INTERNATIONAL STANDARD 1600

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Plastics — Cellulose acetate — Determination of light absorption before and after heating

Matières plastiques — Acétate de cellulose — Détermination de l'absorption de lumière avant et après chauffage

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 1600 and found it technically suitable for transformation. International Standard ISO 1600 therefore replaces ISO Recommendation R 1600-1970 to which it is technically identical.

ISO Recommendation R 1600 was approved by the Member Bodies of the following countries :

Austria	Iran	Romania
Belgium	Israel	South Africa, Rep. of
Brazil	Italy	Spain
Czechoslovakia	Japan	Sweden
Egypt, Arab Rep. of	Korea, Rep. of	Switzerland
Germany	Netherlands	Turkey
Hungary	Poland	United Kingdom
India	Portugal	U.S.A.

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France*

The Member Body of the following country disapproved the transformation of ISO/R 1600 into an International Standard :

Canada

* Subsequently, this Member Body approved the Recommendation.

Printed in Switzerland

Plastics — Cellulose acetate — Determination of light absorption before and after heating

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of light absorption of cellulose acetate before and after 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 its performance 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 REFERENCES

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO/R 585, *Plastics — Determination of moisture content of non-plasticized cellulose acetate.*

3 PRINCIPLE

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

For the determination of optical density before heating, 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 internal density of 25 mm thickness is calculated as "light absorption before heating".

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

4 REAGENTS

4.1 Dimethyl phthalate, analytical grade, having at 20 °C/20 °C a relative density within the range 1,191 to 1,195 and a purity more than 99 % (*m/m*).

4.2 Triacetin.

4.3 Ethyl lactate.

4.4 Acetone.

4.5 Propan-2-ol.

All reagents must be free from visible colour.

5 APPARATUS

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

5.2 Mould (see figure for a suitable type), with polished surfaces substantially flat and parallel, to produce mouldings of thickness within the range 1,5 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 in the blue and 640 nm in the red. The arrangement of test specimen and photocell should be such that all light emerging from the photocell side of the specimen at angles 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 blue filters and Ilford 608 red filters) is suitable.

5.4 Stainless steel grinder, electrically operated.

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

6 TEST SAMPLE

6.1 If proceeding according to 7.1.1 (first method), the sample of cellulose acetate shall be in the form of powder passing entirely through a sieve of 710 μm mesh size (in accordance with ISO 565); it should be ground if necessary, avoiding excessive heating of the sample.

If proceeding according to 7.1.2 (second method), the cellulose acetate need not be ground.

6.2 Determine the moisture content of the sample according to ISO/R 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 in a glass bottle, to within ± 0,5 g, the quantity of the sample corresponding to 100 g of dry cellulose acetate. In 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 according to 7.2 to 7.9.

7.1.2 *Second method*

Place 200 g, weighed with an accuracy of ± 1 g, of the cellulose acetate in a 2 l vessel. The moisture content of the product shall be less than 0,5 %; if not, there is a risk of forming bubbles in the moulding. Add about one half of the following mixture :

- dimethyl phthalate (4.1) : 75 ± 0,5 ml (90 ± 0,6 g);
- propan-2-ol (4.5) : 400 ± 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 and 60 rev/min. After 2 h of mixing, tap the vessel with the palm of the hand in order to dislodge any powder which may have become stuck to the sides. Replace on the roller mixer for 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 controlled at 60 ± 2 °C for 3 h to eliminate part of the solvent. At the end of this time replace the product in the original clean vessel. Homogenize it for 1 or 2 min, by rapid mixing with a mixing rod equipped with a blade turning at 10 000 rev/min. (This operation is to destroy any agglomerates that may have been produced in the stoving.) Stopper the vessel, then let it stand again at room temperature for about 20 h. Proceed according to 7.9.

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

7.3 Place a suitable quantity of the heated mixture in the mould, which is at a temperature of 200 ± 2 °C. Apply contact pressure for 2 min then full pressure (at least 8 MN/m^2 at the moulding area) for a further 10 ± 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 Apply cooling immediately, until the moulding is rigid enough for ejection without deformation. The rate of cooling shall be such that the mould temperature 2 min after the onset 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 heating times, at full pressure, one-third of those used in 7.3.

7.6 Prepare two test specimens for measurement of optical density from each moulding.

7.7 The test specimen thickness shall not exceed 13 mm, and should preferably be such that the measured density is not less than 0,06. With material of good colour, the measured density in the red 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 mouldings, without using heat or impairing the outer polished surfaces. A suitable method is by coating the surfaces to be joined with a cement comprising equal volumes of triacetin (4.2), ethyl lactate (4.3), and acetone (4.4), leaving for a few minutes till tacky, then giving a second application of cement and laminating the mouldings together under slight pressure between polished plates in a press, using a jig to hold the pieces in position without slipping.

7.8 Measure optical densities of each test specimen for red light and for blue light, using the photoelectric absorptiometer and filters (5.3), immediately after moulding and specimen preparation, or otherwise after keeping in the dark.

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 light absorptions before and after heating, expressed as optical density of 25 mm thickness, are calculated by the equation

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

where

D is the optical density of 25 mm thickness;

d is the observed density;

t is the thickness in millimetres.

8.2 For the two test specimens prepared according to 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 "light absorptions before heating".

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

9 TEST REPORT

The test report shall include the following particulars :

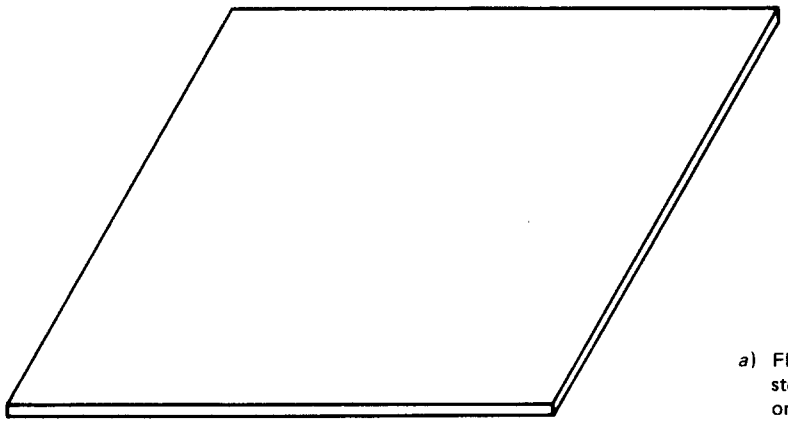
a) reference to this International Standard or to an equivalent national standard;

b) complete identification of the product tested, including type, manufacturer's code number, source, trade name, etc.;

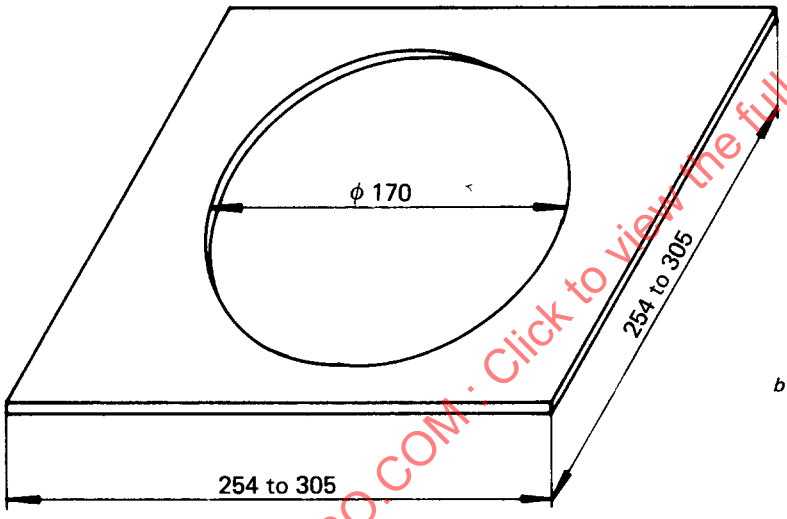
c) light absorptions before and after heating;

d) the date of test.

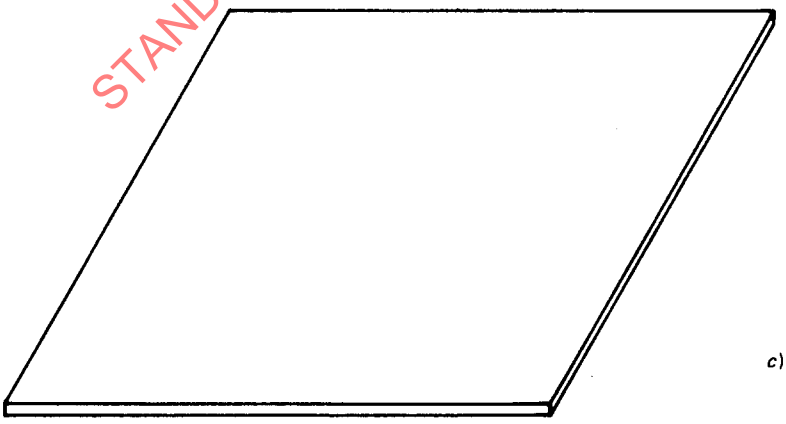
Dimensions in millimetres



a) Flat plate 1,5 mm thick, of stainless steel or nickel-plated steel polished on the lower surface



b) Stainless steel moulding template, 1,5 to 5 mm thick



c) Flat plate 1,5 mm thick, of stainless steel or nickel-plated steel polished on the upper surface

FIGURE — **Mould (see 5.2)**