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International Standard



3451 / 3

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**Plastics — Determination of ash —
Part 3: Unplasticized cellulose acetate**

Plastiques — Détermination du taux de cendres — Partie 3: Acétate de cellulose non plastifié

First edition — 1984-05-15

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UDC 678.544.4 : 543.822

Ref. No. ISO 3451/3-1984 (E)

Descriptors : plastics, unplasticized cellulose acetate, tests, determination, ashes.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (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 authorized 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.

International Standard ISO 3451/3 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in March 1980.

It has been approved by the member bodies of the following countries:

Belgium
Brazil
Canada
China
Czechoslovakia
Egypt, Arab Rep. of
Finland
France
Germany, F.R.
Hungary

India
Ireland
Israel
Italy
Japan
Korea, Rep. of
Mexico
Netherlands
Poland
Portugal

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Romania

South Africa, Rep. of

Spain

Sweden

Switzerland

United Kingdom

USA

USSR

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 872-1968, of which it constitutes a technical revision.

Plastics — Determination of ash — Part 3: Unplasticized cellulose acetate

1 Scope and field of application

1.1 This part of ISO 3451 specifies a method for determining the ash, consisting of inorganic residue, yielded by unplasticized cellulose acetate.

1.2 This method applies to unplasticized cellulose acetate free of additives, fillers, dyes or other materials which may affect the result.

1.3 When plasticizers, additives, fillers or dyes which may affect the result are present, they shall be separated by a method agreed between the interested parties.

1.4 The ash content at 575 ± 25 °C is a reasonable measure of the mineral salts and inorganic foreign matter in the cellulose acetate. The weight of ash obtained varies with the temperature of ignition. Higher temperatures such as 850 °C will convert calcium carbonate and other carbonates to the oxides and thus give lower values for the ash. The composition of ash may vary with the pulping process used in manufacture, which limits the significance of the ash determination in absolute terms.

2 References

ISO 585, *Plastics — Non-plasticized cellulose acetate — Determination of moisture content*.

ISO 3451/1, *Plastics — Determination of ash — Part 1: General methods*.

3 Principle

Ignition of a test portion and calcination in a muffle furnace maintained at 575 ± 25 °C or 850 ± 50 °C (as agreed between the interested parties), in accordance with ISO 3451/1, method A (direct calcination).

4 Apparatus

4.1 Crucible, of capacity 50 to 200 ml, made of silica, platinum or porcelain.

4.2 Bunsen burner, with silica triangle and tripod, or other suitable heating device.

4.3 Muffle furnace, thermostatically controlled at 575 ± 25 °C or 850 ± 50 °C.

4.4 Analytical balance, accurate to 0,1 mg.

4.5 Desiccator, containing a drying agent, for example anhydrous chloride.

4.6 Weighing bottle.

5 Procedure

5.1 Prepare the crucible (4.1) by heating in the muffle furnace (4.3) at 575 ± 25 °C or 850 ± 50 °C until constant mass is reached. Allow to cool in the desiccator (4.5) to room temperature for at least 1 h and weigh to the nearest 0,1 mg.

5.2 Introduce into the tared weighing bottle (4.6) a quantity of test sample, predried as prescribed in ISO 585 or with a known moisture content, corresponding to 5 to 50 mg of ash (usually 10 to 25 g) and weigh it to the nearest 0,1 mg.

NOTE — If the crucible will accommodate the quantity of test portion corresponding to the 5 to 50 mg of ash, this quantity may be weighed directly into the crucible. High bulk materials may be compressed into tablets which may be broken up into fragments of appropriate size.

5.3 Half fill the crucible with the test portion from the weighing bottle. Heat the crucible directly on the heating device (4.2) so that the sample burns slowly and loss of ash is avoided. Allow to cool and add another part of the test portion. Repeat this operation until the total quantity of the test portion is consumed.

5.4 Place the crucible in the muffle furnace, preheated to 575 ± 25 °C or 850 ± 50 °C, and calcine for 30 min.

5.5 Remove the crucible from the muffle furnace, place in the desiccator, allow to cool to room temperature for 1 h and weigh to the nearest 0,1 mg.

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5.6 Calcine again under the same conditions until constant mass is reached, i.e. until the results of two consecutive weighings do not differ by more than 0,5 mg.

6 Number of determinations

Carry out two determinations. Calculate the arithmetic mean of the results. If the single test results differ by more than 10 % of their mean, repeat the procedure until two successive results do not differ from each other by more than 10 % of their mean.

7 Expression of results

The ash expressed in grams per 100 g of dry cellulose acetate, is given by the formula

$$\frac{m}{m_0} \times 100$$

where

m is the mass of ash, in grams;

m_0 is the mass of dry acetate, in grams, calculated from the actual mass of the test portion and its moisture content determined in accordance with ISO 585.

8 Test report

The test report shall include the following particulars:

- a) reference to this International Standard;
- b) complete identification of the sample; including type manufacturer's code number, source, trade name, etc.;
- c) temperature used for calcination;
- d) treatment of the sample before the test, if any;
- e) single results of the two determinations and the average for the ash.

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