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Plastics - Melamine-formaldehyde mouldings - Determination of extractable formaldehyde (ISO 4614:1977)

Kunststoffe - Formteile aus Melamin-Formaldehyd-Harz - Bestimmung des extrahierbaren Formaldehyds (ISO 4614:1977)

Plastiques - Pièces moulées à base de résine mélamine-formaldéhyde - Détermination du formaldéhyde extractible (ISO 4614:1977)

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**Ta slovenski standard je istoveten z: EN ISO 4614:1999**

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**ICS:**

83.080.01	Polimerni materiali na splošno	Plastics in general
83.080.10	Duromeri	Thermosetting materials

**SIST EN ISO 4614:2000** en

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

EN ISO 4614

May 1999

ICS 83.080.00

English version

Plastics - Melamine-formaldehyde mouldings - Determination of extractable formaldehyde (ISO 4614:1977)

Plastiques - Pièces moulées à base de résine mélamine-formaldéhyde - Détermination du formaldéhyde extractible (ISO 4614:1977)

Kunststoffe - Formteile aus Melamin-Formaldehyd-Harz - Bestimmung des extrahierbaren Formaldehyds (ISO 4614:1977)

This European Standard was approved by CEN on 16 April 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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## Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 1999, and conflicting national standards shall be withdrawn at the latest by November 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

## Endorsement notice

The text of the International Standard ISO 4614:1977 has been approved by CEN as a European Standard without any modification.

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ALTERNATIVE APPROVED  
PROCESSED BY TECHNICAL COMMITTEE  
CEN/TC 249 "Plastics" (Secretariat: IBN)  
Approved by CEN/TC 249 on 1999-08-10  
CEN/TC 249 Secretariat: IBN  
CEN/TC 249 Secretariat: IBN

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**INTERNATIONAL STANDARD****4614**

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

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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*

First edition — 1977-07-01

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Ref. No. ISO 4614-1977 (E)

**Descriptors** : plastics, castings, melamine resins, food industry, food packaging, chemical analysis, determination of content, formaldehyde, extraction analysis.

Price based on 4 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 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
Austria	India	Romania
Belgium	Iran	Spain
Brazil	Ireland	Sweden
Canada	Israel	Switzerland
Czechoslovakia	Japan	Turkey
Finland	Mexico	United Kingdom
France	Netherlands	Yugoslavia
Germany	Poland	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

U.S.A.  
U.S.S.R.

# 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<sup>2</sup> and capacity 150 to 250 cm<sup>3</sup> 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.

## ISO 4614-1977 (E)

**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).

<b>Solution B, ml</b>	1,0	0,8	0,6	0,4	0,2	0,1
<b>Extraction liquid, ml</b>	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 coordinates.

**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 coordinates.

### 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.