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ISO

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ISO RECOMMENDATION

R 1875

PLASTICS

DETERMINATION OF ETHYL ETHER SOLUBLE MATTER IN PLASTICIZED CELLULOSE ACETATE

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BRIEF HISTORY

The ISO Recommendation R 1875, *Plastics – Determination of ethyl ether soluble matter in plasticized 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. 1875, which was circulated to all the ISO Member Bodies for enquiry in September 1969.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Hungary	Sweden
Belgium	Italy	Switzerland
Canada	Netherlands	Turkey
Chile	New Zealand	U.A.R.
Czechoslovakia	Poland	United Kingdom
France	Romania	U.S.A.
Germany	South Africa, Rep. of	U.S.S.R.
Greece	Spain	

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 1875 | December 1971

PLASTICS

DETERMINATION OF ETHYL ETHER SOLUBLE MATTER IN PLASTICIZED CELLULOSE ACETATE

1. SCOPE

1.1 This ISO Recommendation describes a method for determining the percentage of matter which can be extracted by ethyl ether from plasticized cellulose acetate.

1.2 This method is intended for plasticized cellulose acetate in any form, such as moulding material, sheet, manufactured articles, etc.

1.3 The extractable matter comprises principally the plasticizer.

2. PRINCIPLE

2.1 Conversion of plasticized cellulose acetate to film by dissolving and casting, then extraction by ethyl ether in a Soxhlet apparatus.

2.2 Removal of the ether by heating under vacuum, then weighing of the residual matter.

3. REAGENTS

3.1 *Dichloromethane*, analytical reagent grade, with relative density (20 °C/20 °C) from 1.321 to 1.331, and not less than 95 % (V/V) distilling between 39 and 40.5 °C under a pressure of 1013 mbar (760 mm Hg).

3.2 *Methanol*, analytical reagent grade, with relative density (20 °C/20 °C) from 0.792 to 0.795 and distillation range from 64.5 to 65.5 °C under a pressure of 1013 mbar (760 mm Hg).

3.3 *Diethyl ether*, analytical reagent grade, free from peroxides, with relative density (20 °C/20 °C) from 0.714 to 0.716 and distillation range from 34 to 35 °C under a pressure of 1013 mbar (760 mm Hg).

The residue on evaporation should be less than 1.5 mg per 100 ml.

4. APPARATUS

4.1 *Clear glass bottle*, 150 ml, tightly stoppered.

4.2 *Flat rectangular glass sheet*, 4 mm in thickness and about 200 mm by 400 mm.

4.3 *Film casting device* to lay a cast film of about 0.1 mm thickness. A suitable type is shown in the Figure.

4.4 *Soxhlet extraction apparatus* with

4.4.1 *extraction flask*, 250 ml, and

4.4.2 *condenser*.

4.5 *Thimble* for Soxhlet extractor.

4.6 *Thermostatic oven*, capable of operation at 50 ± 2 °C and at 105 ± 2 °C.

4.7 *Vacuum thermostatic oven*, capable of operation at 50 ± 2 °C.

4.8 *Desiccator* containing *dry silica gel*.

4.9 *Analytical balance*, accurate to the nearest 0.001 g.

4.10 *Shaking device* to hold the glass bottle (4.1).

5. PREPARATION OF TEST SAMPLE

5.1 Prepare a mixture of 90 parts of dichloromethane (3.1) and 10 parts of methanol (3.2) by volume, at room temperature.

5.2 Weigh 10 ± 0.2 g of plasticized cellulose acetate and introduce it into the glass bottle (4.1). Add 100 ± 2 ml of dichloromethane-methanol mixture and put the stoppered glass bottle on the shaker (4.10) until complete solution.

5.3 Using the film casting device (4.3), spread a layer of the solution on the glass sheet, so that after complete evaporation of the solvent at room temperature, a film of approximately 0.1 mm thickness will result.

5.4 Remove the film and cut it in strips about 5 mm wide, and of length suitable for the extraction thimble.

6. PROCEDURE A

6.1 Weigh the thimble (4.5), previously extracted by ether (3.3) and dried at 105 ± 2 °C, to the nearest 0.001 g, using a dried weighing bottle.

6.2 Place in the thimble about 2 g of freshly prepared test sample.

6.3 Weigh the 250 ml extraction flask (4.4.1), previously dried at 50 ± 2 °C, to the nearest 0.001 g and fill with about 200 ml of ether (3.3).

6.4 Connect the extraction flask to the Soxhlet extractor (4.4), and this to the condenser (4.4.2). Extract for 3 hours on a water bath.

6.5 Disconnect the extraction flask and evaporate most of the ether by using a water aspirator. Place in the vacuum thermostatic oven (4.7) at 50 ± 2 °C for 2 hours for complete removal of the ether. Place in the desiccator (4.8) until cool and weigh the extraction flask to the nearest 0.001 g.

6.6 Pull out the thimble from the Soxhlet extractor and let most of the ether evaporate at room temperature. Place in the vacuum thermostatic oven (4.7) at 50 ± 2 °C for 30 minutes and then in the thermostatic oven (4.6) at 105 ± 2 °C, for 3 hours.

6.7 Let the thimble cool in the desiccator and weigh to the nearest 0.001 g, using a dried weighing bottle.

7. PROCEDURE B

7.1 Dry a suitable quantity of the freshly prepared test sample at 105 ± 2 °C for 1 hour.

7.2 Weigh the thimble (4.5), previously extracted by ether (3.3) and dried at 105 ± 2 °C, to the nearest 0.001 g, using a dried weighing bottle.

7.3 Place in the thimble about 2 g of the freshly dried test sample and immediately reweigh to the nearest 0.001 g, using a dried weighing bottle.

7.4 Proceed according to clauses 6.3, 6.4 and 6.5.

8. EXPRESSION OF RESULTS

8.1 The ethyl ether soluble matter, expressed in grammes per 100 g of plasticized cellulose acetate, is calculated from one of the following equations :

Procedure A : Ether soluble matter $= 100 \times \frac{m_1}{m_1 + m_2}$

Procedure B : Ether soluble matter $= 100 \times \frac{m_1}{m_3}$