International Standard 1391/5

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Paraformaldehyde for industrial use — Methods of test — Determination of aldehyde content — Titrimetric method

Paraformaldéhyde à usage industriel — Méthodes d'essai — Dosage de l'aldéhyde — Méthode titrimétrique

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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.

International Standard ISO 1391/5 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in January 1980.

It has been approved by the member bodies of the following countries :

Australia	France	Philippines
Austria	Germany, F. R.	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Bulgaria	Italy	Switzerland
China	Korea, Rep. of	Thailand
Czechoslovakia	Mexico	United Kingdom
Egypt, Arab Rep. of	Netherlands	USSR

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

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Paraformaldehyde for industrial use — Methods of test — Determination of aldehyde content — Titrimetric method

1 Scope and field of application

This part of ISO 1391 specifies a titrimetric method for the determination of the aldehyde content of paraformaldehyde for industrial use.

The method is applicable to solid paraformaldehyde of aldehyde contents, expressed as formaldehyde (HCHO), in the range 67 to 97 % (*m/m*).

This document should be read in conjunction with part 1 of ISO 1391 (see the annex).

2 Principle

Reaction of the aldehyde present in a test portion with neutralized sodium sulphite solution. Titration of the liberated sodium hydroxide with sulphuric acid solution using thymolphthalein as indicator.

3 Reactions

$$CH_2O + Na_2SO_3 + H_2O \rightarrow CH_2(OH)(SO_3Na) + NaOH$$

$$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity, which has been boiled and then cooled prior to use in a bottle closed with a stopper fitted with an absorption tube containing soda lime.

4.1 Sodium sulphite, approximately 130 g/l solution.

Dissolve approximately 130 g of anhydrous sodium sulphite or approximately 250 g of sodium sulphite heptahydrate ($Na_2SO_3.7H_2O$) in water and dilute to 1 000 ml.

Prepare this solution at the time of use.

4.2 Sodium hydroxide, 4 g/l solution.

4.3 Sulphuric acid, 4,9 g/l solution.

4.4 Sulphuric acid, standard volumetric solution, $c(1/2\ H_2SO_4) = 1$ mol/l.

4.5 Thymolphthalein, 2 g/l solution.

Dissolve 0,2 g of thymolphthalein in 60 ml of 95 % (*V/V*) ethanol in a 100 ml flask, add just enough of the sodium hydroxide solution (4.2) to produce a pale blue colour, dilute to 100 ml with water and mix.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Conical flasks, 500 ml capacity, of borosilicate glass and fitted with ground glass stoppers.

5.2 Magnetic stirrer.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, 1,1 ± 0,1 g of the laboratory sample into one of the conical flasks (5.1).

6.2 Neutralization of sodium sulphite solution

Measure 75 ml of the sodium sulphite solution (4.1) into one of the conical flasks (5.1). Add 2 drops (approximately 0,1 ml) of the thymolphthalein solution (4.5) and then the sulphuric acid solution (4.3) until the blue colour just disappears.

6.3 Determination

Add 75 ml of water to the test portion (6.1) in the conical flask. Stir the resulting suspension with the magnetic stirrer (5.2) for approximately 5 min. Add 2 drops (approximately 0,1 ml) of the thymolphthalein solution (4.5) and the sodium hydroxide solution (4.2), drop by drop, until a pale blue colour is just perceptible. Add the neutralized sodium sulphite solution. Stir the mixture with the magnetic stirrer (5.2) until all the paraformaldehyde has dissolved and the reaction is complete (5 min is usually adequate). Titrate the solution with the standard volumetric sulphuric acid solution (4.4) until the blue colour just disappears.

7 Expression of results

The aldehyde content, expressed as a percentage by mass of formaldehyde (HCHO), is given by the formula

$$V \times 0{,}030\ 03 \times \frac{100}{m} = \frac{3{,}003 \times V}{m}$$

where

m is the mass, in grams, of the test portion (6.1);

V is the volume, in millilitres, of the sulphuric acid solution (4.4) used in the titration;

0,030 03 is the mass, in grams, of formaldehyde corresponding to 1 ml of sulphuric acid solution, $c(1/2\ H_2SO_4) = 1$ mol/l exactly.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

Annex

ISO publications relating to paraformaldehyde for industrial use

ISO 1391/1 — General.

ISO 1391/2 — Determination of ash.

ISO 1391/3 — Determination of iron content — 2,2'-Bipyridyl photometric method.

ISO 1391/4 — Determination of water-insoluble matter.

ISO 1391/5 — Determination of aldehyde content — Titrimetric method.