

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION R 1391

PARAFORMALDEHYDE FOR INDUSTRIAL USE

METHODS OF TEST

1st EDITION

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## BRIEF HISTORY

The ISO Recommendation R 1391, *Paraformaldehyde for industrial use – Methods of test*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1391, which was circulated to all the ISO Member Bodies for enquiry in February 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

|                |                |                       |
|----------------|----------------|-----------------------|
| Austria        | Ireland        | Romania               |
| Belgium        | Italy          | South Africa, Rep. of |
| Brazil         | Japan          | Spain                 |
| Czechoslovakia | Korea, Rep. of | Sweden                |
| France         | Netherlands    | Switzerland           |
| Germany        | New Zealand    | Thailand              |
| Hungary        | Poland         | Turkey                |
| Iran           | Portugal       | United Kingdom        |

The following Member Body opposed the approval of the Draft :

India

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

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## PARAFORMALDEHYDE FOR INDUSTRIAL USE

## METHODS OF TEST

## 1. SCOPE

This ISO Recommendation describes methods of test for paraformaldehyde for industrial use.

## 2. SAMPLE\*

The laboratory sample should have a mass of not less than 500 g. It should be preserved in a clean, dry and air-tight glass-stoppered bottle of such a size that it is nearly filled by the sample. If it has been necessary to seal the container, care should be taken to avoid contaminating the contents in any way.

If the material is in the form of lumps or flakes the test sample should be prepared from it by grinding and thoroughly mixing immediately before carrying out the tests described.

## 3. DETERMINATION OF ASH

## 3.1 Apparatus

Ordinary laboratory apparatus.

## 3.2 Procedure

Slowly burn about 50 g, weighed to the nearest gramme, of the laboratory sample in several portions in a platinum or silica basin previously weighed to the nearest 0.1 mg, and ignite finally in a furnace at  $600 \pm 30$  °C until all carbonaceous matter has disappeared. Cool in a desiccator and weigh to the nearest 0.1 mg. Repeat this series of operations of ignition, cooling, and weighing until the mass recorded is constant.

## 3.3 Expression of results

Ash is given, as a percentage by mass, by the following formula :

$$2 \times m_1$$

where  $m_1$  is the mass, in grammes, of residue.

\* Sampling of chemical products will form the subject of a further ISO Recommendation.

#### 4. DETERMINATION OF IRON CONTENT

##### 4.1 Principle

Conversion of any iron present in the test portion into the sulphate by evaporation of the test portion to dryness with sulphuric acid and photometric determination of the iron present using 2,2'-bipyridyl.

NOTE. — Although this method specifies the use of a spectrophotometer or photoelectric absorptiometer, it is permissible to employ, as an alternative method a visual procedure comparing the sample solution with a series of standard matching solutions (see clause 4.5.5).

##### 4.2 Reagents

Distilled water or water of equivalent purity should be used in the test.

4.2.1 *Sulphuric acid*  $\rho$  1.84 (g/ml), solution approximately 96 % (m/m), diluted 1 + 6 (V/V).

4.2.2 *Hydrogen peroxide*, 150 g/l solution.

4.2.3 *Hydroxylammonium chloride*, 100 g/l solution.

4.2.4 *Ammonium acetate*, 500 g/l solution.

4.2.5 *2,2'-bipyridyl*, 5 g/l hydrochloric acid solution.

Dissolve 0.5 g of 2,2'-bipyridyl in 100 ml of N hydrochloric acid solution.

4.2.6 *Standard iron solution*, containing 0.010 g of Fe per litre.

Dissolve 0.7022 g of ammonium iron (II) sulphate hexahydrate  $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$  in 50 ml of dilute sulphuric acid (4.2.1) and dilute to 1000 ml.

Dilute 100 ml of the solution thus obtained to 1000 ml.

1 ml of the resulting solution contains 10  $\mu\text{g}$  of iron (Fe).

##### 4.3 Apparatus

Ordinary laboratory apparatus and

4.3.1 *Spectrophotometer or photoelectric absorptiometer.*

##### 4.4 Preparation of the calibration curve

4.4.1 Place in a series of 400 ml beakers the following quantities of the standard iron solution (4.2.6) : 0, 2.0, 4.0, 7.0, 10.0, 15.0 and 20.0 ml.

4.4.2 Add to each, in successive small portions, 10 ml of the hydrogen peroxide solution (4.2.2) and 10 ml of the dilute sulphuric acid (4.2.1) then heat on a sand bath until acid fumes are evolved.

4.4.3 Allow to cool again to room temperature and transfer the solutions quantitatively to 100 ml one-mark volumetric flasks. Add, to each, 2 ml of the hydroxylammonium chloride solution (4.2.3). Mix and allow to stand for 2 minutes. Then add 30 ml of the ammonium acetate solution (4.2.4) and 5 ml of the 2,2'-bipyridyl solution (4.2.5). Dilute to the mark and mix thoroughly.

4.4.4 Measure the optical densities of the solutions in the spectrophotometer or the photoelectric absorptiometer (4.3.1), determining the optical density at a wavelength between 510 and 520 nm.

4.4.5 Prepare a calibration chart plotting optical densities as a function of the quantities of iron, in microgrammes, in 100 ml of the solutions.