INTERNATIONAL STANDARD

ISO 4574

Second edition
2019-05

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

Reference number
ISO 4574:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 4574:1978), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— Clause 2 has been updated;

— Clause 3 has been added and the subsequent clauses have been renumbered;

— the document has been editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 document 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.

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

1 Scope

This document specifies a method for determining the hot plasticizer absorption of PVC polymers intended for general use (designated "G" in ISO 1060-1) by hot mixing in a planetary mixer and measuring the amount of plasticizer absorbed.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4608:1998, *Plastics — PVC resins for general use — Determination of hot plasticizer absorption*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Conditioning of 200 parts of plasticizer in the bowl of a planetary mixer at a temperature of 75 ± 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 min 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 Annex A).

5 Reagent

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

6 Apparatus

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

6.1.1 Jacketed stainless-steel bowl.

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

6.1.3 Beater.

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

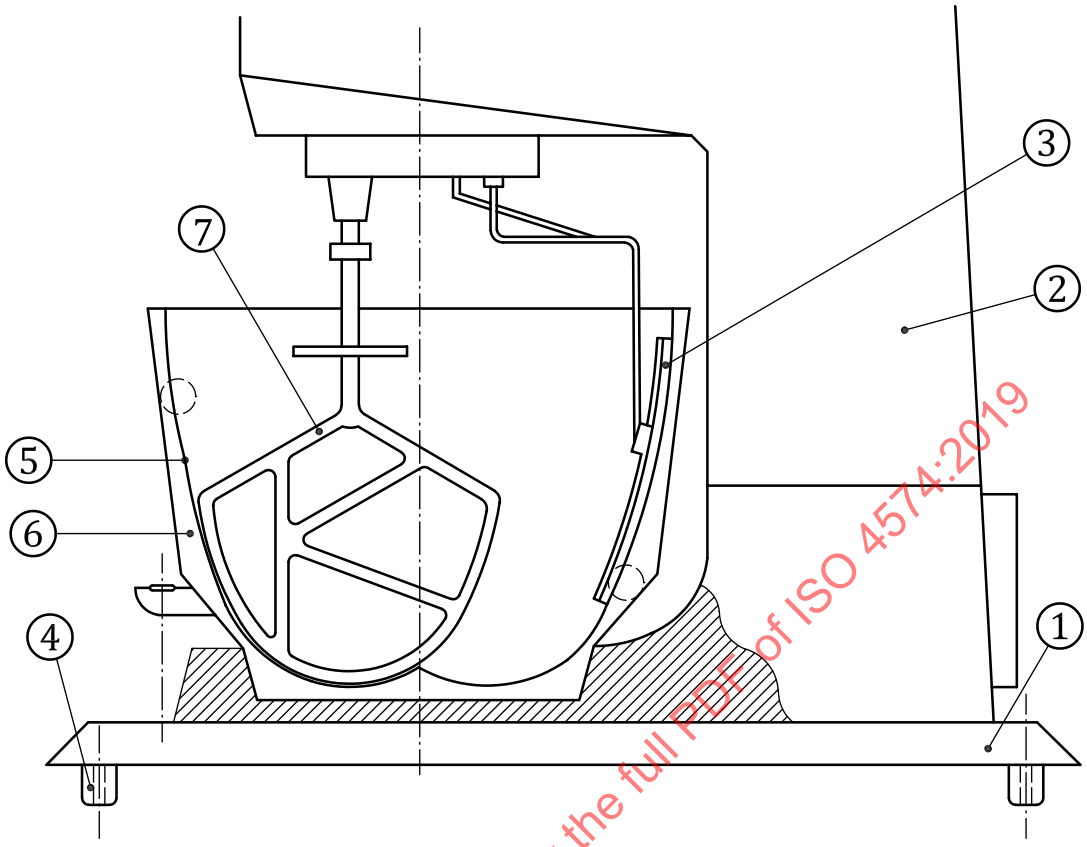
6.1.5 Rotating wiper or scraper, for cleaning the inside of the bowl.

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.

NOTE It might 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 can be obtained from the Secretariat of ISO/TC 61 or from the ISO Central Secretariat.

6.2 Centrifuge, the rotor of which turns in a horizontal plane, having an acceleration under the conditions of test of 2,5 × 10^4 ms^{-2} to 3,0 × 10^4 ms^{-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.

It is permissible to use a higher acceleration to reduce the centrifuging time, for example 3,5 × 10^4 ms^{-2} and 30 min, provided that it has been proved that the results obtained are equivalent.



Key

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

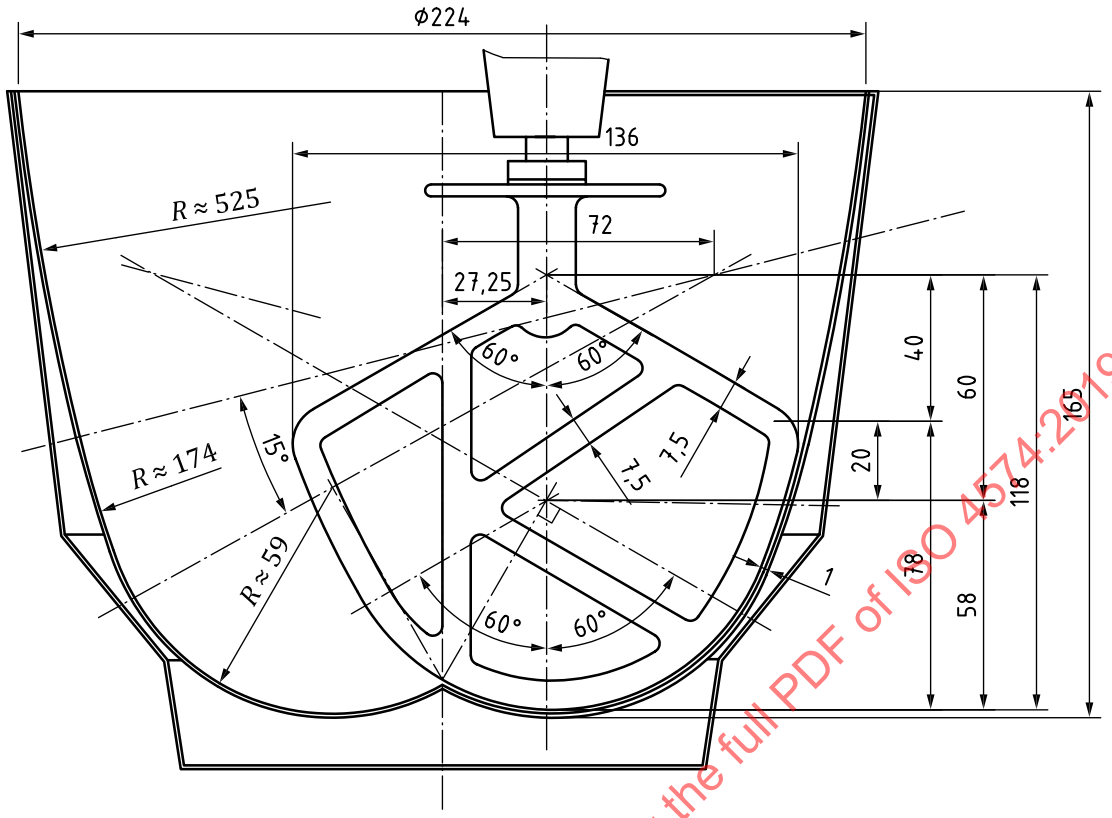


Figure 2 — Principal dimensions of the bowl and the beater

6.3 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 with a diameter of approximately 0,8 mm.

6.4 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.

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

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

6.7 Two vessels, of capacity about 1 l, one for weighing the plasticizer and the other for weighing and conditioning the polymer under test.

6.8 Thermometer, graduated in 0,1 °C.

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

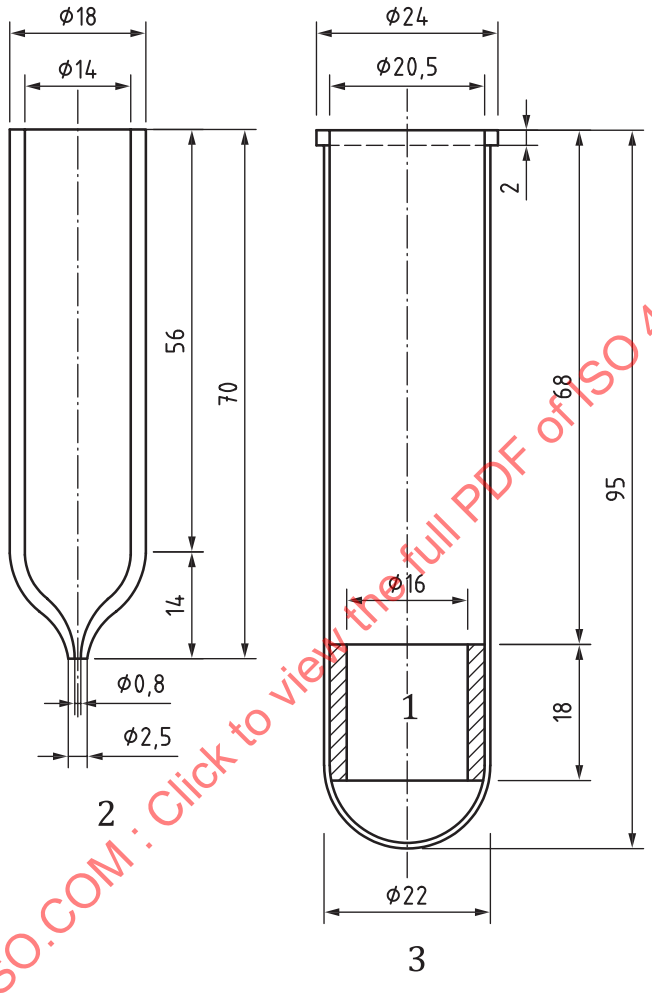
6.10 Aluminium foil, approximately 0,05 mm thick, for making a light deformable scoop for removing some of the mixture without stopping the beater and without damaging the beater blade.

6.11 Timer.

If necessary:

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

Dimensions in millimetres



Key

1 PVC pipe
2 centrifuge tube
3 sheath

Figure 3 — Example of centrifuge tube and sheath

7 Procedure

7.1 Preliminary regulation of thermostat

Weigh, to the nearest 0,1 g, 600 g of the DOP (see Clause 5) and place in the bowl (6.1.1) of the mixer.

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

1) 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}).

Regulate the thermostat (6.1.2) so that the temperature of the DOP Is stabilized at 75 ± 0,2 °C. Verify the temperature with the thermometer (6.8).

Pour the DOP out of the bowl, clean the bowl and the beater (6.1.3) and dry them.

7.2 Measurement of mass of DOP absorbed by the cotton wool

Carry out a test with a piece of cotton wool having a mass of 0,100 ± 0,002 g, but without resin, according to the conditions indicated in ISO 4608:1998, 5.1.

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

7.3 Determination

Into each of 12 centrifuge tubes (6.3), insert, using moderate pressure, a piece of cotton wool (6.5) of mass 0,100 ± 0,002 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 ± 0,2 °C.

While the DOP is being conditioned, weigh, into one of the vessels (6.7), to the nearest 0,1 g, 300 g of the polymer to be tested. When the temperature of the DOP has reached 75 ± 0,2 °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;2)]

— start the timer (6.11).

NOTE When using the mixer referred to in the note of 6.1.5, 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 (6.10) and place it in one of the centrifuge tubes already prepared. Place the tube in its sheath (6.4) and allow it to cool; if necessary, place it in solid carbon dioxide (6.12) 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.

The use of the mixer referred to in the note of 6.1.5 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 (6.2) at an acceleration of 2,5 × 10^4 ms^{-2} to 3,0 × 10^4 m 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.

2) 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}).

8 Calculation and expression of results

For each tube, calculate the amount of absorbed DOP, in parts per hundred of resin (p.h.r.) by using Formula (1):

$$100\left[2-3\frac{m_2-(m_3-m_0)}{m_2-m_1}\right] \quad (1)$$

where

m_0 is the mass, in grams, of DOP absorbed by the cotton wool (see 7.2);

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 1 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.

Plot a graph showing the quantity of absorbed DOP, in parts per hundred of resin, as a function of time. An example is shown in Figure 4.

Derive the rate of plasticizer absorption (RPA) as the slope of the line passing through the origin and tangent to the curve before the final levelling off of the curve. Read the hot plasticizer absorption as the asymptotic value of the amount of absorbed plasticizer per hundred parts of resin.

The hot plasticizer absorption (HPA) at 75 °C and 30 min is defined by the ordinate of the point corresponding to 30 min, in p.h.r.

NOTE 2 Interlaboratory tests carried out on three resins have shown a variation of 1,8 to 3,9 for the mean rate of absorption of the plasticizer (RPA), and of 8,8 to 14,1 for the hot plasticizer absorption (HPA), i.e. the amount of DOP absorbed in 30 min at 75 °C.

9 Test report

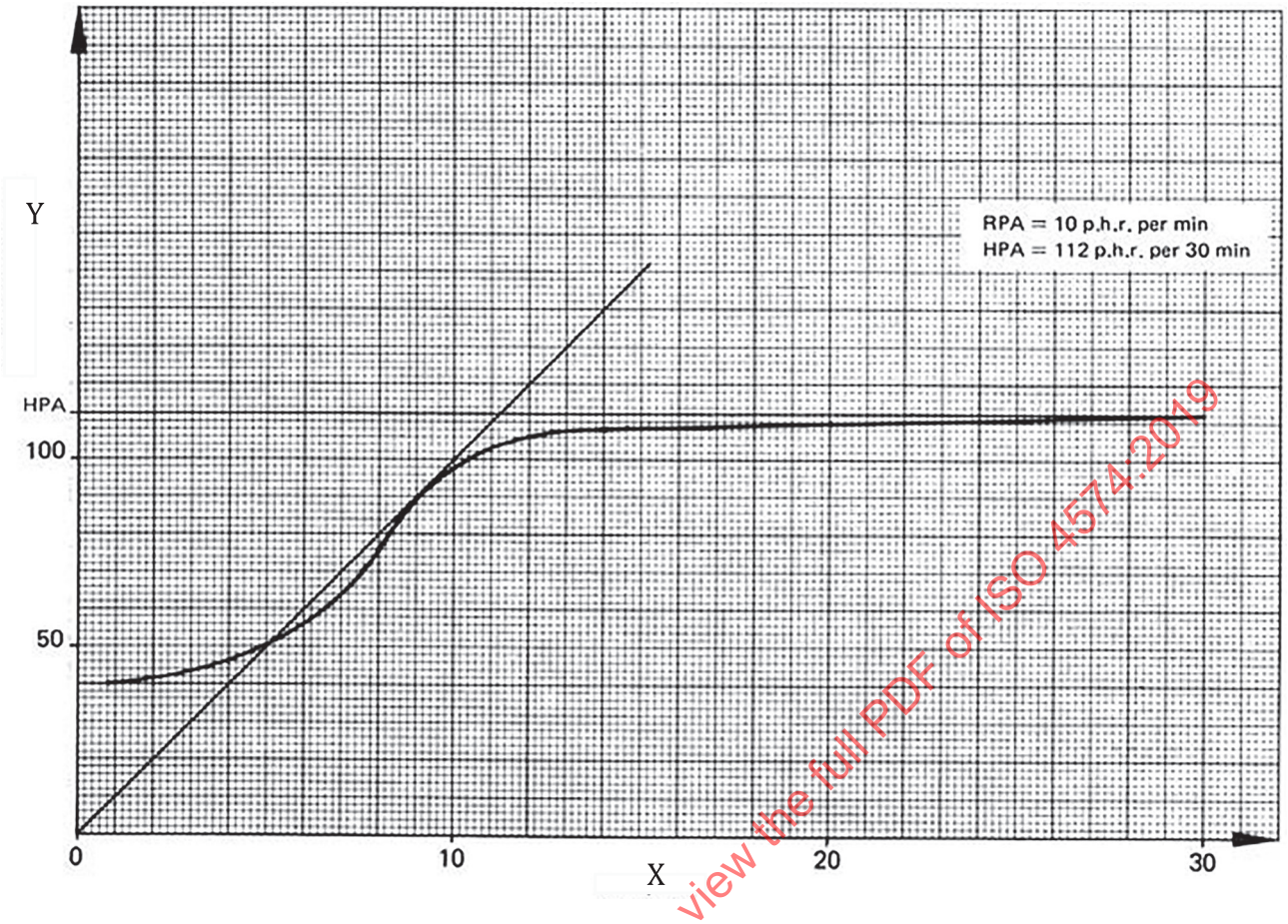
The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 4574:2019;

b) the complete identification of the material tested;

c) the hot plasticizer absorption (HPA), expressed in parts per hundred of resin (p.h.r.);

d) the mean rate of plasticizer absorption (RPA).



Key

X time, min

Y DOP absorbed, p.h.r.

Figure 4 — Example of graph of quantity of DOP absorbed versus time