INTERNATIONAL STANDARD ISO 119-1977 (E)/ERRATUM

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Plastics — Phenol-formaldehyde mouldings — Determination of free phenols — Iodometric method

ERRATUM

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Sub-clause 7.4, second paragraph, first line :

Replace "liberated" by "remaining".

INTERNATIONAL STANDARD 119

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Plastics — Phenol-formaldehyde mouldings — Determination of free phenols — Iodometric method

Plastiques — Pièces moulées à base de phénoplastes — Dosage des phénols libres — Méthode iodométrique

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ISO 119-1977 (E)

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.

International Standard ISO 119 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in November 1975.

It has been approved by the member bodies of the following countries :

Australia	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Israel	Spain
Chile	Italy	Sweden
Czechoslovakia	Japan	Switzerland
Egypt, Arab Rep. of	Mexico	Turkey
Finland	Netherlands	United Kingdom
France	New Zealand	U.S.A.
Germany	Peru	U.S.S.R.
Hungary	Poland	

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 119-1959, of which it constitutes a technical revision.

Printed in Switzerland

INTERNATIONAL STANDARD ISO 119-1977 (E)

Plastics — Phenol-formaldehyde mouldings — Determination of free phenols — Iodometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an iodometric method for the semi-quantitative determination of the amount of free phenols in phenol-formaldehyde mouldings.

NOTE — This International Standard does not provide an absolute measure of the free phenols present.

The amount of free phenols in a moulded article is influenced to a major extent by the degree of cure. Its evaluation is also of interest where the possibility of contamination of foodstuffs or other materials in contact with the article has to be considered.

2 REFERENCES

ISO/R 385, *Burettes.*

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO 648, *Laboratory glassware — One-mark pipettes.*[1)]

3 PRINCIPLE

Hot aqueous extraction of free phenols from a powdered test portion. Iodination of the aqueous extract by a solution of iodine in the presence of sodium tetraborate. Acidification, then immediate titration of the excess iodine with standard volumetric sodium thiosulphate solution, using a starch solution as indicator. Calculation of the result assuming the reaction of six atoms of iodine with each molecule of phenol.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

4.1 Sodium tetraborate, decahydrate ($Na_2B_4O_7.10H_2O$).

4.2 Sulphuric acid, 2 N solution.

4.3 Iodine, about 0,05 N solution.

Dissolve 6,35 g of iodine in approximately 40 ml of a 500 g/l solution of potassium iodide and dilute to 1 000 ml with water.

4.4 Sodium thiosulphate, 0,05 N standard volumetric solution.

4.5 Starch, approximately 2,5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Means for reducing the mouldings to a powder.

5.2 Sieve, with nominal apertures of 250 μm, complying with the requirements of ISO 565.

5.3 Balance, accurate to 0,01 g.

5.4 Glass-stoppered flask, 250 ml capacity.

5.5 Glass-stoppered iodine flask, 250 ml capacity.

5.6 Sintered glass crucible, or filter funnel with sintered disk, of pore size 40 to 90 μm.

Alternatively a filter funnel with hardened medium speed filter paper may be used.

5.7 Pipettes, capacities 5 and 10 ml, complying with the requirements of ISO 648.

5.8 Burette, complying with the requirements of ISO/R 385.

1) At present at the stage of draft. (Revision of ISO/R 648.)

6 PREPARATION OF TEST SAMPLE

Reduce a fully representative sample of the mouldings to powder by filing, milling, grinding, turning or drilling, taking care that no undue heating of the material occurs. Sieve the powder thus obtained, using the sieve (5.2), and use for the test that portion passing through the sieve. Keep the sample in a tightly stoppered flask until required.

The extraction with water (see 7.3) shall begin within 1 h of grinding the moulding.

NOTE — The method of reduction to powder can affect the results. In cases of dispute, or for referee purposes, the method should be agreed between the interested parties.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,01 g, 5 ± 0,1 g of the sieved material (see clause 6).

7.2 Blank test

Carry out a blank test by replacing the 5 ml of aqueous extract (7.3) with 5 ml of water and proceeding exactly as described in 7.4.

7.3 Preparation of the test solution

Place the test portion (7.1) in the flask (5.4) and cover it with ten times its mass of water at a temperature of 90 to 100 °C. Stopper the flask and shake it so that the powder is thoroughly wetted. Allow it to cool at room temperature, for 1 h, with occasional shaking. Then filter the contents of the flask through the sintered glass crucible or filter funnel (5.6).

7.4 Determination

Transfer, with a pipette (5.7), 5 ml of the filtered extract, corresponding to 0,5 g of the powder, to the iodine flask (5.5). Add 10 ml of the iodine solution (4.3) and 3 to 4 g of the sodium tetraborate (4.1), and dilute to about 100 ml with water. Immediately stopper the flask. Allow the mixture to stand for 10 min, and then add 20 to 30 ml of the sulphuric acid solution (4.2).

Immediately titrate the liberated iodine with the sodium thiosulphate solution (4.4), adding 2 ml of the starch solution (4.5) as indicator near the end-point of the titration and continuing until the blue starch/iodine colour has disappeared. A residual pale colour from the mouldings may be present at the end of the titration.

8 EXPRESSION OF RESULTS

The free phenols content, expressed as a percentage by mass of phenol (C_6H_5OH), is given by the formula

$$\frac{(V_1 - V_2) \times T \times 0{,}0157 \times 100}{0{,}5} = \frac{(V_1 - V_2) \times T \times 1{,}57}{0{,}5}$$

where

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (4.4) used in the blank test (7.2);

V_2 is the volume, in millilitres, of the sodium thiosulphate solution (4.4) used for the determination (7.4);

T is the normality of the sodium thiosulphate solution (4.4);

0,0157 is the mass, in grams, of phenol corresponding to 1 ml of exactly 1 N sodium thiosulphate solution;

0,5 is the mass, in grams, of the powdered sample corresponding to 5 ml of filtered extract (see 7.4).

If $T = 0{,}05$ N, the formula for calculation of free phenols content reduces to

$$0{,}157\ (V_1 - V_2)$$

9 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) full details necessary for the identification of the sample;

c) the method used for reducing the mouldings to powder;

d) amount of free phenols;

e) date of the test.