INTERNATIONAL STANDARD

ISO 3521

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Plastics — Unsaturated polyester and epoxy resins — Determination of overall volume shrinkage

Plastiques — Résines d'époxydes et de polyesters non saturés — Détermination du retrait global en volume

Reference number
ISO 3521:1997(E)

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.

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.

International Standard ISO 3521 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 3521:1976), of which it constitutes a technical revision.

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Plastics — Unsaturated polyester and epoxy resins — Determination of overall volume shrinkage

1 Scope

This International Standard specifies a method for the determination of the overall volume shrinkage of unsaturated polyester and epoxy resins.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1675:1985, *Plastics — Liquid resins — Determination of density by the pyknometer method.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 overall volume shrinkage: The sum of the shrinkage during curing and the shrinkage after curing of a casting when cooled to ambient temperature.

4 Principle

The overall volume shrinkage is calculated from the specific gravity of a last specimen before and after curing.

First, the specific gravity of the resin composition is determined

a) at the initial temperature of mixing of the components, excluding the initiators normally added to unsaturated polyesters (see 6.1.3, note 1);

b) at 23 °C after curing and conditioning of the last specimen.

The overall volume shrinkage is then calculated as a percentage of the change in the specific gravity before and after curing, as follows:

$$\text{Overall volume shrinkage} = \frac{\text{Uncured specific gravity} - \text{Cured specific gravity}}{\text{Uncured specific gravity}} \times 100$$

The specific gravity at the moment of mixing is determined for the mixed components at known intervals and the results extrapolated to zero time.

For components that react at elevated temperature, the specific gravity of the mixture is determined by calculation from the individual specific gravities of the components.

The specific gravity at 23 °C of the last specimen after curing and conditioning is determined by weighing in silicone oil.

5 Apparatus

5.1 Balance, accurate to 1 mg, with a device for measurement of specific gravity (if possible, a rapid-acting balance).

5.2 Sinker, weighing 25 g ± 5 g, and of dimensions such that wall effects are avoided. Its volume at the curing temperature and its volume at 23 °C ± 0,1 °C shall be known.

5.3 Temperature-stabilized silicone-oil bath, containing oil whose specific gravity at the hardening temperature and at 23 °C ± 0,1 °C is known.

5.4 Test tubes, length approximately 180 mm. diameter 20 mm.

5.5 Stopwatch.

5.6 Desiccator.

6 Procedure

6.1 Determination of specific gravity of compositions that cure at temperatures higher than ambient

6.1.1 Preparation of apparatus

Determine, to an accuracy of ± 10^{-3} ml, the volume V_s of the sinker at the curing temperature of the composition. Weigh the sinker in air at ambient temperature (mass m_s). Then weigh the sinker together with its suspension wire in silicone oil maintained at the curing temperature of the composition (mass m_{s+w}).

Determine the mass (mass m_w) of the suspension wire in air at ambient temperature.

If the specific gravity of the silicone oil at the curing temperature of the composition is not known, determine it in accordance with ISO 1675 but at the curing temperature.

6.1.2 Epoxy resins

Weigh out each of the components in the proportions used to form the composition, taking a quantity large enough to carry out the determination described below and to cast the test specimen prepared in 6.3.

Heat each of the components separately to the curing temperature. Mix the components, starting the stopwatch immediately after the introduction of the last component. This moment is taken as zero time. Continue to mix until a homogeneous mixture is obtained, then pour the mixture into the test tube all at once. Hang the sinker (preheated to the curing temperature) in the mixture and note the total mass (m_{s+w}), i.e. the apparent mass of the sinker and its suspension wire, after each of a series of equal intervals of time. The length of the time interval and the number of readings taken will depend on the composition being examined.

Proceed simultaneously with the casting of the test specimen prepared in 6.3, using the rest of the composition prepared.

Determine the mass (m_{S+W}) of the composition at zero time by extrapolation.

6.1.3 Unsaturated polyester resins

Weigh out each of the components, except initiators, in the proportions used to form the composition, taking a quantity large enough to carry out the test described below and to cast the test specimen prepared in 6.3.

NOTE — The initiators used in the curing of unsaturated polyester resins are normally peroxides that must not be heated, because of the risk of explosion (see Manufacturer's Safety Data Sheet). As the quantity of initiator used with unsaturated polyester resins is small (1 % to 2 %), the specific gravity of the other components in the mixture provides sufficient accuracy.

Mix the components weighed out.

Put aside the quantity of mixture needed to cast the test specimen (see 6.3) and heat the remainder of the mixture to the curing temperature. Start the stopwatch as soon as this temperature is reached (zero time).

Pour the mixture immediately into a test tube. Hang the sinker preheated to the curing temperature, in the mixture, and note the total mass (m_{S+W}), i.e. the apparent mass of the sinker and the suspension wire, after each of a series of equal intervals of time. The length of the time interval and the number of readings taken will depend on the composition being examined.

6.2 Determination of specific gravity of compositions that cure at ambient temperature

Since, because of the highly exothermic nature of the reaction, it is difficult to carry out measurements on compositions based on resins that react at ambient temperature, determine the specific gravity of each component of such compositions separately, in accordance with ISO 1675, at 23 °C ± 0,1 °C.

6.3 Determination of apparent specific gravity of cast specimen

6.3.1 Preliminary measurements

If the specific gravity of the silicone oil at 23 °C ± 0,1 °C is not known, determine it at that temperature in accordance with ISO 1675.

6.3.2 Measurement of mass and apparent mass of cast specimen

Place 25 g ± 5 g of the mixed composition in a test tube.

After the mixture has cured completely, cool the test tube containing the cast specimen in a desiccator to ambient temperature. The results of the test will depend essentially on the curing conditions, which shall be agreed between the interested parties. For any given composition, these conditions are the curing time and the curing temperature.

Weigh the test specimen in air (m_C).

Determine its apparent mass (m_{C+W}) in silicone oil at 23 °C ± 0,1 °C, then weigh the suspension wire in air (m_W).

Carry out the weighings in air and in silicone oil quickly to avoid humidity effects. When weighing the specimen in silicone oil, allow the specimen and bath to reach thermal equilibrium before reading off the mass.

When weighing is completed, clean the specimen carefully with filter paper to remove adhering silicone oil, wash it in petroleum ether and leave it to post-cure for 1 h at the curing temperature or the post-heating temperature, in accordance with the material specifications. (Use a temperature of 110 °C for compounds that harden at normal ambient temperature.)

Cool the specimen in a desiccator to ambient temperature.

Again weigh the specimen in air (mass m_c).

Again determine the apparent mass (m_{c+w}) in silicone oil at 23 °C ± 0,1 °C.

If the difference Δm between the total mass of the specimen plus suspension wire in air and the total apparent mass in silicone oil after post-curing is less than 0,2 % of the same difference before post-curing, take the apparent mass after post-curing for the calculation of the specific gravity.

If the values of the difference Δm differ by more the 0,2 %, clean the specimen and repeat the post-curing, using the conditions defined above, as many times as necessary to obtain values of Δm for two successive post-curings that differ by less than 0,2 %. Take the apparent mass determined after the last post-curing to calculate the specific gravity.

7 Expression of results

7.1 Compositions that cure at temperatures higher than ambient (see 6.1)

The specific gravity of the composition, ρ_0, in grams per millilitre, at zero time is given by the equation

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

where

m_s is the mass, in grams, of the sinker in air;

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

m_{s+w} is the apparent mass, in grams, of the sinker and suspension wire in the composition at zero time;

V_s is the volume, in millilitres, of the sinker, calculated from the equation

$$V_s = \frac{m_s + m_w - m'_{s+w}}{\rho'_{Si}}$$

m'_{s+w} being the apparent mass, in grams, of the sinker and suspension wire in silicone oil at the curing temperature of the composition;

ρ'_{Si} being the specific gravity of the silicone oil, in grams per millilitre, at the curing temperature of the composition;

m_s and m_w being as defined above.

7.2 Compositions that cure at ambient temperature (see 6.2)

The specific gravity of the composition ρ_0, in grams per millilitre, at zero time is given by the equation

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

where

m_A is the mass, in grams, of component A used in preparing the cast specimen;

m_B is the mass, in grams, of component B used in preparing the cast specimen;

ρ_A is the specific gravity, in grams per millilitre, of component A;

ρ_B is the specific gravity, in grams per millilitre, of component B.

7.3 Apparent specific gravity of cast specimen (see 6.3)

The specific gravity at 23 °C ± 0,1 °C of the test specimen, ρ_C, is given by the equation

$$\rho_C = \frac{m_C \times \rho_{Si}}{m_C + m_W - m_{C+W}}$$

where

m_C is the mass, in grams, of the specimen in air;

m_W is the mass, in grams, of the suspension wire in air;

m_{C+W} is the apparent mass, in grams, of the specimen and suspension wire in silicone oil;

ρ_{Si} is the specific gravity, in grams per millilitre, of the silicone oil at 23 °C ± 0,1 °C.

7.4 Overall volume shrinkage

The overall volume shrinkage is given, as a percentage, by the formula

$$\frac{\rho_C - \rho_0}{\rho_0} \times 100$$

where ρ_0 and ρ_C are as calculated above.

8 Precision

The precision of this test method is not known because interlaboratory data are not available. Interlaboratory data are being obtained and a precision statement will be added at the following revision.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the product examined;

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

d) details of any deviations from the procedure specified in this International Standard, and any unusual incidents observed during the determination.

ICS 83.080.10

Descriptors: plastics, polyester resins, epoxy resins, castings, tests, mechanical tests, determination, shrinkage.

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