SLOVENSKI STANDARD
SIST ISO 1597:1996

01-junij-1996

Polimerni materiali - Nemehčani acetat celuloze - Določanje deleža ocetne kisline

Plastics -- Unplasticized cellulose acetate -- Determination of acetic acid yield

Plastiques -- Acétate de cellulose non plastifié -- Détermination du titre en acide acétique

Ta slovenski standard je istoveten z: **ISO 1597:1994**

ICS:

83.080.20	Plastomeri	Thermoplastic materials

SIST ISO 1597:1996 **en**

INTERNATIONAL STANDARD

ISO 1597

Second edition
1994-10-15

Plastics — Unplasticized cellulose acetate — Determination of acetic acid yield

Plastiques — Acétate de cellulose non plastifié — Détermination du titre en acide acétique

Reference number
ISO 1597:1994(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 1597 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 1597:1975), of which it constitutes a minor revision.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Plastics — Unplasticized cellulose acetate — Determination of acetic acid yield

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 two methods for the determination of the acetic acid yield of unplasticized cellulose acetate.

These methods are intended for cellulose acetate without plasticizers and free of additives, fillers, dyes or other materials which affect the tests. When such materials are present, they shall first be removed by a method agreed between the contracting parties.

The methods are applicable to cellulose acetate having any acetic acid yield.

Method A is applicable to cellulose acetate in the form of finely divided powder. Method B is applicable to cellulose acetate in any physical form (powder, grains, flakes, etc.).

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, *Plastics — Unplasticized cellulose acetate — Determination of moisture content.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 acetic acid yield: The quantity of acetic acid, in grams, in 100 g of dry cellulose acetate, as calculated from the amount of sodium hydroxide necessary for the complete hydrolysis of the cellulose acetate.

4 Principle

4.1 Method A

A test portion of finely divided cellulose acetate is left in contact with a mixture of acetone and aqueous sodium hydroxide solution.

The amount of alkali consumed in hydrolysing the cellulose acetate is then determined by titration.

4.2 Method B

A test portion of cellulose acetate is dissolved in dimethylsulfoxide, and aqueous sodium hydroxide solution added.

The amount of alkali consumed in hydrolysing the cellulose acetate is then determined by titration.

When using method B, care must be taken to avoid direct contact of dimethylsulfoxide, which is toxic, with the human skin.

5 Method A

5.1 Reagents

During the determination, use only reagents of recognized analytical grade and distilled water as specified in 5.1.1.

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

5.1.2 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.

5.1.3 Sulfuric acid, solution, $c(H_2SO_4) \approx 0{,}5$ mol/l.

5.1.4 Sodium hydroxide, solution, $c(NaOH) \approx 1$ mol/l.

5.1.5 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0{,}5$ mol/l, carbonate-free.

5.1.6 Phenolphthalein solution, 10 g/l in ethanol.

In order to ensure that there will be a positive back-titration value in the blank tests, the molarity of the sulfuric acid (5.1.3) must be greater than half that of the sodium hydroxide (5.1.4).

5.2 Apparatus

5.2.1 Glass flasks, capacity 250 ml, with ground-glass stoppers.

5.2.2 Burettes, capacity 50 ml, graduated at 0,1 ml intervals.

5.2.3 Analytical balance, accurate to 1 mg.

5.3 Test sample

5.3.1 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.

5.3.2 Determine the moisture content of the sample of cellulose acetate in accordance with ISO 585.

5.4 Procedure

5.4.1 Carry out two tests and two blank tests for each determination.

5.4.2 Weigh, to the nearest 1 mg, 1,5 g ± 0,1 g of the test sample into a 250 ml flask (5.2.1). For the blank tests, prepare flasks containing only 65 ml of acetone (5.1.2) and proceed as indicated in 5.4.6, 5.4.7 and 5.4.8.

5.4.3 Shake the test portion evenly over the base of the flask and, without lifting the flask from the bench, carefully run in 15 ml of distilled water (5.1.1) around the sides of the flask to ensure even distribution over the base.

5.4.4 Add 65 ml of acetone (5.1.2). In order to prevent the formation of lumps, the first 10 ml shall be added very slowly, pouring it carefully around the sides of the flask while the flask is turned gently without its base leaving the bench.

5.4.5 Allow the flask and contents to stand for 30 min, then shake for 3 h or allow to stand overnight.

5.4.6 Add 25 ml of 1 mol/l sodium hydroxide solution (5.1.4) slowly with continual swirling. Shake for 3 h.

5.4.7 Wash down the stopper with distilled water (5.1.1), adding a total of approximately 50 ml of water to the contents of the flask. Add 25 ml of sulfuric acid solution (5.1.3) and about 0,5 ml of the phenolphthalein solution (5.1.6). Allow to stand, shaking if necessary, until any signs of pink coloration have disappeared from the insoluble matter.

5.4.8 Titrate with standard volumetric sodium hydroxide solution (5.1.5).

6 Method B

6.1 Reagents

6.1.1 Dimethylsulfoxide, analytical grade. The colour shall be less than that of 25 µmol/l iodine solution.

6.1.2 Sulfuric acid, solution, $c(H_2SO_4) \approx 0{,}25$ mol/l.

6.1.3 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0{,}5$ mol/l, carbonate-free.

6.1.4 Phenolphthalein solution, 10 g/l in ethanol.

6.2 Apparatus

6.2.1 Glass flasks, capacity 250 ml, with ground-glass stoppers.

6.2.2 Burettes, capacity 50 ml, graduated at 0,1 ml intervals.

6.2.3 Graduated cylinder, capacity 50 ml, graduated at 1 ml intervals.

6.2.4 Thermostatic bath, capable of being maintained at 80 °C ± 2 °C.

6.2.5 Analytical balance, accurate to 1 mg.

6.2.6 Suitable shaking machine.

6.3 Test sample

6.3.1 It is not necessary to grind the sample of cellulose acetate, irrespective of its form (powder, grains, flakes, etc.).

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

6.4 Procedure

6.4.1 Carry out two tests and two blank tests for each determination.

6.4.2 Weigh out, to the nearest 1 mg, 1,5 g ± 0,1 g of the test sample and put it in a 250 ml flask (6.2.1) containing 50 ml of dimethylsulfoxide (6.1.1), measured with the graduated cylinder (6.2.3). For the blank test prepare flasks containing only 50 ml of dimethylsulfoxide and proceed as indicated in 6.4.4, 6.4.6 and 6.4.7.

6.4.3 Put the flasks into the thermostatic bath at 80 °C ± 2 °C and shake as often as possible until the test portion is completely dissolved. Remove the flasks from the bath and let them cool at room temperature.

6.4.4 Add to each flask, with the burette (6.2.2), 47 ml of sodium hydroxide solution (6.1.3).

To ensure a fine precipitate, add the sodium hydroxide solution millilitre by millilitre, rather rapidly, shaking vigorously, up to 46 ml; let the level stabilize in the burette and complete to 47 ml, adding the last millilitre drop by drop.

NOTE 1 47 ml, is added rather than 50 ml to ensure that there will be a positive back-titration value for the blank.

6.4.5 Put the flask on the shaker (6.2.6) for 3 h to assist saponification. The flasks shall be placed in the vertical position to avoid as far as possible contact of the reagents with the neck of the flask.

6.4.6 Carefully wash down the necks and the stoppers of the flasks and add 50 ml of sulfuric acid solution (6.1.2). Replace the flasks on the shaking machine for half an hour.

6.4.7 Titrate the excess of sulfuric acid in the presence of phenolphthalein (6.1.4) with sodium hydroxide solution (6.1.3). Shake vigorously to keep the cellulose in suspension during the titration.

7 Expression of results

7.1 The acetic acid yield, expressed as grams of acetic acid per 100 g of dry cellulose acetate, is given by 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 (5.1.5 or 6.1.3) used;
- V_1 is the volume, in millilitres, of sodium hydroxide solution (5.1.5 or 6.1.3) required for the titration;
- V_2 is the volume, in millilitres, of sodium hydroxide solution (5.1.5 or 6.1.3) required for the blank test;
- m is the mass, in grams, of dry cellulose acetate in the test portion, calculated from the mass of the test portion and its moisture content determined in accordance with ISO 585;
- 6 is the mass, in milligrams, of acetic acid corresponding to 1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l.

7.2 The result is the mean of two determinations (i.e. of four tests and four blanks) and shall be expressed to one decimal place.

8 Precision

The precision of the two methods is the same and corresponds to 0,2 % of the mean value obtained.

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

c) the method used (A or B);

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

e) the result obtained (see 7.2);

f) the date of the test.