INTERNATIONAL STANDARD

ISO 9397

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

Plastiques — Résines phénoliques — Dosage du formaldéhyde libre — Méthode au chlorhydrate d'hydroxylamine

Reference number
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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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.

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 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.*

3 Choice of method

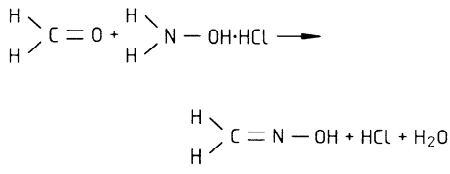
The methods available for the determination of free formaldehyde in condensation resins are summarized in table 1.

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, $c(\mathrm{NaOH}) = 1$ mol/l and $c(\mathrm{NaOH}) = 0{,}1$ mol/l.

5.3 Hydrochloric acid, standard volumetric solutions, $c(\mathrm{HCl}) = 1$ mol/l and $c(\mathrm{HCl}) = 0{,}1$ 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 °C ± 1 °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 ± 0,2
2 to 4	3,0 ± 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 °C ± 1 °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 °C ± 1 °C.

Stir for 10 min ± 1 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.

7.4 Blank test

Conduct a blank test in parallel with the determination, using the same procedure and the same reagents as in the determination, but omitting the test portion.

8 Expression of results

8.1 Method of calculation

The free-formaldehyde content, expressed as a percentage by mass, is given by the formula

$$\frac{3c(V_1 - V_0)}{m}$$

where

c is the actual concentration, in moles per litre, of the solution of sodium hydroxide (5.2) utilized;

V_0 is the volume, in millilitres, of the solution of sodium hydroxide (5.2) utilized for the blank test (7.4);

V_1 is the volume, in millilitres, of the solution of sodium hydroxide (5.2) utilized for the determination (7.3);

m is the mass, in grams, of the test portion (7.2).

8.2 Precision

Repeatability:	0,2 % (m/m) formaldehyde (independent of formaldehyde content of sample)
Reproducibility:	0,4 % (m/m) formaldehyde

9 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the resin tested;

c) the result of the test, as specified in 8.1;

d) the date of the test.

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Descriptors: plastics, thermosetting resins, phenoplasts, chemical analysis, determination of content, formaldehyde.

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