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Plastics — Aqueous dispersions of polymers and copolymers — Determination of residue at 105 °C

Plastiques — Dispersions aqueuses de polymères et copolymères — Détermination du résidu à 105 °C

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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 1625 was developed by Technical Committee ISO/TC 61, *Plastics*.

It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 1625-1970, which had been approved by the member bodies of the following countries :

Austria	Iran	South Africa, Rep. of
Belgium	Israel	Sweden
Czechoslovakia	Italy	Switzerland
Egypt, Arab Rep. of	Japan	Turkey
France	Korea, Rep. of	United Kingdom
Germany	Netherlands	U.S.A.
Hungary	Poland	U.S.S.R.
India	Portugal	

No member body had expressed disapproval of the document.

Plastics — Aqueous dispersions of polymers and copolymers — Determination of residue at 105 °C

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of residue of aqueous dispersions at 105 °C.

The procedure is suitable for all aqueous polymer and copolymer dispersions which are chemically stable at the test temperature and which do not contain other volatile matter such as organic solvents.

The residue of unplasticized aqueous polymer and copolymer dispersions consists essentially of the polymer or copolymer and of a small quantity of additives, such as emulsifiers, protective colloids, etc.

For plasticized dispersions, the plasticizer is included in the residue.

Under the conditions of temperature and time adopted in this method, the product should not show any thermal degradation; otherwise different conditions should be used and specified in the test report.

2 PRINCIPLE

Stoving at 105 °C, for 1 h, of a test portion spread in a thin, even layer, and weighing of the residue obtained.

3 APPARATUS

3.1 Spreading and evaporation apparatus

It is essential to obtain an evenly spread film of about 0,15 mm, for which one of the following devices is suitable.

3.1.1 Apparatus A

The unit illustrated in figures 1, 2 and 3, consisting of two glass plates of diameter about 60 mm, one of the plates being provided with a support for the other one. The inside circular surfaces shall be perfectly flat and smooth.

3.1.2 Apparatus B

Aluminium foils about 0,1 mm thick, cut into rectangles of about 60 mm × 120 mm.

NOTE — The apparatuses described in 3.1.1 and 3.1.2 are recommended particularly for very viscous dispersions, because they are capable of spreading the film automatically

3.1.3 Apparatus C

Metal or glass dish of about 70 mm diameter, having a rim with a minimum height of 3 mm.

NOTE — This apparatus is recommended particularly for very fluid dispersions, because it avoids any overflow.

3.2 Oven, with natural air ventilation, capable of being controlled at 105 ± 2 °C.

3.3 Desiccator containing a suitable desiccant, for example calcium chloride or silica gel.

3.4 Balance, accurate to 0,000 1 g.

4 PROCEDURE

4.1 Using apparatus A

4.1.1 Place the two parts of the unit (3.1.1), arranged as shown in figure 3, in the oven (3.2), controlled at 105 ± 2 °C, and leave them for about 30 min. Allow them to cool in the desiccator (3.3) for about 30 min, then weigh the unit to the nearest 0,000 1 g.

4.1.2 With the aid of a glass rod or a small spatula, pour $1 \pm 0,2$ g of the dispersion being tested onto the centre of the lower plate. This operation is easier if the upper plate is removed from its support.

Put the upper plate on the lower one, pressing it gently. The dispersion squeezed between the two plates spreads evenly. The diameter of the plates is such that if the quantity of dispersion specified above has been poured onto the centre of the lower plate, overflow of dispersion can be avoided.

If the dispersion is very fluid, however, check that no overflow takes place.

Weigh the assembly, leaving the plates one upon the other, to the nearest 0,000 1 g.

4.1.3 Separate the two plates and suspend the upper one on its support.

Place the unit in the oven, controlled at 105 ± 2 °C, and leave it there for $1 \text{ h} \pm 5 \text{ min}$.

4.1.4 Remove the unit from the oven, place it in the desiccator and allow it to cool for about 30 min.

Weigh the unit to the nearest 0,000 1 g, with the upper plate suspended on its support.

4.2 Using apparatus B

4.2.1 Leave the aluminium rectangle (3.1.2) in the oven (3.2), controlled at $105 \pm 2^\circ\text{C}$, for about 30 min. Allow it to cool in the desiccator (3.3) for about 30 min, then weigh it to the nearest 0,000 1 g.

4.2.2 Fold the rectangle double so as to form approximately a square; mark the fold, then unfold the sheet.

Pour in the middle of one square $1,0 \pm 0,2$ g of the dispersion being tested; promptly fold the sheet again and spread the material as evenly as possible, pressing gently with the fingers, taking care not to let the dispersion flow over the outer edges.

Weigh the whole to the nearest 0,000 1 g.

4.2.3 Open out the sheet completely, place it in the oven, controlled at $105 \pm 2^\circ\text{C}$, and leave it there for $1 \text{ h} \pm 5 \text{ min}$.

4.2.4 Remove the sheet from the oven, place it in the desiccator and allow it to cool about for 30 min.

Weigh the sheet to the nearest 0,000 1 g.

4.3 Using apparatus C

4.3.1 Leave the dish (3.1.3) in the oven (3.2), controlled at $105 \pm 2^\circ\text{C}$, for about 30 min. Allow it to cool in the desiccator (3.3) for about 30 min, then weigh it to the nearest 0,000 1 g.

4.3.2 Pour $1 \pm 0,2$ g of the dispersion to be tested into the dish.

Rapidly weigh the whole to the nearest 0,000 1 g.

4.3.3 Spread the dispersion as far as possible over the whole surface of the dish by manual swirling.

4.3.4 Place the dish in the oven, controlled at $105 \pm 2^\circ\text{C}$, and leave it there for $1 \text{ h} \pm 5 \text{ min}$.

4.3.5 Remove the dish from the oven. Make sure that the spreading of the dispersion has been complete. If not, it is advisable to carry out the test using one of the other two types of apparatus.

Allow the dish to cool in the desiccator for about 30 min.

4.3.6 Weigh the dish to the nearest 0,000 1 g.

5 EXPRESSION OF RESULTS

5.1 The residue at 105°C of the tested dispersion, expressed as a percentage of the original mass, is given by the formula

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

where

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

m_1 is the mass, in grams, of residue after stoving.

5.2 Make two determinations. The results should agree within 0,5 % absolute value. If they do not, carry out additional duplicate determinations until two successive results satisfy this condition.

5.3 Give as the final result the average of the two determinations.

6 TEST REPORT

The test report shall include the following particulars :

- a) identification characteristics of the product tested;
- b) apparatus used (A, B, or C);
- c) residue.

Apparatus A

Dimensions in millimetres

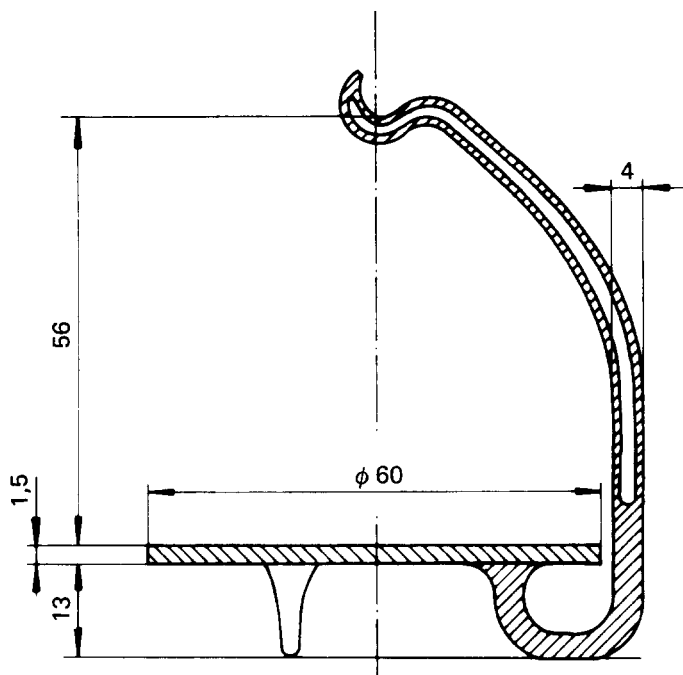


FIGURE 1 — Lower glass disc

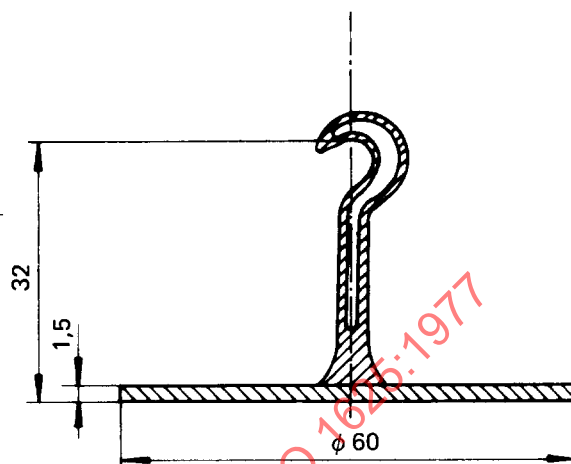


FIGURE 2 — Upper glass disc

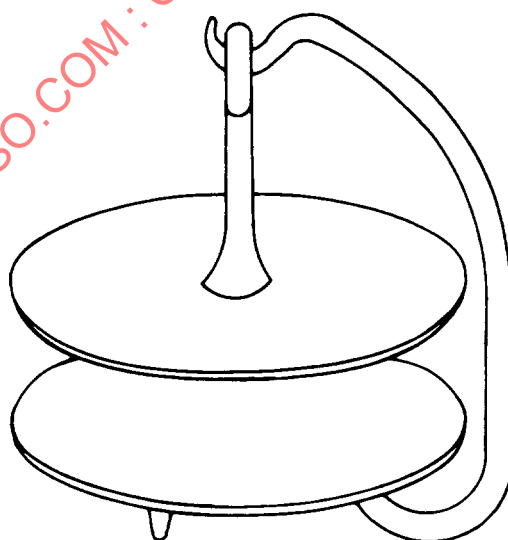


FIGURE 3 — Arrangement of discs

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