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Plastics — Preparation of PVC pastes for test purposes — Dissolver method

Plastiques — Préparation des pâtes PVC pour essai — Méthode utilisant un dispositif pour dissolution

Reference number
ISO 11468:1997(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 11468 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

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INTERNATIONAL STANDARD © ISO **ISO 11468:1997(E)**

Plastics — Preparation of PVC pastes for test purposes — Dissolver method

1 Scope

This International Standard specifies a method, using a dissolver, for the preparation of PVC pastes made from polymerized vinyl chloride (VC). Such PVC pastes are used to characterize the various VC polymers with reference to their rheological properties, as well as being a means of establishing consistency between various deliveries.

The method can also be used to prepare pastes of other compositions (for example with stabilizers) but such pastes cannot be used for resin designation purposes.

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 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 1060-2:—[1], *Plastics — Homopolymer and copolymer resins of vinyl chloride — Part 2: Preparation of test specimens and determination of properties.*

3 Principle

A PVC paste is prepared by mixing a PVC resin and a plasticizer in a dissolver.

For the purposes of this International Standard, a plastisol is defined as a liquid PVC composition consisting of a suspension of PVC resin in a liquid plasticizer.

4 Apparatus and materials[2]

4.1 Dissolver, consisting of a cylindrical mixing vessel, preferably double-walled, and a motor-driven spindle to which is attached a horizontal coarsely toothed mixing disc (dissolver disc). The dissolver disc transfers energy to the PVC paste, causing the paste ingredients to be driven centrally downwards along the spindle axis and upwards along the walls of the mixing vessel (see figure 1).

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1) To be published. (Revision of ISO 1060-2:1985)

2) Suitable equipment is available from VMA-Getzmann GmbH, Verfahrenstechnik, Euelerhammerstr. 13, 51580 Reichshof, Germany.

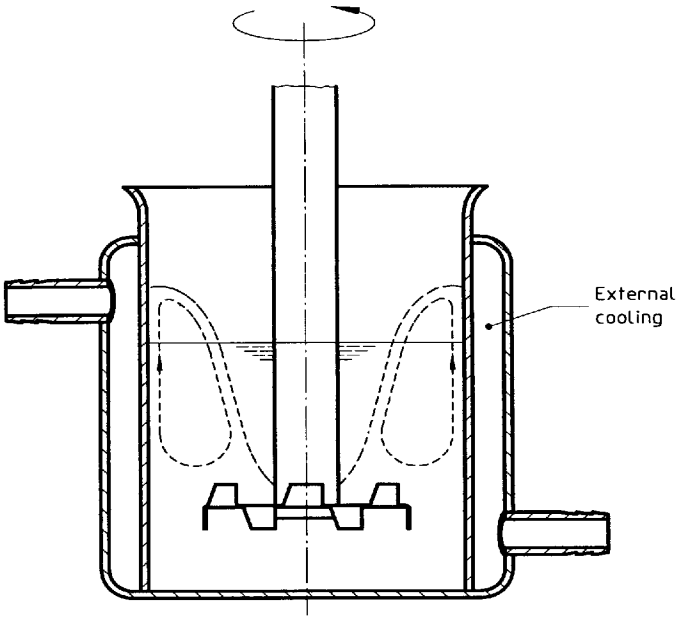

Figure 1 — Dissolver (double-walled)

The size of the mixing vessel may vary depending on the amount of paste required. An appropriate size should be selected from table 1.

The drive motor and the spindle shall be fixed to a stand in such a way that their height is adjustable. A suitable device to hold the mixing vessel in place shall be fitted at the base of the stand so that the spindle is situated centrally along the axis of the mixing vessel.

The speed of rotation (see table 1) shall remain constant over the whole of the mixing cycle.

Table 1 — Dimensions of dissolver and mixing vessel

Dissolver	**1**	**2**	**3**
Disc			
Diameter of disc, mm	40 ± 1	50 ± 1	80 ± 1
Number of teeth	12	12	12
Thickness of disc, mm	approx. 1,5	approx. 1,5	approx. 1,5
Diameter of spindle, mm	approx. 15	approx. 15	approx. 15
Speed of rotation of spindle, rpm	2 500 ± 100	2 000 ± 100	1 250 ± 50
Height of disc above bottom of beaker, mm	20 ± 1	25 ± 1	40 ± 1
Mixing vessel			
Internal diameter, mm	65 ± 1	80 ± 1	120 ± 1
Total height, mm	⩾ 85	⩾ 110	⩾ 180
Amount of paste, g	200 ± 10	400 ± 20	1 500 ± 75

The dissolver disc shall be made of stainless steel. The teeth shall be in the shape of trapeziums or parallelograms inclined at 30° to the vertical and attached around the circumference of the disc such that they alternate vertically upwards and downwards (see figure 2).

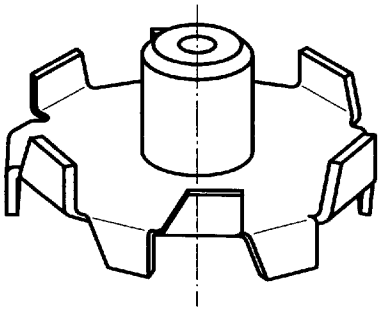

Figure 2 — Dissolver disc

The diameter and thickness of the disc, the number of teeth, the diameter of the spindle and the height above the beaker bottom are given in table 1.

The mixing vessel shall be made of stainless steel with an internal diameter and a total height as given in table 1.

To remove the frictional heat generated during mixing, an efficient external cooling system, designed to operate with, for instance, a water-cooled double-walled mixing vessel, shall be used.

For safety reasons, the upper part of the spindle which is not surrounded by the mixing vessel shall be protected by a jacket.

The electric drive to the spindle shall be connected to a trip switch which is set in such a way that the drive unit can only operate when the dissolver disc is in the vessel.

4.2 Balance, having an accuracy of 0,5 g.

4.3 Spatula, made of flexible plastic.

4.4 Stopwatch.

4.5 Water bath, maintained at 23 °C ± 1 °C.

4.6 Plasticizer.

5 Sampling and conditioning

A representative sample of the PVC resin, free of agglomerates, shall be conditioned in atmosphere 23/50 in accordance with ISO 291 until moisture equilibrium has been attained. The resin sample shall then be stored in this condition until used.

6 Composition of paste

The amount of paste prepared in one mixing cycle shall be within the limits prescribed in table 1. For designation purposes, standard paste A or B as described in ISO 1060-2 can be used. The exact formulation shall be reported in the paste-preparation report.

7 Paste preparation

Weigh out, to an accuracy of 1 g, the quantity of PVC resin sample and plasticizer prescribed for the paste formulation used (see clause 6). Condition the plasticizer and the PVC resin sample at a temperature of 23 °C ± 1 °C. Transfer the plasticizer to the mixing vessel and stir in the PVC resin manually until the powder has been fully wetted. Scrape any unmixed ingredients from the mixing-vessel walls with a spatula (4.3) and incorporate them into the mixture. The filled mixing vessel shall now be held at a temperature of 23 °C by

a) circulating water from a water bath (4.5) between the walls of the mixing vessel in the case of a double-walled mixing vessel (preferred method)

or

b) placing the mixing vessel in the water bath in the case of a single-walled vessel.

Position the mixing vessel centrally under the dissolver disc and lower the disc to the height prescribed in table 1.

Stir the mixture at the speed prescribed in table 1 for 150 s ± 5 s. Then stop the mixer. Using the spatula, clean the spindle and mixing-vessel walls of any adhering paste or unmixed ingredients, and incorporate into the mixture. Finally, stir the mixture for a further 150 s ± 5 s at the same speed as previously. Check the temperature of the paste.

The temperature of the paste shall not exceed 35 °C during the whole procedure. If the PVC resin does not form a paste under these conditions or if the paste temperature exceeds 35 °C, prepare a paste with a higher plasticizer concentration.

If the paste is to be used for viscosity measurements, de-aerate the paste under a partial vacuum of e.g. 700 Pa and maintain it at this pressure for 5 min after the foam has collapsed. Afterwards, store it at a temperature of 23 °C ± 1 °C, e.g. in the water bath, until it is used.

8 Paste-preparation report

The paste-preparation report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the PVC resin used;

c) the paste formulation used;

d) the temperature of the paste at the end of the mixing stage;

e) the date of preparation of the paste;

f) any deviations from this International Standard.

ICS 83.080.20

Descriptors: plastics, polyvinyl chloride, test specimens, pastes, specimen preparation.

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