SLOVENSKI STANDARD
SIST EN 2564:2018

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Nadomešča:
SIST EN 2564:2001

Aeronavtika - Laminati iz ogljikovih vlaken - Ugotavljanje deleža vlaken, smole in poroznosti

Aerospace series - Carbon fibre laminates - Determination of the fibre, resin and void contents

Luft- und Raumfahrt - Kohlenstoffaser-Laminate - Bestimmung der Faser-, Harz- und Porenanteile

Série aérospatiale - Stratifiés de fibres de carbone - Détermination de la teneur en fibres, en résine et du taux de porosité

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Aerospace series - Carbon fibre laminates - Determination of the fibre, resin and void contents

Série aérospatiale - Stratifiés de fibres de carbone - Détermination de la teneur en fibres, en résine et du taux de porosité

Luft- und Raumfahrt - Kohlenstoffaser-Laminate - Bestimmung der Faser-, Harz- und Porenanteile

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 2564:2018) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2019, and conflicting national standards shall be withdrawn at the latest by April 2019.

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1 Scope

This European Standard specifies the methods for determining the fibre content by volume and mass and, by correlation, the resin content by volume and mass and void content by volume, of carbon fibre laminates, for aerospace applications.

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.

EN 2743, *Aerospace series — Fibre reinforced plastics — Standard procedures for conditioning prior to testing unaged materials*

ISO 1183, *Plastics — Methods for determining the density and relative density of non-cellular plastics*

ISO 10119, *Carbon fibre — Determination of density*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

4 Principle

Determination of the difference in mass of specimens before and after extraction of the resin by sulphuric acid digestion.

Two methods are applicable:

— Method A is the reference method,

— Method B is a simplified method.

5 Apparatus and reagents

5.1 Balance accurate to ± 0,1 mg.

5.2 Tweezers.

5.3 Cutting device, such as a diamond blade saw or any other suitable apparatus.

5.4 250 ml double necked pear-shaped flask equipped with 50 ml dropping funnel, air inlet and a water pump.

5.5 Beakers of various capacities, including 400 ml.

5.6 20 ml sintered glass crucible (n° 2 porosity) and suitable filtration assembly.

5.7 Desiccator containing a suitable drying agent (for example silica gel, calcium chloride or phosphorus pentoxide).

5.8 Concentrated sulphuric acid (specific gravity: 1,84 to 1,89).

5.9 Hydrogen peroxide solution (300 g/l to 500 g/l).

5.10 Acetone (propanone).

5.11 Distilled water

5.12 Heat source with suitable temperature control.

5.13 Fume cupboard (for method B, see 9.2).

5.14 Thermometer capable of measuring 300 °C.

5.15 Sand bath or equal (for method B, see 9.2).

5.16 Electric oven capable of maintaining a temperature of (120 ± 5) °C.

5.17 Protective clothing, including rubber gloves, for handling hydrogen peroxide and sulphuric acid solutions.

4.18 Safety screen.

6 Specimens

6.1 Shape and dimensions

The samples size shall be adjusted to reach minimum 1 – 5 g sample weight.

Specimens of different dimensions may be used, subject to agreement between the user and manufacturer.

6.2 Number and distribution

If not other specified a minimum of three specimens shall be used. These shall be evenly distributed in the laminate and cut at least 10 mm from its edges.

7 Health, safety and environmental aspects

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

8 Atmosphere for testing

According to EN 2743 B.

9 Procedure

Use protective clothing and rubber gloves for handling hydrogen peroxide and sulphuric acid solutions.

Alternatively other acid´s can be agreed by the user and manufacturer, for example nitric acid.

Appropriate health and safety measures including modifications to the procedures below will need assessing and documenting by the test-house associated with the different solution.

The reactions shall be carried out behind the safety screen for eye protection.

9.1 Method A

9.1.1 Dry the specimens to constant mass in the desiccator that is until the difference in mass of two successive weighing is less than 2 mg. Record m_1.

9.1.2 Determine the density (ρ_c) of each specimen in accordance with ISO 1183, method A.

9.1.3 Drop the specimen into the double necked, pear-shaped flask and carefully pour 20 ml of concentrated sulphuric acid into the flask. Fit the dropping funnel to one neck and the water pump to the other.

9.1.4 Pour a suitable amount of the hydrogen peroxide solution (e.g. 20 ml of 500 g/l concentration or 40 ml of 300 g/l concentration) into the dropping funnel and heat the flask. Depending of the material it is recommended to use following temperatures:

— Resin systems cured at < 180 °C = 160 °C ± 20 °C.

— Resin systems cured at ≥ 180 °C = 220 °C ± 30 °C.

— Thermoplastics with melting temperature ≥ 250 = 260 °C ± 30 °C.

In case of not specified material the temperature of disaggregation should be used of 160 °C.

Discontinue heating of the flask and allow the hydrogen peroxide solution to drip into the acid at a rate of about one drop every 2 s, increasing to one drop per second after 5 min. If, after all the hydrogen peroxide solution has been used, the solution is still brown, add further 5 ml portions of hydrogen peroxide at the latter rate and continue heating until the solution becomes and remains clear.

9.1.5 Stop heating. When the neck of the flask is cool enough to handle, ensure the air inlet is open, turn the water pump off, remove the attachments from the flask and cool the flask and contents down to ambient temperature in running water.

9.1.6 Pour the contents of the flask into a 400 ml beaker containing 100 ml of distilled water; wash with distilled water any fibres adhering to the flask into this beaker.

9.1.7 Filter the contents of the beaker through the sintered glass crucible, previously dried and weighed (m_2), washing the beaker and contents of the crucible with distilled water until washings are free from acid, then wash with 10 ml of acetone.

9.1.8 Dry the crucible and contents at 120 °C for at least 45 min, cool in a desiccator for 20 min and weigh (m_3) to ± 1 mg.

9.2 Method B

The simplified method may be used in laboratories where a large number of tests are carried out provided that adequate safety precautions are in place.

The procedure is carried out in a fume cupboard, using open beakers instead of the pear-shaped flask, the beaker being heated on a sand bath. As safety feature reflux condenser could be used.

In this case, only 300 g/l hydrogen peroxide solution shall be used.

9.2.1 Dry the specimens to constant mass in the desiccator that is until the difference in mass of two successive weighing is less than 2 mg. Record m_1. As alternative drying conditions (e.g. 8 h at 50 °C) can be used after a validation of effectivity.

9.2.2 Determine the density (ρ_c) of each specimen in accordance with ISO 1183, method A.

9.2.3 Place each specimen in a beaker with concentrated sulphuric acid. The quantity of sulphuric acid should be adjusted. It is recommended to use:

— 20 ml sample weight < 1 g

— 50 ml sample weight 1 – 5 g

— 75 ml samples weight > 5 g.

9.2.4 Place each beaker on the sand bath or equal and start the heating.

The temperature of disaggregation has to be adjusted depending of the material.

It is recommended to use following temperatures:

— Resin systems cured at < 180 °C = 160 °C ± 20 °C.

— Resin systems cured at ≥ 180 °C = 220 °C ± 30 °C.

— Thermoplastics with melting temperature ≥ 250 = 260 °C ± 30 °C.

In case of not specified material the temperature of disaggregation should be used of 160 °C.

9.2.5 Heat until the resin begins to break down, which is shown by a black coloration of the sulphuric acid.

If there is a high weight of sample it would be best to carry on with heating approx. 30 minutes. Afterwards switch of the sand bath or equal and let the beaker cool down to ambient (warm to the touch)

Add hydrogen peroxide solution very slowly. The quantity of hydrogen peroxide solution should be adjusted. It is recommended to use:

— 25 ml samples weight < 1 g

— 30 ml samples weight 1 – 5 g

— 35 ml samples weight > 5 g

The solution becomes clear and the fibres rise to the surface. Start heating until the evolution of gas ceases and white fumes of sulphur trioxide re-appear. If the solution becomes dark again, repeat the operation.