

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1600

PLASTICS

DETERMINATION OF LIGHT ABSORPTION OF CELLULOSE ACETATE
BEFORE AND AFTER HEATING

1st EDITION

April 1970

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BRIEF HISTORY

The ISO Recommendation R 1600, *Plastics – Determination of light absorption of cellulose acetate before and after heating*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1600 which was circulated to all the ISO Member Bodies for enquiry in May 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

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PLASTICS

**DETERMINATION OF LIGHT ABSORPTION OF CELLULOSE ACETATE
BEFORE AND AFTER HEATING****1. SCOPE**

- 1.1 This ISO Recommendation describes 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 imperfection in the specimens.
- 1.2 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. PRINCIPLE OF METHOD

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 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.

3. REAGENTS

- 3.1 *Dimethylphthalate*, 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).
- 3.2 *Triacetin*.
- 3.3 *Ethyl lactate*.
- 3.4 *Acetone*.

All reagents must be free from visible colour.

4. APPARATUS

- 4.1 *Hydraulic press*, capable of a pressure of at least 8 MN/m^2 (80 kgf/cm^2) on the moulding surface area and of heating to 200°C and water cooling.
- 4.2 *Mould* (see Figure, page 8 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.
- 4.3 *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.
- 4.4 *Stainless steel grinder*, electrically operated.
- 4.5 *Oven*, thermostatically controlled at $70 \pm 2^\circ\text{C}$.

5. TEST SAMPLE

- 5.1 The sample of cellulose acetate must be in the form of powder passing entirely through a sieve of mesh $710 \mu\text{m}$; it should be ground if necessary, avoiding excessive heating during the operation.
- 5.2 The moisture content of the sample is determined according to ISO Recommendation R 585, *Plastics - Determination of the moisture content of non-plasticized cellulose acetate*.

6. PROCEDURE

- 6.1 Weigh in a glass bottle, with a precision of $\pm 0.5 \text{ g}$, the quantity of the test sample corresponding to 100 g of dry cellulose acetate. In another glass bottle weigh 45 g of dimethylphthalate (3.1) with a precision of $\pm 0.5 \text{ g}$. Slowly add the dimethylphthalate to the cellulose acetate with constant stirring and continue to stir for a least 5 minutes after all the dimethylphthalate has been added.
- 6.2 Heat the mixed material for 2 hours at $70 \pm 2^\circ\text{C}$ to remove moisture and complete the absorption of the plasticizer.
- 6.3 Place a suitable quantity of the heated mixture in the mould, which is at a temperature of $200 \pm 2^\circ\text{C}$. Apply contact pressure for 2 minutes then full pressure (at least 8 MN/m^2 at the moulding area) for a further 10 ± 0.5 minutes for a moulding 1.5 mm thick. This time should be increased by 0.5 minutes for each 0.5 mm above 1.5 mm thickness.
- 6.4 Apply cooling immediately, until the moulding is rigid enough for ejection without deformation. The rate of cooling should be such that the mould temperature 2 minutes after the onset of cooling is at least 30°C below the moulding temperature.
- 6.5 Prepare a further moulding in a similar manner and thickness, but with heating times, at full pressure, one-third of those used in clause 6.3.
- 6.6 Prepare two test specimens for measurement of optical density from each moulding.
- 6.7 The test specimen thickness should 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 (3.2), ethyl lactate (3.3), and acetone (3.4), 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.