

SLOVENSKI STANDARD

SIST ISO 4574:1996

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Polimerni materiali - PVC za splošno uporabo - Določanje absorpcije mehčal v vročem

Plastics -- PVC resins for general use -- Determination of hot plasticizer absorption

Plastiques -- Résines de polychlorure de vinyle à usages généraux -- Détermination de la prise de plastifiant à chaud

Ta slovenski standard je istoveten z: ISO 4574:1978

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INTERNATIONAL STANDARD



4574

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Plastics — PVC resins for general use — Determination of hot plasticizer absorption

Plastiques — Résines de polychlorure de vinyle à usages généraux — Détermination de la prise de plastifiant à chaud

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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 4574 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in November 1976.

It has been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	Portugal
Belgium	Israel	Romania
Brazil	Italy	Spain
Bulgaria	Korea, Rep. of	Switzerland
Canada	Mexico	Turkey
Finland	Netherlands	United Kingdom
France	New Zealand	Yugoslavia
Germany	Peru	
Hungary	Philippines	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Czechoslovakia
U.S.A.

Plastics — PVC resins for general use — Determination of hot plasticizer absorption

0 INTRODUCTION

A need exists to evaluate the amount of plasticizer absorbed by a PVC polymer which is to be used in dry blending operations. This International Standard specifies a standardized technique under set conditions and is intended to supplement the results obtained using ISO 4608.

Since these tests do not correspond in detail to particular industrial processes used for the manufacture of dry blends from PVC polymer and plasticizer, the test results are empirical and need interpretation in the light of experience.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the hot plasticizer absorption of PVC polymers intended for general use (designated "G" in ISO 1060/1, *Plastics — Homopolymer and copolymer resins of vinyl chloride — Part 1 : Designation*) by hot mixing in a planetary mixer and measuring the amount of plasticizer absorbed.

2 REFERENCE

ISO 4608, *Plastics — PVC resins for general use — Determination of hot plasticizer absorption*.

3 PRINCIPLE

Conditioning of 200 parts of plasticizer in the bowl of a planetary mixer at a temperature of $75 \pm 0,2$ °C. Addition of 100 parts of the resin to be tested and mixing with the plasticizer. Taking of samples of this mixture at various times (systematically from 1 to 30 min), removal of excess plasticizer by centrifuging, and determining the quantity of plasticizer absorbed by the polymer. Plotting of a graph of quantity of plasticizer absorbed versus time, from which can be determined, for the polymer under test,

- the mean rate of plasticizer absorption (RPA);
- the hot plasticizer absorption (HPA) at 75 °C and 30 min (see the annex).

4 REAGENT

Di-(2-ethylhexyl) phthalate (DOP).

5 APPARATUS

5.1 Planetary mixer, having the shape and general dimensions shown in figures 1 and 2, and comprising the following items :

5.1.1 Jacketed stainless steel bowl.

5.1.2 Thermostat and pump, for circulating demineralized water in the jacket (see note 1) to regulate the temperature in the bowl at $75 \pm 0,2$ °C.

5.1.3 Beater

5.1.4 Motor, sufficiently strong to produce the required frequency of rotation and to maintain it throughout the mixing procedure.

5.1.5 Rotating wiper or scraper, for cleaning the inside of the bowl.

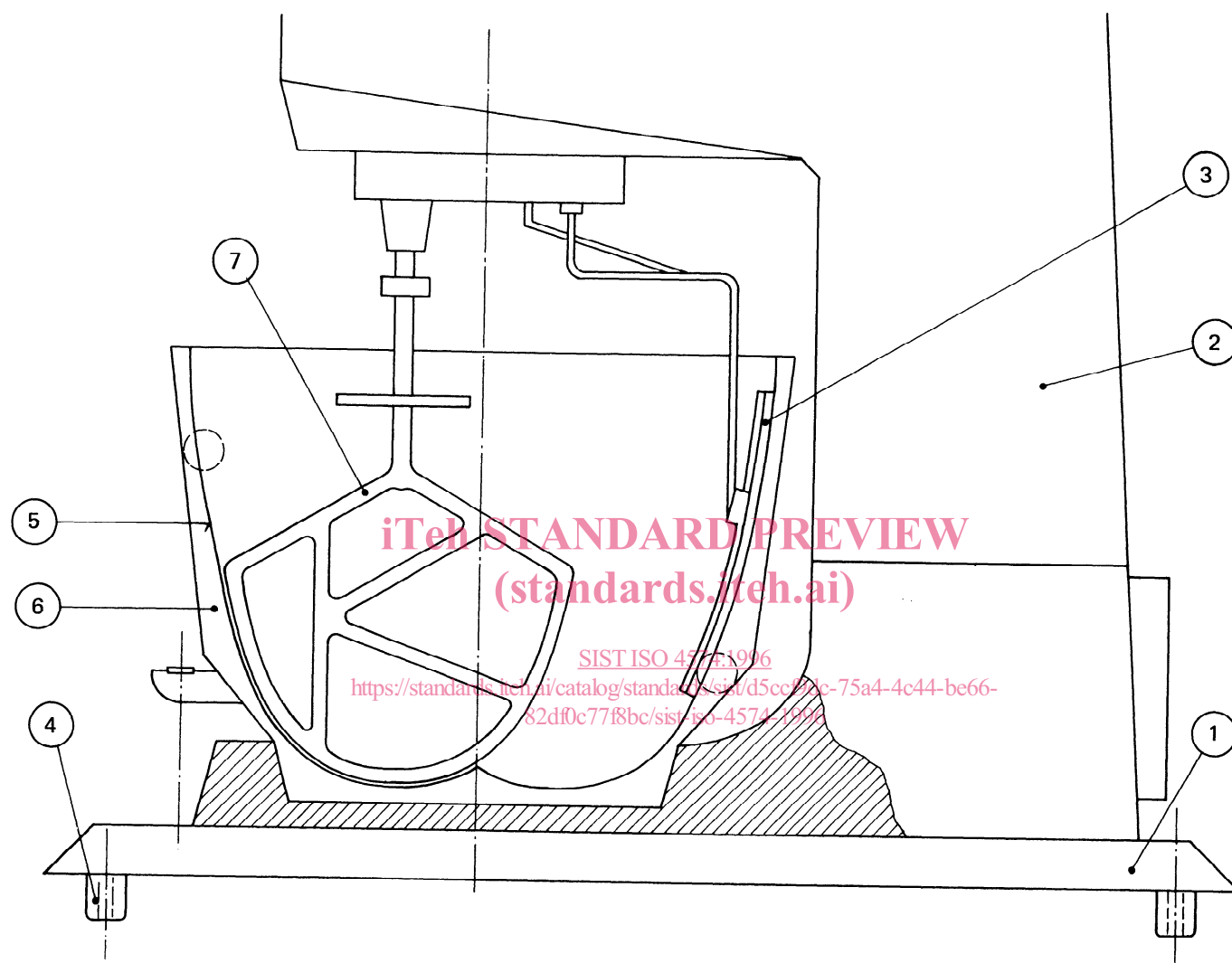
NOTES

1 If the test is carried out at a temperature other than that specified and in particular at a temperature exceeding 85 °C, it is necessary to use oil in the jacket instead of demineralized water.

2 It may be of interest to record the resistance to torque during the preparation of the mixture. A suitable mixer for this purpose is obtainable commercially. Details may be obtained from the Secretariat of ISO/TC 61 or from the ISO Central Secretariat.

5.2 Centrifuge, the rotor of which turns in a horizontal plane, having an acceleration under the conditions of test of $2,5 \times 10^4 \text{ m}\cdot\text{s}^{-2}$ to $3,0 \times 10^4 \text{ m}\cdot\text{s}^{-2}$ measured at the level of the bottom of the tube, and equipped, if necessary, with a cooling system to prevent the temperature of the mixture at the end of centrifuging from exceeding 30 °C.

NOTE — It is permissible to use a higher acceleration to reduce the centrifuging time, for example $3,5 \times 10^4 \text{ m}\cdot\text{s}^{-2}$ and 30 min, provided that it has been proved that the results obtained are equivalent.



- 1 – Base
- 2 – Planetary type mixer
- 3 – Wiper or scraper
(rotating to clean inside of bowl)
- 4 – Feet
- 5 – Stainless steel bowl
- 6 – Jacket (for temperature control)
- 7 – Special beater

FIGURE 1 – General sketch of the modified planetary mixer

Dimensions in millimetres

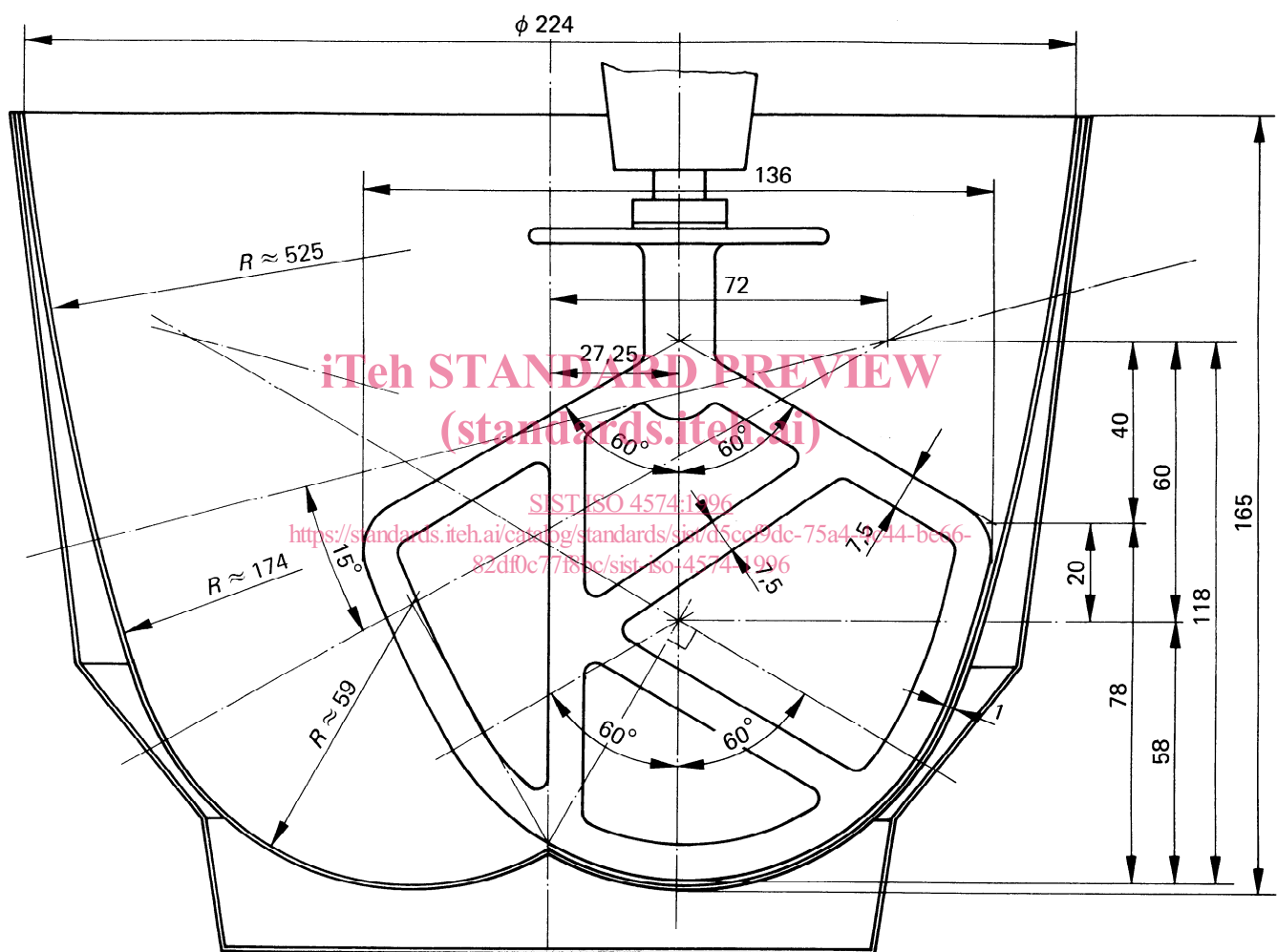


FIGURE 2 — Principal dimensions of the bowl and the beater

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5.3 Centrifuge tubes, of suitable dimensions to fit the centrifuge used, consisting of a tube usually made of glass with a conical bottom, pierced by a hole of diameter about 0,8 mm.

5.4 Plastic sheaths (polyamide, polyethylene, etc.), having a piece of tubing (for example of polyvinyl chloride) at the bottom to support the centrifuge tube.

NOTE — Examples of tube and sheath are given in figure 3.

5.5 Cotton wool of pharmaceutical quality, having a DOP absorption, measured under the test conditions of ISO 4608, of approximately 10 %.

5.6 Balances, accurate to 0,1 g (for weighing the materials) and to 0,01 g (for weighing the centrifuge tubes).

5.7 Two vessels, of capacity about 1 litre, one for

weighing the plasticizer and the other for weighing and conditioning the polymer under test.

5.8 Thermometer, graduated in 0,1 °C.

5.9 Apparatus, for measuring to the nearest 0,1 °C the temperature of plasticizer in the bowl and also that of the mixture; for example a thermocouple and millivoltmeter.

5.10 Aluminium foil, about 0,05 mm thick, for making a light deformable scoop for removing some of the mixture without stopping the beater and without damaging the beater blade.

5.11 Timer.

If necessary :

5.12 Solid carbon dioxide, for quick freezing of the samples taken.

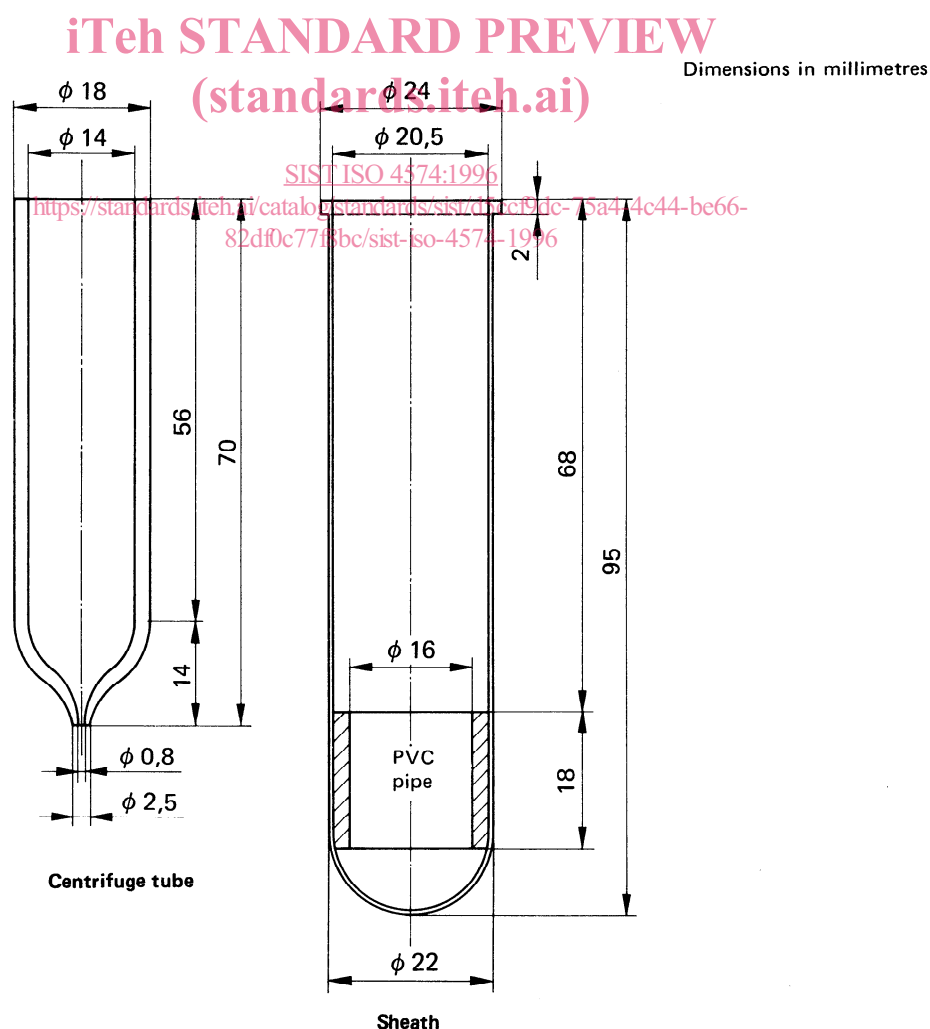


FIGURE 3 — Example of centrifuge tube and sheath

6 PROCEDURE

6.1 Preliminary regulation of thermostat

Weigh, to the nearest 0,1 g, 600 g of the DOP (clause 4) and place in the bowl (5.1.1) of the mixer.

Set the mixer to operate at a rotational frequency of 60 min^{-1} .*

Regulate the thermostat (5.12) so that the temperature of the DOP is stabilized at $75 \pm 0,2^\circ\text{C}$. Verify the temperature with the thermometer (5.8).

Pour the DOP out of the bowl, clean the bowl and the beater (5.1.3) and dry them.

6.2 Measurement of mass of DOP absorbed by the cotton wool

Under the conditions indicated in sub-clause 5.2 of ISO 4608, carry out a test with a piece of cotton wool having a mass of $0,100 \pm 0,002 \text{ g}$, but without resin.

Determine the mass, in grams, of DOP absorbed by the cotton wool.

6.3 Determination

Into each of twelve centrifuge tubes (5.3), insert, using moderate pressure, a piece of cotton wool (5.5) of mass $0,100 \pm 0,002 \text{ g}$. Weigh each tube with the piece of cotton wool to the nearest 0,01 g.

Weigh, to the nearest 0,1 g, 600 g of the DOP and place in the bowl of the mixer. Operate the mixer at a rotational frequency of 60 min^{-1} for at least 15 min. Stop the mixer and verify that the temperature of the DOP is $75 \pm 0,2^\circ\text{C}$.

While the DOP is being conditioned, weigh, into one of the vessels (5.7), to the nearest 0,1 g, 300 g of the polymer to be tested. When the temperature of the DOP has reached $75 \pm 0,2^\circ\text{C}$, carry out the three following operations simultaneously :

- place the polymer in the mixer;
- restart the mixer at a rotational frequency of 60 min^{-1} ;
- start the timer (5.11).

NOTE — When using the mixer referred to in note 2 of 5.1, simultaneously start measuring the torque.

After mixing for 1 min and without stopping the mixer, remove a sample of about 5 g of the mixture by means of an aluminium scoop (5.10) and place it in one of the

centrifuge tubes already prepared. Place the tube in its sheath (5.4) and allow it to cool; if necessary, place it in solid carbon dioxide (5.12) to cool it quickly.

Take other samples in the same way, starting when the mixture passes from its pasty state to the moist premix state, and at intervals of time based on visual changes in the mixture, but in every case at a mixing time of 30 min. Then stop the mixer.

NOTE — The use of the mixer referred to in note 2 of 5.1 allows the samples to be taken at appropriate times as observed by torque measurement. When the torque begins to increase, the samples should be taken with a frequency proportional to the rate of increase of torque until the torque is stabilized.

Weigh the tubes containing the samples to the nearest 0,01 g.

Replace the tubes in their sheaths and place them in the centrifuge holders. Operate the centrifuge (5.2) at an acceleration of $2,5 \times 10^4 \text{ m}\cdot\text{s}^{-2}$ to $3,0 \times 10^4 \text{ m}\cdot\text{s}^{-2}$ for 60 min. Other conditions may be used if they have been shown to produce equivalent results. The centrifuge may be cooled. The centrifuging must be completed within 60 to 90 min after the removal of the samples from the mixture.

Remove the tubes from their sheaths, wipe them carefully to remove any DOP which may be on the outside of the tubes, and weigh them to the nearest 0,01 g.

6. CALCULATION AND EXPRESSION OF RESULTS

For each tube, calculate the amount of absorbed DOP, in parts per hundred of resin (p.h.r.) by the formula

$$100 \left[2 - 3 \frac{m_2 - (m_3 - m_0)}{m_2 - m_1} \right]$$

where

m_0 is the mass, in grams, of DOP absorbed by the cotton wool (see 6.2);

m_1 is the mass, in grams, of the centrifuge tube and cotton wool;

m_2 is the mass, in grams, of the centrifuge tube, cotton wool and sample before centrifuging;

m_3 is the mass, in grams, of the centrifuge tube, cotton wool (and DOP absorbed by the cotton wool), the resin and DOP absorbed by the resin, after centrifuging.

NOTE — The amount of absorbed plasticizer calculated by this formula is less than the actual quantity because a part of the polymer is dissolved in the unabsorbed DOP and eliminated by centrifuging.

* This frequency is that of the rotation of the beater about the axis of the bowl (not the frequency of rotation of the beater about itself, which is about 140 min^{-1}).