International Standard 308

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Phenolic moulding materials — Determination of acetone-soluble matter (apparent resin content of material in the unmoulded state)

Plastiques — Matières à mouler à base de phénoplastes — Détermination des matières solubles dans l'acétone (teneur apparente en résine des matières à l'état non moulé)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 308 was developed by Technical Committee ISO/TC 61, *Plastics*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 308-1976), which had been approved by the member bodies of the following countries :

Australia	India	Spain
Austria	Israel	Sweden
Belgium	Italy	Switzerland
Chile	Japan	Turkey
Czechoslovakia	Netherlands	United Kingdom
France	New Zealand	USA
Germany, F. R.	Poland	USSR
Hungary	Romania	

No member body had expressed disapproval of the document.

Plastics — Phenolic moulding materials — Determination of acetone-soluble matter (apparent resin content of material in the unmoulded state)

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 sample of finely divided phenolic moulding material. The method applies only to moulding materials based upon novolak resins and not to those based upon resols, as the latter type of resin may not be completely soluble in acetone.

In this International Standard, the amount of acetone-soluble matter is reported as the apparent resin content because although the extract consists mainly of phenolic resin and hexamine, other acetone-soluble components such as lubricants and colorants or natural resins from the filler are normally also present and will therefore be reported as resin.

2 Principle

Hot extraction of the acetone-soluble matter from a finely divided test portion. Drying of the extract under controlled conditions and weighing of the dry extract.

3 Reagent

Acetone, pure.

4 Apparatus

4.1 Device, for reducing coarse materials to a finer state of division.

4.2 Balance, accurate to 0,001 g.

4.3 Extraction apparatus, of the type shown in the figure. (A glass filter crucible may be used instead of a single-thickness extraction thimble.)

It is permissible to use a modified Soxhlet apparatus, provided that the material in the extraction thimble is surrounded by the vapour of the solvent at its boiling point. Any other extraction apparatus may be used, provided that it can be shown to give similar results.

4.4 Drying oven, capable of being controlled at approximately 105 °C.

4.5 Desiccator.

4.6 Weighing bottle, with ground glass stopper.

NOTE — The single-thickness extraction thimble free from acetone-soluble matter together with a loose plug of cotton wool, if used, also free from acetone-soluble matter, should be dried for 2 h in the oven (4.4) at 105 °C approximately and stored in the desiccator (4.5) until required.

Dimensions in millimetres

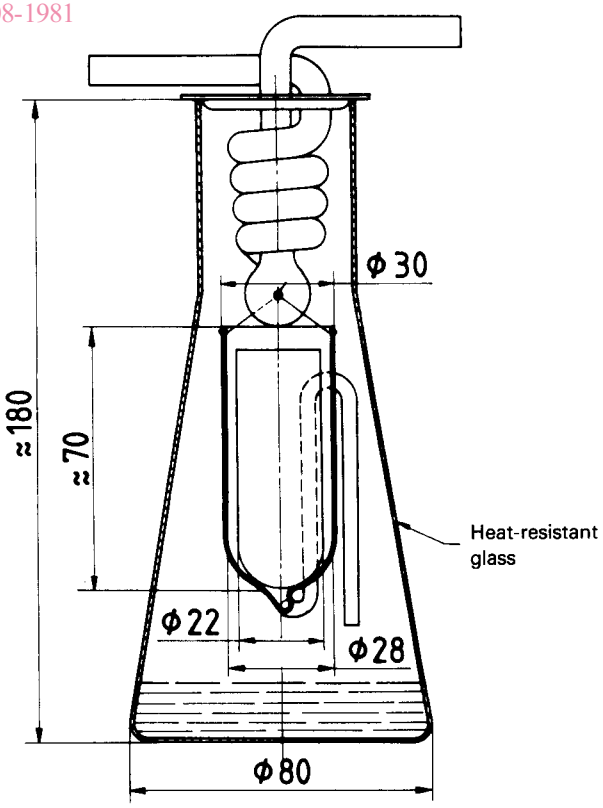

Figure — Extraction apparatus

5 Preparation of sample

5.1 Take a fully representative sample of the moulding material. If the material is in the form of preforms, flakes, coarse pieces or sheet (felted, oriented or woven), reduce it to powder or small pieces using the device (4.1) before the test, taking care to avoid overheating. The thickness of the particles obtained shall not exceed 1,5 mm and their other dimensions shall not exceed 5 mm. The sample should not be ground too finely or it may tend to agglomerate in the extraction thimble. Take care that no resin is lost while the sample is being reduced to powder or small pieces.

5.2 Dry at least 6 g of the material at room temperature, in vacuo over a sulphuric acid solution, ϱ 1,84 g/ml, or other desiccant, for 24 h.

6 Procedure

6.1 Carry out the test on two test portions of the dried sample (clause 5).

6.2 Quickly transfer the dried extraction thimble from the desiccator (see the note to clause 4) to the weighing bottle (4.6), close the weighing bottle with the stopper and weigh to the nearest 0,001 g on the balance (4.2). Remove the stopper from the weighing bottle and place a test portion of approximately 3 g of the dried sample in the extraction thimble. Replace the stopper in the weighing bottle and weigh to the nearest 0,001 g.

NOTE — If it is desired to know the mass of the empty extraction thimble or to avoid repeating the test in case of breakage, the weighing bottle may be tared or may be weighed separately.

6.3 After folding over the extraction thimble or closing it with a loose plug of absorbent cotton wool, so that none of the material can float out, place it in the siphon tube of the extraction apparatus (4.3). Assemble the condenser, siphon tube and flask to which 100 ml of acetone (clause 3) has been added.

6.4 Regulate the heating so that siphoning takes place at a rate of 15 to 30 times per hour, and continue the extraction for 16 ± 0,5 h. At the end of this time, dry the extraction thimble and contents at room temperature, in vacuo over a sulphuric acid solution, ϱ 1,84 g/ml, or other desiccant, for 24 ± 1 h and then weigh in the same weighing bottle to the nearest 0,001 g.

7 Expression of results

The amount of acetone-soluble matter in the sample (apparent amount of resin in the unmoulded material) expressed, as a percentage by mass, is given by the formula

$$\frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the extraction thimble and weighing bottle;

m_1 is the mass, in grams, of the extraction thimble, weighing bottle and test portion before extraction;

m_2 is the mass, in grams, of the extraction thimble, weighing bottle and test portion after extraction.

Take the arithmetic mean of the values obtained from the two test portions as the apparent amount of resin in the material under test, provided that these values do not differ by more than 2,0 % (in absolute value).

8 Test report

The test report shall contain the following particulars :

a) reference to this International Standard;

b) complete identification of the sample;

c) method used to reduce the material to a finely divided state;

d) apparent amount of resin in each test portion;

e) arithmetic mean of the values obtained from the two test portions;

f) date of test.