### ISO

#### INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

# ISO RECOMMENDATION R 1625

**PLASTICS** 

### AQUEOUS DISPERSIONS OF POLYMERS AND COPOLYMERS DETERMINATION OF DRY SOLIDS CONTENT AT 105 °C

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#### **BRIEF HISTORY**

The ISO Recommendation R 1625, Plastics - Aqueous dispersions of polymers and copolymers - Determination of dry solids content at  $105\,^{\circ}C$ , 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. 1625 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 Israel Sweden Belgium Italy Switzerland Czechoslovakia Japan Turkey Korea, Rep. of France U.A.R. Germany Netherlands United Kingdom Hungary Poland U.S.A. India Portugal U.S.S.R. Iran South Africa, Rep. of

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.

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ISO Recommendation

R 1625

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#### **PLASTICS**

### AQUEOUS DISPERSIONS OF POLYMERS AND COPOLYMERS DETERMINATION OF DRY SOLIDS CONTENT AT 105 °C

#### 1. SCOPE

This ISO Recommendation describes a procedure for the determination of dry solids content of aqueous dispersions at 105 °C.

The procedure is suitable for all aqueous polymer and copolymer dispersions which are chemically stable at the test temperature and which do not contain other volatile matter such as organic solvents.

The dry solids content of unplasticized aqueous polymer and copolymer dispersions consists essentially of the polymer or copolymer and of a small quantity of additives, such as emulsifiers, protective colloids, etc.

For plasticized dispersions, the plasticizer is included in the dry solids content.

Under the conditions of temperature and time adopted in this procedure, the product should not show any thermal degradation; otherwise different conditions should be used and specified in the test report.

#### 2. PRINCIPLE

Stoving at 105 °C for 1 hour of a sample spread in a thin regular layer, and weighing of the residue obtained.

#### 3. APPARATUS

3.1 Spreading and evaporation apparatus. It is essential to obtain an evenly spread film of about 0.15 mm for which one of the following devices is suitable:

#### 3.1.1 Apparatus A

The unit illustrated in Figures 1, 2 and 3 consists of two glass plates of diameter about 60 mm, one of the plates being provided with a support for the other one. The inside circular surfaces must be perfectly flat and smooth.

#### 3.1.2 Apparatus B

Aluminium foils about 0.1 mm thick, cut into rectangles of about 60 mm × 120 mm.

NOTE. – The apparatus described in clauses 3.1.1 and 3.1.2 are recommended particularly for very viscous dispersions, because they are capable of spreading the film automatically.

#### 3.1.3 Apparatus C

Metal or glass dish of 70 mm diameter having a rim with a minimum height of 3 mm.

NOTE. - This apparatus is specially recommended for very fluid dispersions, because it avoids any overflow.

- 3.2 Thermo-regulated oven at  $105 \pm 2$  °C, with natural air ventilation.
- 3.3 Desiccator with a suitable desiccant, for example calcium chloride or silica gel.
- 3.4 Balance, accurate to 0.0001 g.

#### 4. PROCEDURE

#### 4.1 Using apparatus A

- 4.1.1 Place the two parts of the unit (3.1.1), arranged as shown in Figure 3, in the oven for about 30 minutes at  $105 \pm 2$  °C. Then allow them to cool in the desiccator for about 30 minutes. At the end of this time, weigh the unit to the nearest 0.0001 g.
- 4.1.2 With the aid of a glass rod or a small spatula, pour  $1 \pm 0.2$  g of the dispersion being tested onto the centre of the lower plate. This operation is easier if the upper plate is taken away from its support.

Put the upper plate on the lower one, pressing it gently. The dispersion squeezed between the two plates spreads evenly. The diameter of the plates is such that if the right quantity of dispersion has been poured onto the centre of the lower plate, overflow of dispersion can be avoided.

If the dispersion is very fluid, check that no overflow takes place.

Weigh the whole assembly, leaving the plates one upon the other, using an analytical balance, to the nearest 0.0001 g.

4.1.3 Separate the two plates and suspend the upper one on its support.

Put the apparatus into the oven regulated at  $105 \pm 2$  °C and leave it there for 1 hour  $\pm 5$  minutes.

4.1.4 Take the unit out of the oven, place in the desiccator and allow to cool for about 30 minutes.

Weigh the apparatus to the nearest 0.0001 g with the upper plate fixed to its support.

#### 4.2 Using apparatus B

- 4.2.1 Leave the aluminium rectangle (3.1.2) in the oven for about 30 minutes at  $105 \pm 2$  °C. Then allow it to cool in the desiccator for about 30 minutes and finally weigh it to the nearest 0.0001 g.
- 4.2.2 Fold the rectangle double so as to form approximately a square; mark the fold, then unfold the sheet.

Pour in the middle of one square  $1.0 \pm 0.2$  g of the dispersion; promptly fold the sheet again and spread the material as evenly as possible, pressing gently with the fingers, taking care not to let the dispersion flow over the outer edges.

Weigh the whole to the nearest 0.0001 g.

- 4.2.3 Open out the sheet completely, place in the oven regulated at  $105 \pm 2$  °C and leave it there for 1 hour  $\pm 5$  minutes.
- 4.2.4 Take the sheet out of the oven, place in the desiccator and allow to cool about for 30 minutes.

Weigh the sheet to the nearest 0.0001 g.

#### 4.3 Using apparatus C

- 4.3.1 Leave the dish in the oven for about 30 minutes at  $105 \pm 2$  °C. Then allow it to cool in a desiccator for about 30 minutes. Weigh the dish to the nearest 0.0001 g.
- **4.3.2** Pour  $1 \pm 0.2$  g of the dispersion to be tested into the dish.

Rapidly weigh the whole unit to the nearest 0.0001 g.

- 4.3.3 Spread the dispersion as far as possible over the whole surface of the dish by manual stirring.
- 4.3.4 Place the dish in the oven, regulated at  $105 \pm 2$  °C, and leave it there for 1 hour  $\pm 5$  minutes.
- 4.3.5 Take the dish out of the oven. Make sure that the spreading of the dispersion has been complete. If not, it is possible that the test should be repeated with one of the other two types of apparatus.

Let the dish cool in the desiccator for about 30 minutes.

4.3.6 Weigh the dish to the nearest 0.0001 g.

#### 5. EXPRESSION OF RESULTS

5.1 The dry solids content of test dispersion, expressed as a percentage of the original mass is calculated from the following formula:

Dry solids content = 
$$\frac{m_1 \times 100}{m}$$

where

 $m_1$  is the mass, in grammes, of dry solids content weighed after stoving;

m is the mass, in grammes, of the test sample.

- 5.2 Make two determinations. The results should agree within 0.5 % absolute value. If they do not, carry out additional duplicate determinations until two successive results satisfy this condition.
- 5.3 Give as the final result the average of the two determinations.

#### 6. TEST REPORT

The test report should specify the following:

- (a) identification characteristics of the product tested;
- (b) apparatus used (A, B, C or other);
- (c) dry solids content.