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PLASTICS

DETERMINATION OF ACETIC ACID YIELD OF UNPLASTICIZED CELLULOSE ACETATE

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BRIEF HISTORY

The ISO Recommendation R 1597, *Plastics – Determination of acetic acid yield of unplasticized cellulose acetate,* 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. 1597, 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	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Sweden
Czechoslovakia	Japan	Switzerland
France	Netherlands	Turkey
Germany	Poland	U.A.R.
Hungary	Portugal	United Kingdom
India	Romania	U.S.A.

No Member Body opposed the approval of the Draft.

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

ISO Recommendation | R 1597 | June 1970

PLASTICS

DETERMINATION OF ACETIC ACID YIELD OF UNPLASTICIZED CELLULOSE ACETATE

1. SCOPE

This ISO Recommendation describes two methods for the determination of acetic acid yield of unplasticized cellulose acetate.

2. FIELD OF APPLICATION

2.1 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.

2.2 The methods apply to cellulose acetate having any acetic acid yield.

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

3. DEFINITION

The *acetic acid yield* is the quantity of acetic acid, in grammes 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 OF THE METHODS

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 should be ground, or Method B used.

5.3.2 The moisture content of the sample of cellulose acetate should be determined according to ISO Recommendation R 585, *Plastics – Determination of the moisture content of non-plasticized cellulose acetate.*

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 clauses 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 minutes, then shake for 3 hours 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 hours.

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 should be less than that of 0.00005 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 The moisture content of the sample of cellulose acetate should be determined according to ISO Recommendation R 585, *Plastics – Determination of the moisture content of non-plasticized cellulose acetate.*

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 clauses 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 hours to assist saponification. The flasks should 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 rinse 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.