INTERNATIONAL STANDARD

ISO 1061

Second edition
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Plastics — Unplasticized cellulose acetate — Determination of free acidity

Plastiques — Acétate de cellulose non plastifié — Détermination de l'acidité libre

Reference number
ISO 1061:1990(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 1061 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1061:1975), of which it constitutes a minor technical revision.

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Plastics — Unplasticized cellulose acetate — Determination of free acidity

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 amount of free acid in unplasticized cellulose acetate.

The free acidity determined by this method includes acidity extractable by water and acidity due to acidic groups directly attached to the cellulose acetate, e.g. carboxyl groups. The latter is usually a very small proportion of the total.

This method is not suitable for cellulose acetate containing any additives which may affect the test.

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.*

1) To be published.

3 Principle

A test portion of cellulose acetate is treated with water and the resultant solution titrated with sodium hydroxide solution.

The free acidity is calculated as the percentage, by mass, of free acetic acid in the cellulose acetate.

4 Reagents

During the determination, use only reagents of recognized analytical grade and distilled water as specified in 4.1.

4.1 Distilled water, freshly boiled to remove carbon dioxide and cooled.

4.2 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,01 mol/l.

4.3 Phenolphthalein, 1 g/l solution in 90 % (V/V) ethanol.

5 Apparatus

5.1 Glass flask, capacity 250 ml or 300 ml, with ground-glass stopper.

5.2 Graduated cylinder, capacity 250 ml, graduated at 2 ml intervals.

5.3 Burette, capacity 25 ml, graduated at 0,05 ml intervals, protected against carbon dioxide by a soda lime tube.

5.4 Analytical balance, accurate to 0,01 g.

6 Test sample

6.1 The sample of cellulose acetate shall be in the form of a powder passing entirely through a sieve of mesh 710 µm as defined in ISO 565; if it does not, it shall be ground.

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

7 Procedure

7.1 Weigh, to the nearest 0,01 g, 10 g or more, depending on the expected free acidity, of the sample of cellulose acetate into the flask (5.1).

7.2 Add 150 ml of distilled water (4.1), measured with the 250 ml graduated cylinder (5.2).

7.3 Condition the stoppered flask at a temperature between 20 °C and 27 °C either for 3 h with gentle shaking for 5 min every 30 min, or for 1 h with continuous shaking.

7.4 Titrate with the sodium hydroxide solution (4.2), using phenolphthalein (4.3) as indicator.

It is important that the titration is carried out rapidly to avoid saponification of the cellulose acetate and absorption of carbon dioxide from the atmosphere.

7.5 Perform a blank test, introducing only 150 ml of distilled water into the flask. Allow to stand for 3 h or shake for 1 h in the same way as for the sample at a temperature between 20 °C and 27 °C. Titrate quickly with the sodium hydroxide solution (4.2), using phenolphthalein (4.3) as indicator.

7.6 Carry out two complete determinations. If the difference between the determinations is greater than 10 % of the mean, repeat the test.

8 Expression of results

8.1 The free acidity, expressed as grams of acetic acid per 100 g of dry cellulose acetate, is calculated from the formula

$$\frac{6c(V_1 - V_2)}{m}$$

where

c is the actual concentration, in moles of NaOH per litre, of the sodium hydroxide solution (4.2) used;

V_1 is the volume, in millilitres, of sodium hydroxide solution (4.2) required to titrate the solution obtained from the test portion;

V_2 is the volume, in millilitres, of sodium hydroxide solution (4.2) required to titrate the blank;

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

6 is the mass, in milligrams, of acetic acid corresponding to 1,00 ml of sodium hydroxide solution, c(NaOH) = 0,100 mol/l.

8.2 Report the mean of the two determinations.

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

d) the free acidity;

e) the date of the test.

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