and ice / surface interface. Temperature fluctuations shall not exceed ± 1 °C.

Adjust the counterweight of the centrifuge rotor according to the ice mass (accuracy of minimum 0,25 g) or install the counterweight directly at the test sample, ideally before the ice formation.

Centrifuge basket shall have similar test temperature at the beginning of the test run. Introduce the test specimen in a way to ensure safe and balanced running of the centrifuge. Close the centrifuge and start the test programme with the appropriate parameters. [Table 1](#) gives the range of test parameter sets.

Table 1 — Range of test parameter for centrifuge testing

Test temperature	°C	-1 to -20
Area of ice formation	cm ²	5 to 12
Radius of test beam	cm	10 to 20
Acceleration of centrifuge rotor	s ⁻¹	30 to 300

8.5 Examination after testing

After centrifuge test, assess the following properties:

- visual examination of the de-iced area and determination of failure mode (adhesive / cohesive);
- any defects on the exposed area determined through a visual examination;
- temperature.

9 Evaluation

In case of adhesive failure mode, the ice adhesion strength can be calculated as the shear stress, τ , in Pascals, according to [Formula \(1\)](#):

$$\tau = \frac{m r \omega^2}{A} \quad (1)$$

where

m is the mass of ice, in kilograms;

r is the radius of test beam, in metres;

ω is the speed of rotation, in radiant per second;

A is the iced area, in square metres.

The use of the adhesion reduction factor (F_{ar}) may be necessary to account for the non-uniform stress distribution during the centrifuge test^[1]. In this case it is necessary to define and test a benchmark material and use [Formula \(2\)](#):

$$F_{ar} = \frac{\tau_{\text{benchmark}}}{\tau_{\text{test surface}}} \quad (2)$$

where

- F_{ar} is the adhesion reduction factor;
- $\tau_{\text{test surface}}$ is the shear stress of the test surface, in Pascals;
- $\tau_{\text{benchmark}}$ is the shear stress of the benchmark material, in Pascals.

Quantitative evaluation applies to test surfaces with adhesive failure mode. In case of cohesive failure with ice residues at the test surface, qualitative results can be expressed, allowing the conclusion that ice adhesion is stronger compared to cohesion of the used ice.

10 Precision

There are currently no general data on precision.

11 Test report

The test report shall contain at least the following information:

- a) a reference to this document, i.e. ISO 19392-6:2023;
- b) all details necessary for complete identification of the product tested (manufacturer, trade name, batch number, sample ID, etc.);
- c) details of the test specimens including:
 - 1) details of the substrate (including material and thickness) and its surface preparation, if performed by the test facility,
 - 2) the method used to apply the product to the substrate, including the drying or curing time and conditions for each layer, if performed by the test facility,
 - 3) if applicable, the conditions under which the panels were aged,
 - 4) details of how the test panels were conditioned before the test and, if applicable, details of any tests carried out previously with the same test panel,
 - 5) the roughness R_a and R_z of the coating, in micrometres,
 - 6) ice type and conditions for ice formation process (see [Table A.1](#));
- d) test parameters in accordance with [Table 1](#);
- e) the result of the test in accordance with [Clause 9](#);
- f) any deviation from the specified test method;
- g) details of any specific points on which agreement between the interested parties was necessary;
- h) any unusual features observed during the test;
- i) the date of testing.