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Textile-glass rovings — Determination of solubility of size

Stratifils (rovings) de verre textile — Détermination de la solubilité de l'ensimage

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 15039 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Textile-glass rovings — Determination of solubility of size

1 Scope

This International Standard specifies two methods for the determination of the percentage of size (coating) on the glass fibre that is soluble in acetone. These test methods are applicable to continuous rovings only.

2 Normative references

The following referenced documents are indispensable for the application 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 3344, *Reinforcement products — Determination of moisture content*

3 Principle

3.1 General

Two different methods are described in this International Standard, both using acetone as the solvent.

a) Method 1, extraction of the size from a specimen in a Soxhlet apparatus.

b) Method 2, marination at room temperature for a more extended period.

3.2 Solubility of the size

The solubility is determined as the difference between the amount of the size that is extracted by acetone during a given time and the total amount of the size that was originally coating the glass.

NOTE Interlaboratory tests have highlighted a difference between the averages of the results obtained from the two methods. A possible explanation for this difference is that the specimens are not dried prior to the extraction in method 1 (the Soxhlet extraction), while they are dried for method 2 (room-temperature marination). However, the distributions of the results about the mean are similar for the two test methods.

4 Apparatus

4.1 For method 1 — Soxhlet extraction

4.1.1 Soxhlet extractor, with a capacity of 100 cm^3 to 125 cm^3.

4.1.2 Erlenmeyer or round-bottom flask, with a capacity of 250 cm^3.

4.1.3 Electrical heating device (hotplate, heating mantle or oil bath).

4.1.4 Boiling chips, minimum five per Erlenmeyer (conical) or round-bottom flask.

4.2 For method 2 — Room-temperature marination

4.2.1 Glass flask, 1 000 cm^3 capacity.

4.3 Apparatus and materials required by either method

4.3.1 Acetone, minimum 98 % purity.

4.3.2 Plastic bag, capable of being sealed.

4.3.3 Scissors.

4.3.4 Test-specimen holder (tray or crucible for treatment in oven and furnace).

4.3.5 Analytical balance, of 100 g capacity, with a resolution of 0,1 mg and a limit of permissible error of 1 mg.

NOTE It is extremely important to make sure that all weight measurements are made on the same analytical balance, otherwise all the absolute errors of the balances will accumulate and affect the accuracy of the results.

4.3.6 Ventilated oven, capable of maintaining a temperature of 105 °C with an accuracy of $^{+5}_{-3}$ °C.

4.3.7 Desiccator, containing silica gel and a saturation indicator.

4.3.8 Muffle furnace, capable of maintaining a temperature of 625 °C with an accuracy of ± 20 °C.

4.3.9 Stop watch.

4.3.10 Safety can, to store used acetone for later disposal or recovery.

4.3.11 Absorbent paper towels.

5 Safety and environmental precautions

5.1 Acetone, like most organic solvents, is highly flammable. Its vapour forms explosive mixtures with air. Keep away from sparks and open flame. **NO SMOKING**.

5.2 Consult the applicable safety sheet for any additional information.

5.3 Store acetone solvent in an approved, labelled safety can. Collect used acetone in an approved, labelled safety can. Dispose of used acetone in accordance with laboratory or regulatory directives.

6 Procedure and calculation

6.1 Method 1 — Soxhlet extraction

6.1.1 Set up the Soxhlet extraction equipment as follows:

6.1.1.1 Make sure that each Erlenmeyer or round-bottom flask is filled up with 200 cm^3 of acetone and at least five boiling chips. Add additional acetone when necessary. Replace the acetone after five uses, or more frequently if significant contamination is observed.

6.1.1.2 Open the cooling water inlet. A cooling water flow indicator is desirable to show that cooling water is flowing.

6.1.1.3 Make sure that the Erlenmeyer or round-bottom flask is in contact with the heating element. Use a blank test to adjust the heating power to obtain a cycle time of (8 ± 1) min per cycle.

6.1.2 From the glass fibre roving to be tested, take two successive specimens of (25 ± 2) g.

6.1.3 Fold the first specimen into a length of approximately 75 mm and tie it together to give an open knot that is not too tight.

NOTE A tight knot would not allow complete extraction.

6.1.4 Using the second specimen, measure the percentage of moisture as per ISO 3344. If the determination of the moisture content is not started within 1 h after taking the specimens, seal this specimen into a plastic bag. It is highly recommended that the moisture test be done the same day. Express the result to the nearest $\pm$ 0,001 %.

6.1.5 Weigh the first specimen on the analytical balance to nearest 0,1 mg (m_0) and place it in the Soxhlet thimble, making sure that it will be fully immersed in the solvent during the successive cycles.

6.1.6 Let the solvent extract for 120 $^{+5}_{0}$ min, which should result in (15 $\pm$ 2) cycles of acetone renewal.

The 120-mm duration of the extraction has been found by experience to be sufficient to achieve practically complete (95 %) extraction of the size which is expected to be soluble in acetone for roving of the SMC type. A longer or shorter duration may be used, provided that a minimum of 95 % extraction can be verified. When the extraction duration is other than 120 min, it shall be noted in the test report.

6.1.7 After the specified time, turn the heater off, take the specimen out of the Soxhlet extractor, drain the acetone from the extractor and let the cooling water circulate for 20 min to 25 min. Gently remove the remaining acetone from the test specimen with paper towels.

6.1.8 Place the test specimen on a suitable tray and let it dry in the oven at 105 °C for (60 ± 5) min.

6.1.9 Let the test specimen cool down to room temperature for 30 min in the desiccator, and weigh immediately to the nearest $\pm$ 0,1 mg (m_1).

6.1.10 Place the test specimen in the furnace at 625 °C for (25 ± 5) min.

6.1.11 Let the test specimen cool down to room temperature for 30 min in the desiccator, and weigh immediately to the nearest $\pm$ 0,1 mg (m_2).

6.1.12 Calculate, to the nearest $\pm$ 0,1 mg, the dry mass, m_D, in grams, of the first specimen as follows:

$$m_D = m_0(1 - H/100)$$

where

m_0 is the initial mass of the first specimen;

H is the percentage moisture content determined using the second specimen.

6.1.13 Calculate the percentage solubility, S, of the size as follows:

$$S = \frac{m_D - m_1}{m_D - m_2} \times 100$$

where

m_1 is the mass of glass plus the non-soluble size remaining after extraction;

m_2 is the mass of the bare glass.

6.2 Method 2 — Room-temperature marination

6.2.1 From the roving to be tested, take three successive lengths, each weighing (20 ± 3) g. Taken together, the three lengths constitute the test specimen.

6.2.2 Fold each length so as to make a loose knot that can be placed on the balance and weighed without difficulty.

6.2.3 Place the test specimen (the three "knots") in the oven at 105 °C for 1 h $\pm$ 5 min.

6.2.4 Cool the specimen in the desiccator for at least 30 min, then weigh the specimen to the nearest 0,1 mg. Note the mass as $m_{0,RT}$.

6.2.5 Place the specimen in the 1 000 cm^3 flask (4.2.1) intended for the extraction; then pour in 700 cm^3 of acetone. Cover the flask and allow to extract for 24 h at (23 ± 3) °C.

6.2.6 Empty the acetone into the safety can, leaving the specimen in the flask.

6.2.7 Pour 300 cm^3 of acetone over the specimen, cover the flask and swirl. Empty the acetone into the safety can.

6.2.8 Gently remove the remaining acetone from the test specimen with paper towels. Dry the specimen in the oven at 105 °C for 1 h $\pm$ 5 min.

6.2.9 Cool the specimen in the desiccator for at least 30 min, then weigh the specimen to the nearest 0,1 mg. Note the mass as $m_{1,RT}$.

6.2.10 Place the specimen in the muffle furnace at 625 °C for (30 ± 5) min.

6.2.11 Cool the specimen in the desiccator for at least 30 min, then weigh the specimen to the nearest 0,1 mg. Note the mass as $m_{2,RT}$.

6.2.12 Calculate the percentage solubility, S, of the size as follows:

$$S = \frac{m_{0,RT} - m_{1,RT}}{m_{0,RT} - m_{2,RT}} \times 100$$

where

$m_{0,RT}$ is the initial mass, in grams, of the dry specimen (glass plus size);

$m_{1,RT}$ is the mass, in grams, of the specimen after extraction and drying;

$m_{0,RT}$ is the mass, in grams, of the specimen after calcination.

7 Test method precision

Inter-laboratory tests were conducted by six laboratories on three different products.

Tables 1 and 2 provide the mean values of the percentage solubility for the three different products and the repeatability and reproducibility standard deviations for the two methods.

Table 1 — Solubility by the Soxhlet extraction method

Soxhlet extraction method	Product A	Product B	Product C
Mean value of the solubility, S, in %	28,7	38,7	66,9
Repeatability standard deviation	0,63	1,87	1,19
Reproducibility standard deviation	0,96	2,60	1,96