INTERNATIONAL STANDARD

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Plastics — Phenolic resins — Determination of free formaldehyde content

Plastiques — Résines phénoliques — Dosage du formaldéhyde libre

Reference number
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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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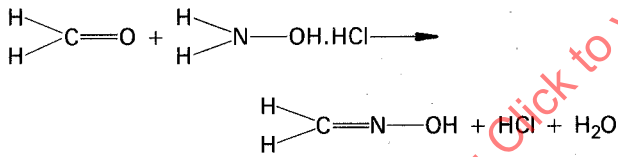
Plastics — Phenolic resins — Determination of free formaldehyde content

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.

2 Principle

Oximation of formaldehyde with hydroxylamine hydrochloride. Back-titration by potentiometry of the hydrochloric acid formed during this reaction, using sodium hydroxide solution.



Oximation reaction

3 Reagents

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

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

3.2 Sodium hydroxide, standard volumetric solutions, c(NaOH) = 1 mol/l and c(NaOH) = 0,1 mol/l.

3.3 Hydrochloric acid, standard volumetric solutions, c(HCl) = 1 mol/l and c(HCl) = 0,1 mol/l.

3.4 Methanol, free of aldehydes and ketones.

3.5 Propan-2-ol, free of aldehydes and ketones.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Balance, accurate to 0,1 mg.

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

4.3 Magnetic stirrer.

4.4 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).

5 Procedure

5.1 Test temperature

Conduct the test at 23 °C ± 1 °C.

5.2 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 the following table.

Table 1

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

5.3 Determination

Add 50 ml of the methanol (3.4), or 50 ml of a mixture of 3 volumes of the propan-2-ol (3.5) and 1 volume of water, to the contents of the beaker, switch on the magnetic stirrer (4.3) and stir until the resin has dissolved and the temperature has stabilized at 23 °C ± 1 °C.

Introduce the electrodes of the pH-meter (4.2) 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) (3.3), adjust the pH to 3,5.