



# SLOVENSKI STANDARD SIST EN ISO 11402:2005

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Phenolic, amino and condensation resins - Determination of free-formaldehyde content  
(ISO 11402:2004)

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Phenol-, Amin- und Kondensationsharze - Bestimmung des Gehalts an freiem  
Formaldehyd (ISO 11402:2004)

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Résines phénoliques, aminiques et de condensation - Dosage du formaldéhyde libre  
(ISO 11402:2004)

**Ta slovenski standard je istoveten z: EN ISO 11402:2005**

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EUROPEAN STANDARD

EN ISO 11402

NORME EUROPÉENNE

EUROPÄISCHE NORM

February 2005

ICS 83.080.10

Supersedes EN ISO 11402:1998, EN ISO 9397:1997

English version

## Phenolic, amino and condensation resins - Determination of free-formaldehyde content (ISO 11402:2004)

Résines phénoliques, aminiques et de condensation -  
Dosage du formaldéhyde libre (ISO 11402:2004)

Phenol-, Amin- und Kondensationsharze - Bestimmung des  
Gehalts an freiem Formaldehyd (ISO 11402:2004)

This European Standard was approved by CEN on 3 February 2005.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

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**EN ISO 11402:2005 (E)****Foreword**

The text of ISO 11402:2004 has been prepared by Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 11402:2005 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 2005, and conflicting national standards shall be withdrawn at the latest by August 2005.

This document will supersede EN ISO 9397:1997 and EN ISO 11402:1998.

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

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The text of ISO 11402:2004 has been approved by CEN as EN ISO 11402:2005 without any modifications.

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INTERNATIONAL  
STANDARD

ISO  
11402

Second edition  
2004-03-15

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**Phenolic, amino and condensation  
resins — Determination of free-  
formaldehyde content**

*Résines phénoliques, aminiques et de condensation — Dosage du  
formaldéhyde libre*

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ISO 11402:2004(E)

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## ISO 11402:2004(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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11402 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 11402:1993), as well as ISO 9020:1994 and ISO 9397:1995. The three standards have been combined into one, the sulfite procedure being taken from ISO 9020 and the hydroxylamine hydrochloride procedure from ISO 9397.

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# Phenolic, amino and condensation resins — Determination of free-formaldehyde content

## 1 Scope

This International Standard specifies three methods for determining the free-formaldehyde content in the following:

- a) **Phenolic resins**, by potentiometric titration in aqueous or organic solution (hydroxylamine hydrochloride procedure). The method is applicable to resins with free-formaldehyde contents up to and including 15 % by mass. For free-formaldehyde contents between 15 % by mass and 30 % by mass, it may be necessary to adjust the concentrations of the standard volumetric solutions used accordingly.
- b) **Amino resins and furan resins** (sulfite procedure). The method is applicable to resins resulting from the polycondensation of urea and melamine with formaldehyde and to furan resins resulting from the polycondensation of furfuryl alcohol with formaldehyde without further modification.
- c) **Condensation resins** (KCN procedure), including urea resins, furan resins, melamine resins and phenolic resins, as well as combinations and modifications of these resins.

The purpose of this International Standard is to establish recognized and useful determination procedures (for instance in order to check whether official regulations on the handling of hazardous workplace materials are observed). As far as possible, all formaldehyde-containing resins are to be included in this connection.

The free-formaldehyde content determined in formaldehyde condensation resins using this International Standard represents the actual content at the time of the determination. The value bears no quantitative relationship to the free-formaldehyde content during or after processing.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

## ISO 11402:2004(E)

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

**3.1 free formaldehyde**  
formaldehyde that is present in unbound form as formaldehyde, formaldehyde dihydrate (dihydroxymethylene) or polyoxymethylene in a condensation resin

### 4 Test procedures

#### 4.1 General

The choice of test procedure for the determination of free formaldehyde in condensation resins is made in accordance with Table 1.

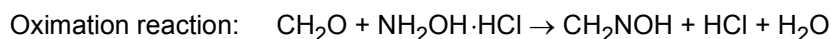
**Table 1 — Selection of procedure**

Procedure	Suitable for testing of
Hydroxylamine hydrochloride procedure	Phenolic resins, furan resins <sup>a</sup> (unmodified with urea or melamine resin)
Sulfite procedure	Urea resins, melamine resins, furan resins <sup>a</sup> , urea-melamine resins, furan-urea resins
KCN procedure <sup>b</sup>	Melamine-phenolic resins, urea-phenolic resins, urea-melamine-phenolic resins
<sup>a</sup> See Annex A, Clause A.2.	
<sup>b</sup> See Annex A, Clause A.3.	

#### 4.2 Hydroxylamine hydrochloride procedure

##### 4.2.1 Principle

The formaldehyde is converted to the oxime with hydroxylamine hydrochloride. The hydrochloric acid formed during this reaction is determined by potentiometric back-titration, using sodium hydroxide solution.



##### 4.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

**4.2.2.1 Hydroxylamine hydrochloride**, 10 % by mass solution, the pH of which has been adjusted to 3,5 by the addition of sodium hydroxide solution.

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

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

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

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

#### 4.2.3 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

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

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

**4.2.3.3 Magnetic stirrer**.

**4.2.3.4 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 % by mass.

#### 4.2.4 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

#### 4.2.5 Procedure

##### 4.2.5.1 Test temperature

Carry out the test at  $(23 \pm 1)$  °C.

##### 4.2.5.2 Test portion

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

**Table 2 — Mass of test portion**

Assumed formaldehyde content % by mass	Mass of test portion g
< 2	5,0 ± 0,2
2 to 4	3,0 ± 0,2
> 4	1 to 2

##### 4.2.5.3 Determination

Add 50 ml of methanol (4.2.2.4), or 50 ml of a mixture of 3 volumes of propan-2-ol (4.2.2.5) and 1 volume of water, to the contents of the beaker, switch on the magnetic stirrer (4.2.3.3) and stir until the resin has dissolved and the temperature has stabilized at  $(23 \pm 1)$  °C.

Introduce the electrodes of the pH-meter (4.2.3.2) 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) (see 4.2.2.3), adjust the pH to 3,5.

Pipette into the solution approximately 25 ml of hydroxylamine hydrochloride solution (4.2.2.1) at  $(23 \pm 1)$  °C.

Stir for  $(10 \pm 1)$  min.