INTERNATIONAL STANDARD

ISO 308

Third edition
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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)

Reference number
ISO 308:1994(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 308 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This third edition cancels and replaces the second edition (ISO 308:1981), of which it constitutes a minor revision.

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

1 Scope

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 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary.*

ISO 800:1992, *Plastics — Phenolic moulding materials — Specification.*

3 Definitions

For the purposes of this International Standard, the definitions of moulding materials based on phenolic resins, novolak resins and resol resins given in ISO 472 and ISO 800 apply.

4 Principle

The acetone-soluble matter is extracted, using hot acetone, from a finely divided test portion. The extract is dried under controlled conditions and weighed.

5 Reagent

5.1 Acetone, pure.

6 Apparatus

6.1 Reduction device, for reducing coarse materials to a finer state of division.

6.2 Balance, accurate to 1 mg.

6.3 Extraction apparatus, of the type shown in figure 1. (A glass filter crucible may be used instead of a single-thickness extraction thimble.)

The single-thickness extraction thimble, which shall be free from acetone-soluble matter, together with a loose plug of cotton wool, if used, which shall also be free from acetone-soluble matter, shall be dried for 2 h in the oven (6.4) at approximately 105 °C and stored in the desiccator (6.5) until required.

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.

6.4 Drying oven, capable of being maintained at approximately 105 °C.

6.5 Desiccator.

6.6 Weighing bottle, with ground-glass stopper.

7 Preparation of sample

7.1 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 reduction device (6.1) 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

Dimensions in millimetres

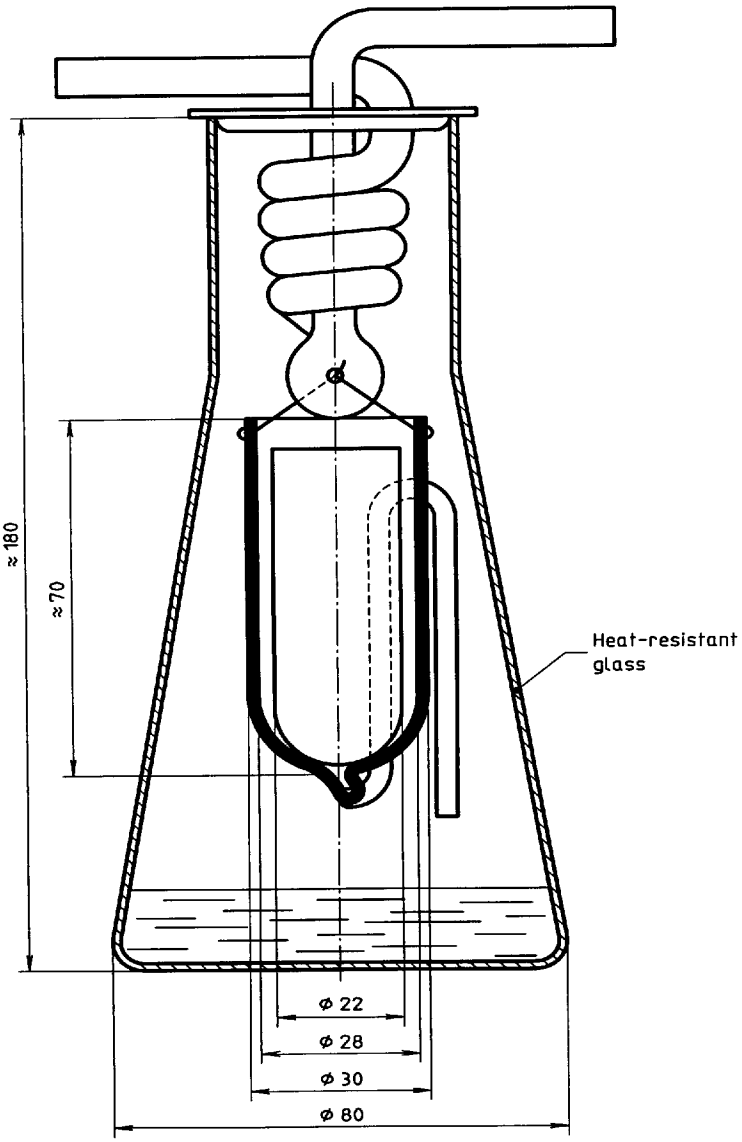

Figure 1 — Extraction apparatus

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.

7.2 Dry at least 6 g of the material at room temperature, *in vacuo* over ρ 1,84 g/ml sulfuric acid or another desiccant, for 24 h.

8 Procedure

8.1 Carry out the test on two test portions of the dried sample (see clause 7).

8.2 Quickly transfer the dried extraction thimble (see 6.3) from the desiccator (6.5) to the weighing bottle (6.6), close the weighing bottle with the stopper and weigh to the nearest 1 mg on the balance (6.2). 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 1 mg.

NOTE 1 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.

8.3 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 (6.3). Assemble the condenser, siphon tube and flask to which 100 ml of acetone (5.1) has been added.

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

9 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).

10 Test report

The test report shall contain the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

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

d) the apparent amount of resin in each test portion;

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

f) the date of the test.

ICS 83.080.10

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