INTERNATIONAL STANDARD 4614

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Plastics — Melamine-formaldehyde mouldings — Determination of extractable formaldehyde

Plastiques — Pièces moulées à base de résine mélamine-formaldéhyde — Détermination du formaldéhyde extractible

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FOREWORD

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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 4614 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in December 1975.

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

Australia	Hungary	Portugal
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Plastics — Melamine-formaldehyde mouldings — Determination of extractable formaldehyde

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the extractable formaldehyde in melamine-formaldehyde mouldings intended for use in contact with food and beverages.

2 PRINCIPLE

Certain liquids, simulating common food and beverage constituents, are placed in contact with mouldings of the sample material, under defined conditions. The formaldehyde content of the liquid is then determined and the quantity of formaldehyde extracted per unit area of contact with the moulding is calculated.

Two procedures for the determination of formaldehyde in the liquid are given.

3 REFERENCE

ISO 2227, *Formaldehyde solutions for industrial use — Determination of formaldehyde content.*

4 TEST SPECIMENS

4.1 Form

Moulded containers, for example beakers or cups, having an internal surface area of 150 to 250 cm^2 and capacity 150 to 250 cm^3 are suitable as test specimens. The quotient of the number expressing the surface wetted in square centimetres by that expressing the volume of liquid in cubic centimetres must lie between 0,75 and 1.

4.2 Number

Six specimens are required. The determination is carried out in duplicate with each of the three extraction liquids (clause 5).

5 EXTRACTION LIQUIDS

5.1 Water, distilled or de-ionized.

5.2 Acetic acid : 30 g/l solution of glacial acetic acid in distilled water.

5.3 Ethanol : 100 g/l solution of ethanol in distilled water.

6 EXTRACTION PROCEDURE

Rinse the test container with warm distilled water and dry thoroughly.

Place a suitable quantity (see 4.1) of the extraction liquid (clause 5) at 80 °C into the test container at room temperature. Cover the container with a watch glass to protect against evaporation and contamination, and allow to stand in air at room temperature for 30 min.

Transfer the extract (without washing) to a 250 ml conical flask, stopper the flask and cool the solution in a cold water bath to 20 ± 0,5 °C.

Immediately carry out the formaldehyde analysis by procedure A (7.3.2) or procedure B (8.3.2).

7 DETERMINATION OF FORMALDEHYDE — PROCEDURE A

7.1 Reagents

7.1.1 All reagents, including distilled water and extraction liquids, shall be free from formaldehyde in amounts detectable by the method described.

7.1.1.1 Water, distilled or de-ionized.

7.1.1.2 Chromotropic acid disodium salt (disodium 4,5-dihydroxy-2,7-naphthalene sulphonate) solution.

Dissolve 0,50 g of chromotropic acid disodium salt in 50 ml of distilled water. Transfer the solution to a 100 ml volumetric flask and make up to volume. Prepare the solution fresh each day.

7.1.1.3 Formaldehyde solutions.

All solutions shall be made up at 20 °C.

7.1.1.3.1 Formaldehyde stock solutions.

Pipette 25,0 ml of industrial formalin (containing approximately 400 g/l formaldehyde) into a 1 000 ml volumetric flask and make up to volume with distilled water. Determine the concentration of formaldehyde in the stock solution using the method described in ISO 2227.

This solution shall not be kept longer than one week.

7.1.1.3.2 Formaldehyde working solution A.

Pipette 10,0 ml of formaldehyde stock solution (7.1.1.3.1) into a 1 000 ml volumetric flask and make up to volume with the extraction liquid (clause 5) to be used. Mix thoroughly.

7.1.1.3.3 Formaldehyde working solution B (approximately 10 mg/l).

Pipette 10,0 ml of solution A (7.1.1.3.2) into a 100 ml volumetric flask and make up to the mark with the extraction liquid (clause 5) to be used.

NOTE — The exact concentration of formaldehyde in solution B can be calculated from the known formaldehyde content of the stock solution (7.1.1.3.1).

Solutions A and B shall be made up immediately prior to use.

7.1.1.4 Sulphuric acid, concentrated, 81 % (*m/m*), of analytical reagent grade.

7.2 Apparatus

7.2.1 Test tubes fitted with ground glass stoppers.

7.2.2 Water bath at 60 °C.

7.2.3 Cold water bath at 20 ± 0,5 °C.

7.2.4 Visible region spectrophotometer fitted with 10 mm path length glass cells.

7.2.5 Volumetric glassware.

7.2.5.1 Pipettes, 1, 10 and 25 ml, complying with the requirements of class A of ISO 648.

7.2.5.2 Two burettes, 5 ml.

7.2.5.3 Burette, 50 ml, complying with the requirements of class A of ISO/R 385.

7.2.5.4 Graduated flasks, 100 and 1 000 ml, complying with the requirements of class A of ISO 1042.

7.3 Procedure

Carry out the procedure specified in 7.3.1 and 7.3.2 for each extraction liquid.

7.3.1 *Establishment of the calibration curve*

To six separate test tubes (7.2.1), using the 5 ml burettes (7.2.5.2), add the volumes of solution B (7.1.1.3.3) shown in the following table and make each solution up to 1,0 ml using the appropriate amount of the extraction liquid (clause 5).

Solution B, ml	1,0	0,8	0,6	0,4	0,2	0,1
Extraction liquid, ml	0	0,2	0,4	0,6	0,8	0,9

This will give the equivalent of solutions containing from approximately 10 to 1 μg/ml formaldehyde. Into each of the test tubes pipette 1,0 ml of chromotropic acid disodium salt solution (7.1.1.2) and add slowly, while shaking, 8,0 ml of sulphuric acid (7.1.1.4) from the 50 ml burette (7.2.5.3). Mix thoroughly by shaking and stopper the tubes. Place the tubes in the water bath at 60 °C for 30 min. Remove the tubes from the water bath and allow them to stand at room temperature for 45 to 60 min (solutions C).

Transfer a portion of each solution C, in turn, to a 10 mm glass cell and measure its absorbance at 570 nm against distilled water.

Carry out a blank determination on the reagents alone using 1,0 ml of the extraction liquid (clause 5) in place of the formaldehyde solution.

Plot a graph of absorbance (10 mm cell) as ordinate against concentration of formaldehyde (μg/ml) as abscissa. This shall be a straight line passing through the origin of co-ordinates.

7.3.2 *Determination of formaldehyde*

Pipette 1,0 ml of extract (clause 6) (see note below) and 1,0 ml of chromotropic acid disodium salt solution (7.1.1.2) into a test tube and add slowly, while shaking 8,0 ml of sulphuric acid (7.1.1.4) from the burette (7.2.5.3). Mix thoroughly by shaking and stopper the tube. Place the tube in the water bath at 60 °C for 30 min. Remove the tube from the water bath and allow it to stand at room temperature for 45 to 60 min (solution C).

Transfer a portion of solution C to a 10 mm glass cell and measure its absorbance at 570 nm against distilled water.

Prepare a blank by treating 1,0 ml of the extraction liquid (clause 5) in exactly the same manner as the extract.

Read off the concentration of formaldehyde in the extract from the calibration curve.

NOTE — The procedure requires that the formaldehyde content of the extracts shall be in the concentration range 1,0 to 10,0 μg/ml. If the formaldehyde content of the extract is found on investigation to be greater than 10 μg/ml, then the extract shall be diluted, and a 1 ml aliquot of this diluted extract used for the determination.

8 DETERMINATION OF FORMALDEHYDE — PROCEDURE B

8.1 Reagents

8.1.1 All reagents, including distilled water and extraction liquids, shall be free from formaldehyde in amounts detectable by the method described.

8.1.1.1 Water, distilled or de-ionized.

8.1.1.2 Acetylacetone solution.

Dissolve 150 g of ammonium acetate in distilled water and add 3,0 ml of glacial acetic acid and 2,0 ml of acetylacetone

to this solution. Transfer the solution to a 1 000 ml volumetric flask and make up to volume with distilled water. Prepare the solution fresh each day.

8.1.1.3 Formaldehyde solutions.

All solutions shall be made up at 20 °C.

8.1.1.3.1 Formaldehyde stock solution.

Pipette 25,0 ml of industrial formalin (containing approximately 400 g/l formaldehyde) into a 1 000 ml volumetric flask and make up to volume with distilled water. Determine the concentration of formaldehyde in the stock solution using the method described in ISO 2227.

This solution shall not be kept longer than one week.

8.1.1.3.2 Formaldehyde working solution A.

Pipette 10,0 ml of formaldehyde stock solution (8.1.1.3.1) into a 1 000 ml volumetric flask and make up to volume with the extraction liquid (clause 5) to be used. Mix thoroughly.

8.1.1.3.3 Formaldehyde working solution B (approximately 10 mg/l).

Pipette 10,0 ml of solution A (8.1.1.3.2) into a 100 ml volumetric flask and make up to the mark with the extraction liquid (clause 5) to be used.

NOTE — The exact concentration of formaldehyde in solution B can be calculated from the known formaldehyde content of the stock solution (8.1.1.3.1).

Solutions A and B shall be made up immediately prior to use.

8.2 Apparatus

8.2.1 Test tubes fitted with ground glass stoppers.

8.2.2 Water bath at 60 °C.

8.2.3 Cold water bath at 20 ± 0,5 °C.

8.2.4 Visible region spectrophotometer fitted with 10 mm path length glass cells.

8.2.5 Volumetric glassware.

8.2.5.1 Pipettes, 5, 10 and 25 ml, complying with the requirements of class A of ISO 648.

8.2.5.2 Two burettes, 5 ml.

8.2.5.3 Burette, 50 ml, complying with the requirements of class A of ISO/R 385.

8.2.5.4 Graduated flasks, 100 and 1 000 ml, complying with the requirements of class A of ISO 1042.

8.3 Procedure

Carry out the procedure specified in 8.3.1 and 8.3.2 for each extraction liquid.

8.3.1 *Establishment of the calibration curve*

To six separate test tubes (8.2.1), using the 5 ml burettes (8.2.5.2), add the volumes of solution B (8.1.1.3.3) shown in the following table and make each solution up to 5,0 ml using the appropriate amount of the extraction liquid (clause 5).

Solution B, ml	4,0	3,0	2,0	1,0	0,5	0,25
Extraction liquid, ml	1,0	2,0	3,0	4,0	4,5	4,75

This will give the equivalent of solutions containing from approximately 8 to 0,5 µg/ml formaldehyde. Into each of the test tubes pipette 5 ml of acetylacetone solution (8.1.1.2). Mix thoroughly by shaking and stopper the tubes. Place the tubes in the water bath at 60 °C for 20 min. Remove the tubes from the water bath and allow them to stand at room temperature for 45 to 60 min (solutions C).

Transfer a portion of each solution C, in turn, to a 10 mm glass cell and measure its absorbance at 415 nm against distilled water.

Carry out a blank determination on the reagents alone using 5,0 ml of the extraction liquid (clause 5) in place of the formaldehyde solution.

Plot a graph of absorbance (10 mm cell) as ordinate against concentration of formaldehyde (µg/ml) as abscissa. This shall be a straight line passing through the origin of co-ordinates.

8.3.2 *Determination of formaldehyde*

Pipette 5,0 ml of extract (clause 6) (see note below) and 5,0 ml of acetylacetone solution (8.1.1.2) into a test tube. Mix thoroughly by shaking and stopper the tube. Place the tube in the water bath at 60 °C for 20 min. Remove the tube from the water bath and allow it to stand at room temperature for 45 to 60 min (solution C).

Transfer a portion of solution C to a 10 mm glass cell and measure its absorbance at 415 nm against distilled water.

Prepare a blank by treating 5,0 ml of the extraction liquid (clause 5) in exactly the same manner as the extract.

Read off the concentration of formaldehyde in the extract from the calibration curve.

NOTE — The procedure requires that the formaldehyde content of the extracts shall be in the concentration range 0,5 to 8,0 µg/ml. If the formaldehyde content of the extract is found on investigation to be greater than 8 µg/ml, then the extract shall be diluted, and a 5 ml aliquot of this diluted extract used for the determination.

9 EXPRESSION OF RESULTS

From the absorbance of the extract solution, deduce the formaldehyde content of the original extract.

Then :

1) formaldehyde extracted (μg/ml of extraction liquid) $= a \times f$

2) formaldehyde extracted (μg/cm^2 of specimen surface)

$$= \frac{a \times V \times f}{A}$$

where

a is the formaldehyde content, in micrograms per millilitre, of the test solution;

f is the dilution factor (if required by the terms of the note in 7.3.2 and 8.3.2);

V is the total volume, in millilitres, of extraction liquid used;

A is the area of specimen in contact with the extraction liquid, in square centimetres.

For each extraction liquid calculate the individual test results for the two determinations.

10 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the product tested;

c) description of test specimen, design, capacity and ratio of wetted surface to volume of liquid;

d) moulding conditions, curing time and temperature;

e) statement of which test procedure has been used (A or B);

f) the individual test results.