INTERNATIONAL STANDARD

ISO 1598

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Plastics — Cellulose acetate — Determination of insoluble particles

Plastiques — Acétate de cellulose — Détermination des particules insolubles

Reference number
ISO 1598:1990(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 1598 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1598:1975), of which it constitutes a minor revision.

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INTERNATIONAL STANDARD ISO 1598:1990(E)

Plastics — Cellulose acetate — Determination of insoluble particles

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the number of visible particles (including all kinds of contamination and black dirt) in cellulose acetate which are insoluble in a mixture of dimethylphthalate, dichloromethane and methanol, and are of size 0,15 mm or larger.

This method is intended for cellulose acetate having an acetic acid yield above 50 % and containing no additives which affect the test results.

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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 585:—[1], *Plastics — Unplasticized cellulose acetate — Determination of moisture content.*

1) To be published. (Revision of ISO 585:1982)

3 Principle

A solution is prepared by addition of a solvent to the cellulose acetate, and the visible, undissolved particles in this solution, of a size equal to or larger than a standard reference particle of specified size, are counted.

4 Reagents

During the determination, use only reagents of recognized analytical grade, free from insoluble particles.

4.1 Dichloromethane, d_{20}^{20} 1,321 to 1,331, not less than 95 % (*V*/*V*) distilling between 39 °C and 40,5 °C at 1 013 mbar (760 mmHg).

SAFETY PRECAUTIONS — Dichloromethane is harmful by inhalation. Avoid contact with the skin.

4.2 Methanol, d_{20}^{20} 0,792 to 0,795, distillation range 64,5 °C to 65,5 °C at a pressure of 1 013 mbar (760 mmHg).

SAFETY PRECAUTIONS — Methanol is highly flammable and toxic by inhalation or if swallowed. Keep the container tightly closed and away from sources of ignition. Do not smoke. Avoid contact with the skin.

4.3 Dimethylphthalate, d_{20}^{20} 1,191 to 1,195, purity more than 99 % (*m*/*m*), having a moisture content less than 0,1 % (*m*/*m*).

5 Apparatus

5.1 Transparent glass dish, with a flat bottom of area not less than 26 000 mm^2.

Fine lines shall be ruled on the underside to form a network of squares with 25 mm sides. The dish shall be provided with a transparent cover to exclude dust.

5.2 Illuminated viewing stand, comprising:

a) a sheet of white opal glass the size of the dish or larger, evenly illuminated from below;

b) means to support the dish about 80 mm above the opal glass.

5.3 Standard reference particle, consisting of an opaque circular spot with a sharply defined edge on a transparent background. The spot shall have a diameter of 0,15 mm $\pm$ 5 µm. It shall be mounted on a holder so that the spot can be held underneath the dish and close to any particle seen in the cellulose acetate solution.

6 Test sample

6.1 The sample of cellulose acetate shall be in the form of powder passing entirely through a sieve of 710 µm mesh size (as defined in ISO 565); it shall be ground if necessary, avoiding excessive heating of the sample.

6.2 Determine the moisture content of the sample in accordance with ISO 585.

7 Procedure

7.1 Prepare a mixture of 90 parts of dichloromethane (4.1) and 10 parts of methanol (4.2), by volume.

Add this mixture to dimethylphthalate (4.3) in the proportion of 3 parts mixture to 1 part phthalate, by volume. The resulting mixture shall be free from visible particles.

7.2 Weigh, to the nearest 0,5 g, the quantity of the test sample corresponding to 20 g of dry cellulose acetate, and spread it evenly in the dish (5.1).

7.3 Add to the dish 180 g $\pm$ 0,5 g of the solvent mixture.

7.4 Place the cover on the dish and allow to stand at room temperature until dissolution is complete.

7.5 Place the covered dish in the illuminated viewing stand (5.2) and examine the network, square by square, holding the reference particle (5.3) as close as possible to any particle seen; if the apparent projected area of the particle is equal to or greater than that of the reference, the particle shall be included in the count.

7.6 The count shall include visible particles of all types, irrespective of colour or degree of opacity.

7.7 Make two complete determinations, noting the temperature at which they were carried out.

8 Expression of results

8.1 The insoluble-particle content, expressed as the number of particles in 100 g of dry cellulose acetate, is calculated from the formula

$$100 \times \frac{A}{m}$$

where

A is the number of particles counted;

m is the mass, in grams, of dry cellulose acetate in the test portion, calculated from the mass of the test portion and its moisture content determined as specified in 6.2.

8.2 The result is the mean of the two determinations and is expressed to the nearest whole number.

9 Precision

The precision of this test method is not known because inter-laboratory data are not available. This method may not be suitable for use in specifications or in case of disputed results as long as these data are not available.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample tested, including type, manufacturer's code number, source, trade name, etc.;

c) treatment of the sample before the test, if any;

d) the temperature at which the test was carried out;

e) the insoluble-particle content (see 8.2);

f) the date of the test.

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