INTERNATIONAL STANDARD 1597

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

Plastics — Unplasticized cellulose acetate — Determination of acetic acid yield

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

First edition — 1975-06-15

UDC 678.544.4 : 547.292

Ref. No. ISO 1597-1975 (E)

Descriptors : plastics, cellulosic resins, cellulose acetate, chemical analysis, determination of content, acetic acid, volumetric analysis.

Price based on 3 pages

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 1597 and found it technically suitable for transformation. International Standard ISO 1597 therefore replaces ISO Recommendation R 1597-1970 to which it is technically identical.

ISO Recommendation R 1597 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 1597 into an International Standard :

Canada

Printed in Switzerland

Plastics — Unplasticized cellulose acetate — Determination of acetic acid yield

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two methods for the determination of 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 they are present, they must be separated 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 REFERENCE

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

3 DEFINITION

acetic acid yield : The quantity of acetic acid, in grams per 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

Leaving finely divided cellulose acetate in contact with a mixture of acetone and aqueous sodium hydroxide solution.

Determination, by titration, of the amount of alkali consumed in hydrolysing the cellulose acetate.

4.2 Method B

Taking the cellulose acetate into solution in dimethylsulphoxide and adding aqueous sodium hydroxide solution.

Determination, by titration, of the amount of alkali consumed in hydrolysing the cellulose acetate.

NOTE — In applying method B, care must be taken to avoid direct contact of the dimethylsulphoxide with the human skin, because of the toxic hazard.

5 METHOD A

5.1 Reagents

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

5.1.2 Acetone, analytical grade.

5.1.3 Sulphuric acid, approximately N solution.

5.1.4 Sodium hydroxide, approximately N solution.

5.1.5 Sodium hydroxide, standard 0,5 N solution, carbonate-free.

5.1.6 Phenolphthalein solution, 10 g/l in ethanol.

NOTE — In order to ensure that there will be a positive back-titration value in the blank, the normality of the sulphuric acid (5.1.3) should be greater than that of sodium hydroxide (5.1.4).

5.2 Apparatus

5.2.1 Flasks, 250 ml, with ground glass stoppers.

5.2.2 Burettes, 50 ml, graduated in 0,1 ml.

5.2.3 Analytical balance, accurate to 0,001 g.

5.3 Test sample

5.3.1 The sample of cellulose acetate must be in the form of powder passing entirely through a sieve of mesh 710 μm; if it does not, it shall be ground, or Method B used.

5.3.2 Determine the moisture content of the sample of cellulose acetate according to ISO/R 585.

5.4 Procedure

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

5.4.2 Weigh in a 250 ml flask (5.2.1) 1,5 ± 0,1 g of the test sample to the nearest 0,001 g. For the blank test, 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 should be added very slowly, being poured 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 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 approximately 50 ml of water to the contents of the flask. Add 25 ml of sulphuric 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 sodium hydroxide solution (5.1.5).

6 METHOD B

6.1 Reagents

6.1.1 Dimethylsulphoxide, analytical grade. The colour shall be less than that of 0,000 05 N iodine solution.

6.1.2 Sulphuric acid, approximately 0,5 N solution.

6.1.3 Sodium hydroxide, standard 0,5 N solution, carbonate-free.

6.1.4 Phenolphthalein solution, 10 g/l in ethanol.

6.2 Apparatus

6.2.1 Flasks, 250 ml, with ground glass stoppers.

6.2.2 Burettes, 50 ml, graduated in 0,1 ml.

6.2.3 Cylinder, 50 ml, graduated in 1 ml.

6.2.4 Thermostatic bath, at 80 ± 2 °C.

6.2.5 Analytical balance, accurate to 0,001 g.

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 of cellulose acetate according to ISO/R 585.

6.4 Procedure

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

6.4.2 Weigh 1,5 ± 0,1 g of the test sample to the nearest 0,001 g and put it in a 250 ml flask (6.2.1) containing 50 ml of dimethylsulphoxide (6.1.1), measured with the graduated cylinder (6.2.3). For the blank test prepare flasks containing only 50 ml of dimethylsulphoxide 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 ± 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 obtain a fine precipitate, add 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.

6.4.5 Put the flask on the shaker (6.2.6) for 3 h to assist saponification. The flasks shall be placed in a 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 sulphuric acid solution (6.1.2). Replace the flasks on the shaking machine for half an hour.

6.4.7 Titrate the excess of sulphuric 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 calculated from the formula

$$\text{acetic acid yield} = \frac{3\,(V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of exactly 0,5 N standard volumetric sodium hydroxide solution required for the sample;

V_2 is the volume, in millilitres, of exactly 0,5 N standard volumetric sodium hydroxide solution required for the blank test;

m is the mass, in grams, of dry cellulose acetate in the sample, calculated from the actual mass of the sample and its moisture content, as determined in accordance with 5.3.2 or 6.3.2.

7.2 The acetic acid yield is the mean of two determinations and is expressed to one decimal place.

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

8 TEST REPORT

The test report shall include the following particulars :

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

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

c) test method used (Method A or Method B);

d) acetic acid yield;

e) date of test.