
Polimerni materiali - Homo- in kopolimeri vinilklorida za splošno rabo - Določanje absorpcije mehčal pri sobni temperaturi

Plastics -- Homopolymer and copolymer resins of vinyl chloride for general use -- Determination of plasticizer absorption at room temperature

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Plastiques -- Résines d'homopolymères et de copolymères de chlorure de vinyle à usages généraux -- Détermination de la prise de plastifiant à température ambiante

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International Standard



4608

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

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Descriptors : plastics, polyvinyl chloride, tests, determination, plasticizer absorption.

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4608 was prepared by Technical Committee ISO/TC 61, *Plastics*.

ISO 4608 was first published in 1977. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

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Plastics — Homopolymer and copolymer resins of vinyl chloride for general use — Determination of plasticizer absorption at room temperature

1 Scope and field of application

This International Standard specifies a method for determining plasticizer absorption **at room temperature**. It is applicable to PVC general-purpose resins (designated "G" in ISO 1060/1, *Plastics — Homopolymer and copolymer resins of vinyl chloride — Part 1: Designation*).

The object of the test is to determine the quantity of plasticizer absorbed by a resin at room temperature to give a dry mixture.

The results give a **general indication** of the plasticizer absorption of the resins at room temperature. They indicate the usefulness of resins for the manufacture of plasticized dry blends, particularly when taken in conjunction with the results of plasticizer absorption tests under hot conditions.

2 Principle

Addition of an excess of di-2-ethylhexyl phthalate (DOP) to a specific amount of resin. Centrifuging of the mixture under defined conditions and calculation of the amount of plasticizer retained by the resin.

3 Reagent

Di-2-ethylhexyl phthalate (DOP).

4 Apparatus

Ordinary laboratory apparatus, and

- 4.1 Balance**, accurate to 0,1 mg.
- 4.2 Burette**, for example 50 ml, graduated in 0,1 ml.
- 4.3 Centrifuge**, whose rotor turns in a horizontal plane and which has an acceleration under the test conditions of 24 500 to 29 500 $\text{m}\cdot\text{s}^{-2}$ measured at the level of the bottom of the tube, with, if necessary, a cooling system to prevent the temperature of the mixture from exceeding 30 °C at the end of centrifuging for 60 min.

NOTE — It is permissible to use higher acceleration to reduce the centrifuging time, for example 34 500 $\text{m}\cdot\text{s}^{-2}$ and 30 min, provided that it has been verified that the results obtained are equivalent.

4.4 Centrifuge tubes, to fit the centrifuge used, consisting of a tube, usually of glass, with a conical bottom pierced by a hole of about 0,8 mm diameter (see the figure).

4.5 Plastic sheaths, (polyamide, polyethylene, etc.) with a piece of polyvinyl chloride pipe at the bottom to support the centrifuge tube (see the figure).

Dimensions in millimetres

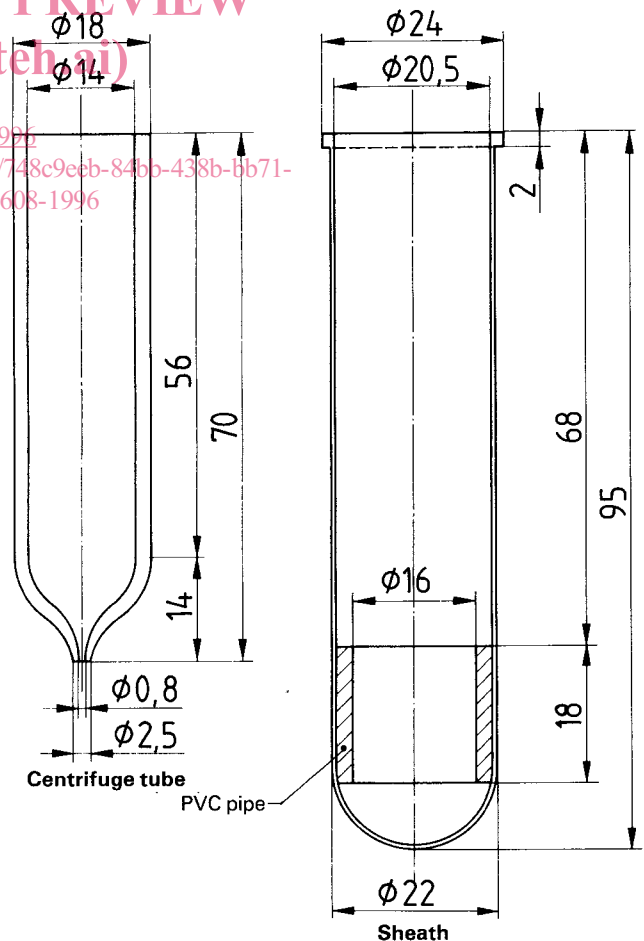


Figure — Example of centrifuge tube and sheath

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4.6 Cotton wool, pharmaceutical quality, having a DOP absorption measured under the test conditions (see 5.1) of approximately 10 %.

NOTE — Alternative materials to cotton wool may be used if it can be shown that they produce equivalent results; for example, glass wool and PTFE-coated polyester felt.

5 Procedure

5.1 Measurement of DOP absorbed by the cotton wool

Under the conditions indicated in 5.2, carry out a test with a piece of cotton wool having a mass of 100 ± 2 mg, **but without resin**.

Determine the amount of DOP absorbed by the cotton wool, in grams (mass m_0).

5.2 Determination

Weigh a piece of cotton wool (100 ± 2 mg), place it in the centrifuge tube and pack it down slightly. Weigh the tube and cotton wool to the nearest $\pm 0,1$ mg, m_1 being the mass, in grams, of the prepared tube.

Weigh directly into the tube, to the nearest 1 mg, 2 g of the resin under test, m_2 being the mass, in grams, of the prepared tube plus resin, to the nearest 0,1 mg.

From the burette, run into the tube 4 ml of DOP (clause 3) and allow it to stand for about 10 min.

Then put the tube into its sheath and place the whole into one of the compartments of the centrifuge rotor (the other compartments being occupied by tubes containing other resins, all the tubes being balanced).

Set the centrifuge to give an acceleration of 24 500 to 29 500 $\text{m}\cdot\text{s}^{-2}$ at the level of the bottom of the tube for 60 min. If necessary, the cooling device shall be switched on during centrifuging. Check that the temperature does not exceed 30 °C.

Take the tube from its sheath, carefully wipe it to remove any DOP on the outside and weigh it to the nearest 0,1 mg, m_3 being the mass, in grams, of the tube containing the resin and absorbed DOP.

6 Expression of results

6.1 Calculation

The room temperature plasticizer absorption, expressed as parts of DOP absorbed per 100 parts of resin (p.h.r.), is given by the formula

$$\frac{(m_3 - m_0) - m_2}{m_2 - m_1} \times 100$$

where

m_0 is the mass, in grams, of DOP absorbed by cotton wool (5.1);

m_1 is the mass, in grams, of the centrifuge tube with cotton wool (5.2);

m_2 is the mass, in grams, of the centrifuge tube with cotton wool and resin sample (5.2);

m_3 is the mass, in grams, of the centrifuge tube with resin and DOP absorbed after centrifuging (5.2).

6.2 Precision

Interlaboratory trials conducted on six resins in nine laboratories have shown the coefficients of variation to be as follows:

— **within laboratory:** $\pm 0,5$ to $\pm 3,5$ % (with some instances of ± 4 , ± 5 and even ± 7 %);

— **between laboratory:** about twice those of within laboratory results.

7 Test report

The test report shall include the following information:

- reference to this International Standard;
- full details necessary for the complete identification of the sample;
- centrifuging conditions (acceleration and time) if these differ from those defined in the method, and the temperature after centrifuging, if greater than 30 °C;
- the room temperature plasticizer absorption.