INTERNATIONAL STANDARD 120

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

Plastics — Phenol-formaldehyde mouldings — Determination of free ammonia and ammonium compounds — Colorimetric comparison method

Plastiques — Pièces moulées à base de phénoplastes — Dosage de l'ammoniac libre et des composés ammoniacaux — Méthode par comparaison colorimétrique

First edition — 1977-10-01

UDC 678.632 : (546.171.1 + 547.298.1) : 543.8 Ref. No. ISO 120-1977 (E)

Descriptors : plastics, phenol formaldehyde resins, mouldings, chemical analysis, determination of content, ammonia, colorimetric analysis, comparison analysis.

Price based on 2 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.

International Standard ISO 120 was developed by Technical Committee ISO/TC 61, *Plastics,* and was circulated to the member bodies in October 1975.

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

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

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 120-1959, of which it constitutes a technical revision.

Printed in Switzerland

Plastics — Phenol-formaldehyde mouldings — Determination of free ammonia and ammonium compounds — Colorimetric comparison method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a colorimetric comparison method for the semi-quantitative determination of the amount of ammonia in phenol-formaldehyde mouldings.

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

The amount of ammonia in a moulded article is of importance when corrosion of metal inserts or contamination of foodstuffs in contact with the article has to be considered.

2 REFERENCES

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

ISO 648, *Laboratory glassware — One-mark pipettes.*

3 PRINCIPLE

Hot aqueous extraction of free ammonia from a powdered test portion. Distillation of the aqueous extract in the presence of potassium permanganate and sodium hydroxide solution. Coloration of the distillate by reaction with Nessler reagent and comparison of the colour with that obtained in each of a series of standard matching solutions.

4 REAGENTS

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

4.1 Potassium permanganate.

4.2 Sodium hydroxide, 2 % *(m/m)* solution.

4.3 Standard matching stock solution, containing 10 mg of NH_3 per litre.

Dissolve 31,5 mg of ammonium chloride in 1 000 ml of water.

4.4 Nessler reagent.

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, conforming to ISO 565.

5.3 Balance, accurate to 0,01 g.

5.4 Glass-stoppered flask, 250 ml capacity.

5.5 Distillation apparatus, comprising a flask of 250 ml capacity, splash head and a condenser.

5.6 Filter funnel, with hardened medium speed filter paper.

5.7 Pipettes, capacities 2 and 10 ml, complying with the requirements of ISO 648.

5.8 Nessler cylinders, 50 ml capacity.

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 this powder, 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 7.2) 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 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, without suction, through the filter funnel (5.6). Transfer, with a pipette (5.7), 10 ml of the filtered extract, corresponding to 1 g of the powder, into the flask of the distillation apparatus (5.5).

NOTE — Before using the distillation apparatus, it is recommended that it should be freed from any ammonia which may be present by distilling a quantity of distilled water in it until the addition of 2 ml of Nessler reagent (4.4) to 50 ml of distillate does not give rise to any colour.

Add to the flask 0,1 g of potassium permanganate (4.1) and 10 ml of the sodium hydroxide solution (4.2). Slowly distil the mixture; collect the first 15 ml of the distillate in a Nessler cylinder (5.8) and dilute to 50 ml with water.

7.3 Determination

Transfer, with a pipette (5.7), 2 ml of the Nessler reagent (4.4) into the Nessler cylinder and determine the ammonia content by matching the colour of the test solution (7.2) with that of one of a series of standard matching solutions to which Nessler reagent has been added at the same time as to the test solution. To make the standard matching solutions transfer, with a pipette, 1, 2, 3, 4, 5 and 6 ml portions of standard matching stock solution (4.3) into each of a series of six Nessler cylinders (5.8) and dilute to 50 ml with water. These correspond to 0,01, 0,02, 0,03, 0,04, 0,05 and 0,06 mg of ammonia.

NOTE — If the 15 ml of distillate is found to contain more than 0,06 mg of ammonia, a 10 ml portion of the filtered extract shall be suitably diluted and a 10 ml portion of this diluted solution used in the distillation. An appropriate dilution factor should be used in the calculation.

8 EXPRESSION OF RESULTS

The amount of free ammonia and ammonium compounds, expressed as a percentage by mass of ammonia (NH_3), is given by the formula

$$\frac{m \times D}{10}$$

where

m is the mass, in milligrams, of ammonia contained in the appropriate standard matching solution from the series (see 7.3);

D is the dilution factor in the case where the filtrate is diluted before distillation :

$$D = \frac{V_2}{V_1}$$

where

V_1 is the volume in millilitres of the portion of the filtered extract before dilution (V_1 = 10 ml);

V_2 is the volume in millilitres of this 10 ml portion of the filtered extract after dilution.

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 ammonia;

e) date of the test.