



**SLOVENSKI STANDARD**  
**SIST EN ISO 9397:1999**

01-maj-1999

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Plastiki - Fenolne smole - Določitev prostega formaldehida - Metoda s hidroksilaminom  
hidrokloridom (ISO 9397:1995)

Plastics - Phenolic resins - Determination of free-formaldehyde content - Hydroxylamine  
hydrochloride method (ISO 9397:1995)

Kunststoffe - Phenolharze - Bestimmung des Gehaltes an freiem Formaldehyd -  
Hydroxylaminhydrochlorid-Methode (ISO 9397:1995)

Plastiques - Résines phénoliques - Dosage du formaldéhyde libre - Méthode au  
chlorhydrate d'hydroxylamine (ISO 9397:1995)

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Ta slovenski standard je istoveten z: **EN ISO 9397:1997**

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**ICS:**

83.080.10      Duromeri      Thermosetting materials

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EUROPEAN STANDARD

EN ISO 9397

NORME EUROPÉENNE

EUROPÄISCHE NORM

February 1997

ICS 83.080.10

Descriptors: see ISO document

English version

Plastics - Phenolic resins - Determination of  
free-formaldehyde content - Hydroxylamine  
hydrochloride method (ISO 9397:1995)

Plastiques - Résines phénoliques - Dosage du  
formaldéhyde libre - Méthode au chlorhydrate  
d'hydroxylamine (ISO 9397:1995)

Kunststoffe - Phenolharze - Bestimmung des  
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This European Standard was approved by CEN on 1997-01-05. 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.

The European Standards exist 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, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

**CEN**

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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EN ISO 9397:1997

### 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 August 1997, and conflicting national standards shall be withdrawn at the latest by August 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, 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 9397:1995 has been approved by CEN as a European Standard without any modification.

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INTERNATIONAL  
STANDARD

**ISO**  
**9397**

Second edition  
1995-05-01

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**Plastics — Phenolic resins —  
Determination of free-formaldehyde  
content — Hydroxylamine hydrochloride  
method**

iTeh STANDARD PREVIEW  
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*Plastiques — Résines phénoliques — Dosage du formaldéhyde libre —  
Méthode au chlorhydrate d'hydroxylamine*

<https://standards.iteh.ai/catalog/standards/sist/876610df-25ea-446a-8ff8-c53f657b91c0/sist-en-iso-9397-1999>



Reference number  
ISO 9397:1995(E)

**ISO 9397:1995(E)****Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9397 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 9397:1989), of which it constitutes a minor revision.

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International Organization for Standardization  
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

# Plastics — Phenolic resins — Determination of free-formaldehyde content — Hydroxylamine hydrochloride method

## 1 Scope

This International Standard specifies a method of chemically determining free formaldehyde in phenolic resins by potentiometric titration, in aqueous or organic solution. The method is applicable to resins with free-formaldehyde contents up to and including 15 % (*m/m*). For free-formaldehyde contents between 15 % (*m/m*) and 30 % (*m/m*), it may be necessary to adjust the concentrations of the standard volumetric solutions used accordingly. This method must not be used in the presence of hexamethylenetetramine.

are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9020:1994, *Binders for paints and varnishes — Determination of free-formaldehyde content of amino resins — Sodium sulfite titrimetric method.*

ISO 11402:1993, *Plastics — Condensation resins — Determination of free formaldehyde.*

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards

## 3 Choice of method

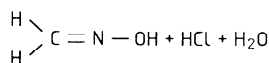
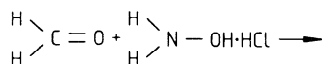
The methods available for the determination of free formaldehyde in condensation resins are summarized in table 1.

Table 1 — Choice of method

Method	Suitable for testing of	To be carried out in accordance with
Hydroxylamine hydrochloride method	Phenolic resins and furan resins (unmodified with urea or melamine resin)	ISO 9397
Sulfite method	Urea resins, melamine resins, furan resins, urea-melamine resins and furan-urea resins	ISO 9020
KCN method	Melamine-phenol resins, urea-phenol resins and urea-melamine-phenol resins	ISO 11402

## 4 Principle

The free formaldehyde in a test portion is subjected to an oximation reaction with hydroxylamine hydrochloride. The hydrochloric acid formed during this reaction is determined by potentiometric titration, using sodium hydroxide solution.



Oximation reaction

## 5 Reagents

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

**5.1 Hydroxylamine hydrochloride**, 10 % (m/m) solution the pH of which has been adjusted to 3,5 by the addition of sodium hydroxide solution.

**5.2 Sodium hydroxide**, standard volumetric solutions,  $c(\text{NaOH}) = 1 \text{ mol/l}$  and  $c(\text{NaOH}) = 0,1 \text{ mol/l}$ .

**5.3 Hydrochloric acid**, standard volumetric solutions,  $c(\text{HCl}) = 1 \text{ mol/l}$  and  $c(\text{HCl}) = 0,1 \text{ mol/l}$ .

**5.4 Methanol**, free of aldehydes and ketones.

**5.5 Propan-2-ol**, free of aldehydes and ketones.

## 6 Apparatus

Ordinary laboratory apparatus, plus the following:

**6.1 Balance**, accurate to 0,1 mg.

**6.2 pH-meter**, sensitive to 0,1 pH-units, equipped with a glass indicating electrode and a standard calomel reference electrode.

**6.3 Magnetic stirrer**.

**6.4 Graduated burettes**, of capacity 10 ml and 25 ml, the latter being for use if the formaldehyde content is likely to be greater than 5 % (m/m).

## 7 Procedure

### 7.1 Test temperature

Conduct the test at  $23 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ .

### 7.2 Test portion

Weigh, to the nearest 0,1 mg, into a 250 ml beaker, a test portion (from 1 g to 5 g, depending on the assumed formaldehyde content) chosen from table 2.

Table 2

Assumed formaldehyde content % (m/m)	Mass of test portion g
< 2	$5,0 \pm 0,2$
2 to 4	$3,0 \pm 0,2$
> 4	1 to 2

### 7.3 Determination

Add 50 ml of methanol (5.4), or 50 ml of a mixture of 3 volumes of propan-2-ol (5.5) and 1 volume of water, to the contents of the beaker, switch on the magnetic stirrer (6.3) and stir until the resin has dissolved and the temperature has stabilized at  $23 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ .

Introduce the electrodes of the pH-meter (6.2) into the solution and, using the 0,1 mol/l solution of hydrochloric acid (for neutralized resins) or the 1 mol/l solution (for highly alkaline resins) (5.3), adjust the pH to 3,5.

Pipette into the solution approximately 25 ml of the hydroxylamine hydrochloride solution (5.1) at  $23 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ .

Stir for  $10 \text{ min} \pm 1 \text{ min}$ .

Titrate rapidly, using the 1 mol/l solution of sodium hydroxide (or the 0,1 mol/l solution for low formaldehyde contents) (5.2) contained in a suitable-capacity burette (6.4), until the pH is 3,5.