SLOVENSKI STANDARD
SIST ISO 1600:1996

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Polimerni materiali - Acetat celuloze - Določanje absorpcije svetlobe oblikovancev, pripravljenih z različno dolgim segrevanjem

Plastics -- Cellulose acetate -- Determination of light absorption on moulded specimens produced using different periods of heating

Plastiques -- Acétate de cellulose -- Détermination de l'absorption de lumière sur éprouvettes moulées produites en utilisant différentes périodes de chauffage

Ta slovenski standard je istoveten z: **ISO 1600:1990**

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83.080.20	Plastomeri	Thermoplastic materials

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INTERNATIONAL STANDARD

ISO 1600

Second edition
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Plastics — Cellulose acetate — Determination of light absorption on moulded specimens produced using different periods of heating

Plastiques — Acétate de cellulose — Détermination de l'absorption de lumière sur éprouvettes moulées produites en utilisant différentes périodes de chauffage

Reference number
ISO 1600:1990(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 1600 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1600:1975), of which it constitutes a minor technical revision.

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Plastics — Cellulose acetate — Determination of light absorption on moulded specimens produced using different periods of heating

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 light absorption of cellulose acetate, employing test specimens taken from two mouldings which have been produced using different periods of heating.

The aim is to provide quantitative measurements which are compatible with visual judgements of yellowness and lightness, and of changes in these properties after moulding. The determinations are carried out on cellulose acetate in plasticized form rather than in solution, since a more reliable guide is thereby obtained to the performance of cellulose acetate in plastics materials.

This method minimizes the effects of haze or imperfections in the specimens.

This method is intended for cellulose acetate having an acetic acid yield of 54 % ± 2,5 %. It may also be applicable to other transparent plastics which are not strongly coloured and which can be moulded under the specified conditions.

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 585:1990[1], *Plastics — Unplasticized cellulose acetate — Determination of moisture content.*

3 Principle

The absorption of visible light by cellulose acetate is normally greatest at the blue end of the visible spectrum, and decreases continuously across the spectrum to the red end. Therefore two measurements of absorption, one at the red end and one at the blue end, are sufficient to characterize the absorption of light by the material.

For the determination of the initial optical density, specimens given the smallest practicable amount of heating are used. The optical densities are measured for blue light and for red light using specified colour filters, and the optical density at 25 mm thickness is calculated as the "initial light absorption".

The "light absorption after further heating" is obtained by similar measurements on further specimens prepared using a longer period of heating during moulding.

1) To be published.

4 Reagents

During the determination, use only reagents of recognized analytical grade, free from visible colour.

4.1 Dimethyl phthalate, analytical grade, d_{20}^{20} 1,191 to 1,195, purity more than 99 % (m/m).

4.2 Triacetin.

4.3 Ethyl lactate.

4.4 Acetone.

SAFETY PRECAUTIONS — Acetone is highly flammable. Keep the container in a well ventilated place and away from sources of ignition. Do not smoke. Take precautionary measures against static discharges.

4.5 Propan-2-ol.

SAFETY PRECAUTIONS — Propan-2-ol is highly flammable. Keep the container tightly closed and away from sources of ignition. Do not smoke.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Hydraulic press, capable of exerting a pressure of at least 8 MN/m² on the moulding surface, with means of heating to 200 °C and with water cooling.

5.2 Mould (see figure 1 for a suitable type), with polished surfaces substantially flat and parallel, to produce mouldings of thickness within the range 1,5 mm to 5 mm. A particular thickness within this range may be specified.

5.3 Photoelectric absorptiometer, to measure optical density at wavelengths in the region of 440 nm at the blue end of the spectrum and 640 nm at the red end. The arrangement of test specimen and photoelectric cell shall be such that all light emerging from the photoelectric cell side of the specimen at angles of up to 45° from the direction of the incident beam is received by the measuring system. An absorptiometer using a metal-filament lamp with filters having light transmission at 440 nm and 640 nm respectively (for example, with Chance O.B. 10[2] blue filters and Ilford 608[2] red filters) is suitable.

5.4 Stainless-steel grinder, electrically operated.

5.5 Oven, capable of being thermostatically maintained at 60 °C ± 2 °C or 70 °C ± 2 °C.

6 Test sample

6.1 If proceeding in accordance with 7.1.1 (first method of incorporating the plasticizer), 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.

If proceeding in accordance with 7.1.2 (second method of incorporating the plasticizer), the cellulose acetate need not be ground.

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

7 Procedure

7.1 Either of the methods described below may be used for the incorporation of plasticizer.

7.1.1 First method

Weigh into a glass bottle, to within ± 0,5 g, the quantity of the sample corresponding to 100 g of dry cellulose acetate. Into another glass bottle, weigh 45 g of dimethyl phthalate (4.1), to within ± 0,5 g. Slowly add the dimethyl phthalate to the cellulose acetate with constant stirring and continue to stir for at least 5 min after all the dimethyl phthalate has been added. Proceed in accordance with 7.2 to 7.9.

7.1.2 Second method

Place 200 g, weighed to the nearest 1 g, of the cellulose acetate in a 2 litre vessel. The moisture content of the sample shall be less than 0,5 %; if not, there is a risk of bubbles forming in the moulding. Add about one-half of the following mixture:

— dimethyl phthalate (4.1):
75 ml ± 0,5 ml (90 g ± 0,6 g);

— propan-2-ol (4.5): 400 ml ± 0,5 ml.

Homogenize by mixing briskly with a glass stirrer. Pour in the rest of the solvent/plasticizer mixture, and, after having mixed again and stoppered the vessel, place it immediately on a roller mixer operating between 50 rpm and 70 rpm. After 2 h of mixing, tap the vessel with the palm of the hand in order to dislodge any powder which may have become

2) Chance O.B. 10 and Ilford 608 are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

stuck to the sides. Place on the roller mixer again and mix for a further 4 h.

Pour the product into a porcelain dish, cover with a sheet of filter paper and let it stand in the open, at room temperature, overnight. Then place in an oven, thermostatically maintained at 60 °C ± 2 °C, and leave for 3 h to eliminate part of the solvent. At the end of this time, place the product in the original clean vessel again. Homogenize it for one or two minutes, by rapid mixing with a mixing rod equipped with a blade turning at 10 000 rpm. (This operation is to destroy any agglomerates that may have been produced during the stoving.) Stopper the vessel, then let it stand again at room temperature for about 20 h. Proceed in accordance with 7.2 to 7.9.

7.2 Heat the mixed material for 2 h at 70 °C ± 2 °C to remove moisture and complete the absorption of plasticizer.

7.3 Place a suitable quantity of the heated mixture in the mould (5.2), pre-heated to a temperature of 200 °C ± 2 °C. Apply contact pressure for 2 min, then full pressure (at least 8 MN/m² at the moulding surface) for a further 10 min ± 0,5 min for a moulding 1,5 mm thick. This time shall be increased by 0,5 min for each 0,5 mm above 1,5 mm thickness.

7.4 Release the pressure and start cooling immediately, continuing the cooling until the moulding is rigid enough to be ejected without being deformed. The rate of cooling shall be such that the mould temperature 2 min after the start of cooling is at least 30 °C below the moulding temperature.

7.5 Prepare a further moulding of the same thickness in a similar manner, but with a heating time, at full pressure, one-third of that used in 7.3.

7.6 For the measurement of the optical density, prepare two test specimens from each moulding as described in 7.7.

7.7 The test specimen thickness shall not exceed 13 mm, and should preferably be such that the measured optical density is not less than 0,06. With material of good colour, the measured optical density at the red end of the spectrum may be less than 0,06 even with a thickness of 13 mm. The required thickness may be obtained by laminating together two or more pieces cut from a moulding, without using heat or impairing the outer polished surfaces. A suitable method is to coat the surfaces to be joined with a cement containing equal volumes of triacetin (4.2), ethyl lactate (4.3) and acetone (4.4), leaving for a few minutes till tacky, then applying a second layer of cement and bonding the pieces together under slight pressure between polished plates in a press, using a jig to hold the pieces in position without slipping.

7.8 Measure the optical densities of each test specimen for red light and for blue light, using the photoelectric absorptiometer and filters (5.3), preferably immediately after moulding and specimen preparation (if this is not possible, the mouldings or test specimens shall be kept in the dark until the measurements can be made).

7.9 Measure the average thickness of each test specimen in the region traversed by the light beam.

8 Expression of results

8.1 The initial light absorptions and the light absorptions after further heating, expressed as the optical density D at 25 mm thickness, are calculated using the equation

$$D = 25 \times \frac{D_m - 0{,}03}{d}$$

where

D_m is the measured optical density;

d is the thickness, in millimetres, of the test specimen.

8.2 For the two test specimens prepared in accordance with 7.5, the mean value of the two results of D for red light is taken as "D red" and the mean value of the two results of D for blue light is taken as "D blue". Report these as "initial light absorptions".

8.3 For the two test specimens prepared in accordance with 7.2, 7.3 and 7.4, calculate the values for "D red" and "D blue" and report these as "light absorptions after further heating".

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 product tested, including type, manufacturer's code number, source, trade name, etc.;

c) the initial light absorptions and the light absorptions after further heating;

d) the date of the test.