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Plastics — Phenolic mouldings — Determination of acetone-soluble matter

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the amount of matter that can be extracted by acetone, at a temperature near its boiling point, from a finely ground sample of a phenolic moulding.

The test for acetone-soluble matter is one means for determining the relative degree of cure of phenolic mouldings made from a given material. The process of extraction is carried out under specified conditions for a specified time. Although a high proportion of soluble matter is determined, extraction is not necessarily complete. The results are only comparative, because the extracted material normally contains substances other than any uncured resin that may be present, for example lubricants, colouring matter and plasticizers.

2 PRINCIPLE

Hot extraction of acetone-soluble matter from a test portion which has been reduced to a finely divided state. Evaporation of the acetone, drying of the extract to constant mass and weighing of the dried extract.

3 REAGENT

Acetone, pure.

4 APPARATUS

4.1 Device for reducing the moulded sample to a finely divided state.

4.2 Sieve with nominal apertures of 425 μm , complying with ISO 565*.

4.3 Sieve with nominal apertures of 250 μm , complying with ISO 565*.

4.4 Analytical balance accurate to 0,0001 g.

4.5 Extraction apparatus of the type shown in the figure.

Alternatively, it is permissible to use a Soxhlet-type apparatus, in which the material in the thimble is surrounded by the vapour of the boiling solvent. Any other extraction apparatus may be used, provided that it can be shown to give similar results.

4.6 Oven, circulating air type, capable of being controlled at $50 \pm 2^\circ\text{C}$.

Dimensions in millimetres

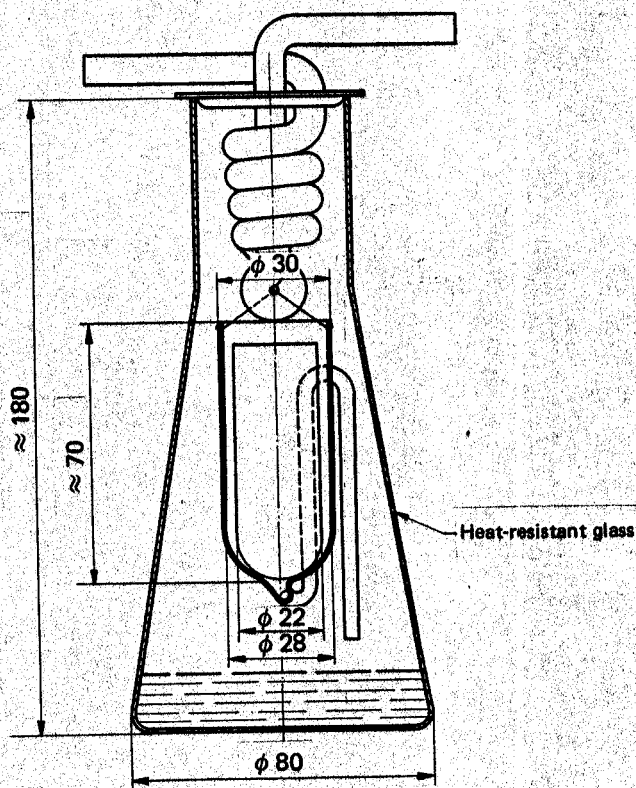


FIGURE — Extraction apparatus

* ISO 565, Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.

5 PREPARATION OF SAMPLE

Reduce a fully representative sample of the moulding to powder by filing, milling, grinding, turning or drilling, taking care that no undue heating of the material occurs. Sieve the powder using sieves (4.2) and (4.3), keeping for extraction only that fraction which passes through sieve (4.2) and is retained by sieve (4.3).

Take precautions to avoid absorption of moisture by the powdered sample.

6 PROCEDURE

6.1 Test two portions of the sieves material (clause 5).

6.2 Weigh a test portion of approximately 3 g to the nearest 0,001 g into a tared open-textured quantitative filter paper or a single-thickness extraction thimble (see 4.5).

6.3 After folding over the thimble or filter paper containing the test portion so that none of the powder can float out, place it in the siphon of the extraction apparatus. Assemble the condenser, siphon tube and flask to which 50 ml of pure acetone (clause 3) has been added.

6.4 Regulate the heating so that siphoning takes place at a rate of 20 to 30 times per hour and continue the extraction for 6 h. At the end of this time remove the flask and pour the contents into a smaller flask or a small dish which has been weighed to the nearest 0,001 g. Wash the empty flask with about 20 ml of acetone and add the washings to the extract.

6.5 Evaporate the acetone by any convenient means, taking care that the temperature does not exceed 50 °C.

Place the vessel containing the residue in the oven (4.6) at a temperature of 50 ± 2 °C for 30 min. Then withdraw the vessel from the oven, allow to cool in a desiccator to room temperature and weigh. Repeat the operations of heating, cooling and weighing until constant mass is obtained, i.e. until the difference between two successive weighings does not exceed 0,003 g.

7 EXPRESSION OF RESULTS

The amount of acetone-soluble matter in the sample is given, as a percentage by mass, by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dry extract.

Take the arithmetic mean of the values obtained from the two test portions as the amount of acetone-soluble matter in the moulding under test.

8 TEST REPORT

The test report shall include the following particulars:

- reference to this International Standard;
- ~~all information necessary for the~~ complete identification of the sample; H3
- method used to reduce the moulding to a fine state of division;
- amount of acetone-soluble matter in each test portion;
- arithmetic mean of the values obtained from the two test portions;
- date of test.

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