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Polimerni materiali - Poliestri in epoksidne smole za vlivanje - Določanje celotnega volumskega skrčka

Plastics -- Polyester and epoxy casting resins -- Determination of total volume shrinkage

Matières plastiques -- Résines de polyesters et d'époxydes coulées -- Détermination du retrait global en volume

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INTERNATIONAL STANDARD ISO 3521

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Plastics — Polyester and epoxy casting resins — Determination of total volume shrinkage

Matières plastiques — Résines de polyesters et d'époxydes coulées — Détermination du retrait global en volume

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3521 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the Member Bodies in August 1974.

It has been approved by the Member Bodies of the following countries :

Belgium	Ireland	Spain
Brazil	Israel	Sweden
Chile	Netherlands	Switzerland
Czechoslovakia	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.A.
Hungary	Romania	U.S.S.R.
India	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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Plastics — Polyester and epoxy casting resins — Determination of total volume shrinkage

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the total volume shrinkage of polyester and epoxy casting resins.

2 PRINCIPLE

The density of the casting resin is determined

a) at the moment of mixing the components at the reaction temperature, but excluding reactive initiators (see note 1 in 5.1.2) in the measurement for unsaturated polyester resins;

b) after curing and conditioning to 23 °C.

The total volume shrinkage is calculated from the percentage change in density.

For components reacting at elevated temperatures, the density at the moment of mixing is determined by making buoyancy measurements of a sinker introduced into the reaction mixture over a period of time and extrapolating to zero time. For components reacting at room temperature, the density at the moment of mixing is determined by calculation from the individual densities at 23 °C.

The density after curing is determined, at 23 °C, by buoyancy measurements of a casting of the resin in silicone oil.

3 DEFINITION

For the purpose of this International Standard, the following definition applies :

total volume shrinkage of a resin compound : The sum of the shrinkage during curing of the resin compound and the shrinkage of the cured casting during the cooling from curing temperature down to room temperature.

4 APPARATUS

4.1 Balance, having an accuracy of ± 0,001 g, and fitted with a **device for measuring density** (preferably a high-speed balance with a wide range on a projected scale).

4.2 Sinker, of mass 25 ± 5 g, of known volume and coefficient of expansion at curing and room temperatures.

4.3 Thermostatically controlled bath of silicone oil of known density at curing and room temperatures.

4.4 Test tube, of length about 180 mm, and diameter 20 mm.

5 PROCEDURE

5.1 Determination of density of resin compounds that cure at temperatures higher than room temperature

Determine the mass of the sinker by weighing in air. Determine its exact volume at the curing temperature by a buoyancy method using silicone oil. Determine the mass of the suspension wire by weighing in air.

5.1.1 *Epoxy resins*

Heat the components of the resin compound separately to the reaction temperature and then mix, taking the moment of mixing of the components as zero time. Immediately after mixing, pour the mixture into the test tube. Then suspend the sinker (preheated to the curing temperature) in the resin compound and determine the apparent mass of the sinker, including the suspension wire, as a function of time (the measuring period depends on the resin compound to be investigated, and is usually about 1 h).

From the values obtained, determine graphically the apparent mass of the sinker, including the suspension wire, at zero time.

5.1.2 *Unsaturated polyester resins* (see note 2)

Mix the non-reacting (see note 1) resin components together and heat to the reaction temperature. Pour the mixture into the test tube. Then suspend the sinker (preheated to the curing temperature) in the non-reacting resin compound and determine the apparent mass of the sinker, including the suspension wire.

NOTES

1 Initiators used for curing unsaturated polyester resins, which are normally peroxide compounds, should not be heated, because of the danger of explosion.

2 As the level of initiator used in unsaturated polyester resin is small (1 to 2 %), the density of the liquid polyester resin, without initiator, is sufficiently accurate.

5.2 Determination of density of resin compounds which cure at room temperature

As it is difficult to carry out density measurements on resin compounds reacting at room temperature in view of their high reactivity and exothermic heat, determine the densities of the components of the resin compound separately at 23 ± 0,5 °C.

5.3 Determination of gross density of the casting

Place a test specimen (casting) weighing 25 ± 5 g in the test tube. After the specified curing time, weigh to an accuracy of 0,001 g. Then determine the apparent mass of the test specimen, including the suspension wire, in silicone oil at 23 ± 0,5 °C, and weigh the suspension wire used in air. After weighing, thoroughly clean the test specimen from adhering silicone oil using filter paper, wash with petroleum ether and post-cure for 1 h at 110 °C or at a different suitable temperature if indicated in a specification.

Again determine the apparent mass of the test specimen and suspension wire in silicone oil at 23 ± 0,5 °C. If the difference between their apparent mass in air and their apparent mass in silicone oil at 23 ± 0,5 °C is less than the value found prior to post-curing by 0,2 % or more of this earlier value, repeat the cleaning and post-curing operations until the difference obtained is less than 0,2 %.

6 EXPRESSION OF RESULTS

6.1 Compounds which cure at elevated temperatures (see 5.1)

The density of the resin compound, ρ_0, at zero time is given, in grams per millilitre, by the formula

$$\rho_0 = \frac{m_s + m_w - m_{(s+w)}}{V_s}$$

where

m_s is the mass, in grams, of the sinker, determined by weighing in air;

m_w is the mass, in grams, of the suspension wire, determined by weighing in air;

$m_{(s+w)}$ is the apparent mass, in grams, of the sinker and suspension wire in the resin compound at zero time;

V_s is the volume, in millilitres, of the sinker.

6.2 Compounds which cure at room temperature (see 5.2)

The density of the resin compound, ρ_0, at zero time is given by the formula

$$\rho_0 = \frac{(m_A + m_B) \times \rho_A \times \rho_B}{(m_A \times \rho_B) + (m_B \times \rho_A)}$$

where

m_A is the mass of component A used to prepare a casting;

m_B is the mass of component B used to prepare a casting;

ρ_A is the density of component A;

ρ_B is the density of component B.

6.3 Casting (see 5.3)

The gross density of the test specimen (casting), ρ_c, is given by the formula

$$\rho_c = \frac{m_c \rho_{si}}{m_c + m_w - m_{(c+w)}}$$

where

m_c is the mass of the cast test specimen, determined by weighing in air;

m_w is the mass of the suspension wire, determined by weighing in air;

$m_{(c+w)}$ is the apparent mass of the test specimen and suspension wire in silicone oil;

ρ_{si} is the density of the silicone oil.

6.4 Total volume shrinkage

The total volume shrinkage, S, during curing and cooling is given, as a percentage, by the formula

$$S = \frac{\rho_c - \rho_0}{\rho_c} \times 100$$

where

ρ_c is the gross density of the cast test specimen;

ρ_0 is the density of the resin compound at zero time at the curing temperature.

7 TEST REPORT

The test report shall include the following particulars :

a) a reference to this International Standard;

b) the type and characteristics of the resin compound tested;

c) the total volume shrinkage, expressed as a percentage;

d) any deviations from the procedure specified in this International Standard, and any unusual features observed during the determinations.