INTERNATIONAL STANDARD

ISO 8988

First edition
1989-03-15

Plastics — Phenolic resins — Determination of hexamethylenetetramine content

Plastiques — Résines phénoliques — Dosage de l'hexaméthylènetétramine

Reference number
ISO 8988 : 1989 (E)

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.

International Standard ISO 8988 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Plastics — Phenolic resins — Determination of hexamethylenetetramine content

1 General

This International Standard specifies two methods for the determination of the hexamethylenetetramine ("hexa") content of phenolic resins. The two methods are equivalent. The Kjeldahl method described in clause 2 is not applicable if there are other components containing nitrogen in the phenolic resin. The perchloric acid method described in clause 3 is only applicable if there are no other basic or acidic additives in the resin.

2 Kjeldahl method

WARNING — For safety reasons, the Kjeldahl determination must be carried out in a well ventilated hood.

2.1 Scope

This clause specifies a method for the determination of total nitrogen, expressed as hexamethylenetetramine, in phenolic resins.

2.2 Principle

Transformation of the hexamethylenetetramine in a test portion to ammonium bisulfate by decomposition in hot concentrated sulfuric acid and in the presence of a catalytic mixture.

Transformation of the ammonium bisulfate to sodium sulfate and ammonia by reaction with sodium hydroxide.

Distillation of the ammonia and collection of the ammonia in hydrochloric acid.

Titration of the excess hydrochloric acid with a standard volumetric solution of sodium hydroxide in the presence of an indicator.

2.3 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, free of nitrogen, and only distilled water or water of equivalent purity.

2.3.1 Sulfuric acid, concentrated.

2.3.2 Kjeldahl catalytic mixture, consisting of 97 g of sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$), 1,5 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) and 1,5 g of selenium (Se).

2.3.3 Sodium hydroxide, 30 % (m/m) solution.

2.3.4 Hydrochloric acid, c(HCl) = 0,10 mol/l.

2.3.5 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,10 mol/l.

2.3.6 Mixed indicator, solution.

Dissolve 60 mg of methyl red and 40 mg of methylene blue in 100 ml of ethanol.

2.4 Apparatus

Ordinary laboratory apparatus and

2.4.1 Kjeldahl flask, capacity 250 ml or 300 ml, for the digestion.

2.4.2 Distillation apparatus (various models are available commercially).

2.4.3 Burette, capacity 50 ml, graduated in 0,1 ml steps, conforming with the requirements of ISO 385-1*).

*) ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1 : General requirements.*

2.4.4 Analytical balance, accurate to 1 mg.

2.4.5 Silicon carbide grits, for use as anti-bumping granules.

2.5 Procedure

2.5.1 Digestion

Into a Kjeldahl flask (2.4.1), weigh 1 g to 2 g of phenolic resin to the nearest 1 mg. Add 5 g of the catalytic mixture (2.3.2) and 25 ml of concentrated sulfuric acid (2.3.1). Heat carefully until the colour of the mixture being digested changes from black or amber to clear. When clear, increase the rate of heating until 5 min beyond the time of the colour change and, possibly, boiling. Allow the digested liquid to cool almost to room temperature, just short of solidification. Add carefully 100 ml of water and transfer the solution quantitatively into the flask of the distillation apparatus, rinsing with water. Add a few silicon carbide grits (2.4.5) to prevent bumping. Add 30 % (m/m) NaOH solution (2.3.3) to this solution until an alkaline reaction is obtained. Then distill over the ammonia given off, together with water vapour, into a receiver containing 50 ml of hydrochloric acid (2.3.4). Continue the distillation until about 300 ml of water has been collected.

2.5.2 Titration

When the distillation is completed, add a few drops of mixed indicator solution (2.3.6) to the contents of the receiver and titrate the excess hydrochloric acid with sodium hydroxide solution (2.3.5), using the burette (2.4.3).

2.6 Expression of results

The hexamethylenetetramine content, expressed as a percentage by mass, is given by the formula

$$\frac{0{,}35\,(V_0 - V_1)}{m_0}$$

where

V_0 is the volume, in millilitres, of hydrochloric acid (2.3.4) in the receiver of the distillation apparatus;

V_1 is the volume, in millilitres, of sodium hydroxide solution (2.3.5) used in the back-titration;

m_0 is the mass, in grams, of the test portion.

2.7 Reproducibility

The results are reproducible to within 0,30 % (m/m) hexamethylenetetramine.

2.8 Number of determinations

Carry out the determination in duplicate. If the results differ by more than 5 %, repeat the determination, again in duplicate. If not, calculate the arithmetic mean of the two individual results.

3 Perchloric acid method

3.1 Scope

This clause specifies a method for the determination of hexamethylenetetramine in phenolic resins by direct titration. The results of the determination may be affected by the presence of acidic or basic additives. In such cases, the use of the Kjeldahl method is recommended.

3.2 Principle

Determination of one of the tertiary amine groups of the hexamethylenetetramine in a test portion by titration with perchloric acid.

3.3 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade, free of nitrogen, and only distilled water or water of equivalent purity.

3.3.1 Hexamethylenetetramine, dry.

3.3.2 Acetone.

3.3.3 Perchloric acid, 70 % (V/V) solution.

WARNING — Perchloric acid is dangerous in the presence of organic matter since an explosion can occur if the perchloric acid is in excess.

3.4 Apparatus

Ordinary laboratory apparatus and

3.4.1 Magnetic stirrer.

3.4.2 Automatic burette, nominal volume at least 15 ml, graduated in 0,1 ml steps, with a stopcock made of polytetrafluoroethylene.

3.4.3 Beakers, capacity 100 ml.

3.4.4 Graduated cylinder, capacity 1 000 ml.

3.4.5 Analytical balances, accurate to 0,1 mg and 1 mg, respectively.

3.4.6 pH-meter.

3.5 Procedure

3.5.1 Preparation and titration of solution of perchloric acid in acetone

Introduce 8 ml of perchloric acid solution (3.3.3) into the 1 000 ml graduated cylinder (3.4.4), and dilute to 1 000 ml with acetone (3.3.2).

Standardize the resulting solution with hexamethylenetetramine (3.3.1) as described below.

Weigh, to the nearest 0,1 mg, about 150 mg to 170 mg of hexamethylenetetramine (3.3.1) into a 100 ml beaker (3.4.3).

Add 30 ml to 40 ml of acetone (3.3.2), and titrate as described in 3.5.2

NOTE — Darkening of the solution will not affect the titration results.

The titre T, expressed in milligrams of hexamethylenetetramine per millilitre of solution, is given by the formula

$$\frac{m_1}{V_2}$$

where

m_1 is the mass, in milligrams, of hexamethylenetetramine;

V_2 is the volume, in millilitres, of perchloric acid solution needed to reduce the pH to just below zero.

3.5.2 Titration

Into a 100 ml beaker weigh, to the nearest 1 mg, a quantity of phenolic resin equal to 100 times the titre determined as in 3.5.1, add 30 ml to 40 ml of acetone (3.3.2) and insert a magnetic stirring rod. Place the beaker on the magnetic stirrer (3.4.1). Insert the glass electrode of the pH-meter (3.4.6) and switch on the stirrer and the pH-meter. When the resin has dissolved, add the perchloric acid solution, prepared as in 3.5.1, slowly dropwise until the pH-value drops suddenly below zero. As the resin dissolves in acetone more rapidly than hexamethylenetetramine, the pH-reading may increase to above zero because residual hexamethylenetetramine may still be in the process of dissolving. Continue the titration until the pH-reading remains constant, slightly below zero.

3.6 Expression of results

The hexamethylenetetramine content, expressed as a percentage by mass, is given by the formula

$$\frac{V_2\, T \times 100}{m_0}$$

where

V_2 is the volume, in millilitres, of perchloric acid solution used for the titration;

T is the titre, expressed in milligrams of hexamethylenetetramine per millilitre, of the perchloric acid solution, as determined in 3.5.1;

m_0 is the mass, in milligrams, of the test portion.

3.7 Number of determinations

Carry out the determination in duplicate. If the results differ by more than 5 %, repeat the determination, again in duplicate. If not, calculate the arithmetic mean of the two individual results.

4 Test report

The test report shall include the following information:

a) a reference to this International Standard and the method used (Kjeldahl method or perchloric acid method);

b) a complete identification of the test resin;

c) the hexamethylenetetramine content, expressed as a percentage by mass, together with

— the individual results,

— the arithmetic mean;

d) the date of the test.

UDC 678.632 : 543.8 : 547.288.15

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

Price based on 3 pages