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INTERNATIONAL STANDARD



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**Formaldehyde solutions for industrial use —
Determination of methanol content**

First edition — 1972-12-01

UDC 661.727.1 : 542.61 : 547.261

Ref. No. ISO 2228-1972 (E)

Descriptors : formaldehyde, chemical analysis, determination of content, volumetric analysis, carbinols.

Price based on 3 pages

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 2228 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in July 1971 by the Member Bodies of the following countries :

Austria	Ireland	South Africa, Rep. of
Czechoslovakia	Israel	Sweden
Egypt, Arab Rep. of	Italy	Switzerland
France	Netherlands	Turkey
Germany	New Zealand	United Kingdom
Hungary	Romania	U.S.S.R.

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Belgium
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Printed in Switzerland

Formaldehyde solutions for industrial use — Determination of methanol content

WARNING

Formaldehyde is toxic. It is therefore necessary to avoid inhaling its vapour during sampling and testing.

1 SCOPE

This International Standard specifies a method for the determination of the methanol content of formaldehyde solutions for industrial use.

NOTE — The result obtained by this method will include the methanol freed from combined forms by the procedure, e.g. by saponification of any methyl formate present. These combined forms are however not usually present at sufficiently high levels to affect the result of the determination.

2 FIELD OF APPLICATION

This method is intended for methanol contents greater than 0,2 % and significant errors may occur if the methanol content is less than this value.

3 PRINCIPLE

Esterification of the methanol present with formic acid using sulphuric acid as a catalyst. Distillation of the methyl formate into sodium hydroxide solution, and back-titration of the excess sodium hydroxide with a standard volumetric sulphuric acid solution, using phenolphthalein as indicator.

4 REAGENTS

Distilled water, or water of equivalent purity, freshly boiled and cooled and neutral to phenolphthalein, shall be used in the test.

4.1 Formic acid, 80 % (*m/m*) solution.

4.2 Sulphuric acid, ρ 1,84 g/ml, approximately 96 % (*m/m*) solution, diluted 1 + 3 by volume.

4.3 Acetone, pure, neutral.

4.4 Sodium hydroxide, approximately N solution.

4.5 Sulphuric acid, N standard volumetric solution.

4.6 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (V/V) ethanol and add approximately 0,1 N sodium hydroxide solution until faintly pink.

5 APPARATUS

Ordinary laboratory apparatus and the following apparatus (see Figure), constructed of borosilicate glass and having ground glass joints, comprising :

5.1 Flask, capacity 100 ml.

5.2 Vigreux type column, of effective length 400 mm and inside diameter 20 mm. The top 300 mm of this column is covered by a sleeve, of 30 mm inside diameter, the upper end of which is open to the atmosphere and the lower end of which is fused to the column. This column is fitted with an upper lateral tube, connected to a **condenser**.

5.3 Thermometer, covering the range 0 to 100 °C and graduated in 0,5 °C divisions.

5.4 Special adaptor enabling the distilled ester to be collected without loss to the atmosphere.

5.5 Conical flask, capacity 200 ml.

6 SAMPLING

Follow the principles given in ISO . . .¹⁾

Attention is drawn to the following recommendation. Place the laboratory sample, representative of the material taken from the bulk, in a clean, dry, and air-tight glass stoppered bottle of such a size that it is nearly filled by the sample.

If it is necessary to seal this bottle care shall be taken to avoid the risk of contamination.

1) Sampling from the consignment of the product will form the subject of a future International Standard.

Owing to polymerization, paraformaldehyde will tend to be deposited on standing and this will occur more rapidly if the temperature is allowed to fall below 25 °C. Accordingly the material shall be sampled as soon as possible after receipt.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, a mass of about 6 g in the case of 40 % formaldehyde solutions containing about 8 % of methanol.

In the case of formaldehyde solutions containing less than 8 % of methanol, increase the mass of the test portion to up to 50 g so that at least 20 ml of N sulphuric acid solution is used in the back-titration.

7.2 Determination

By means of pipettes, place in the distillation flask (5.1), 20 ml of the formic acid solution (4.1), 5 ml of the sulphuric acid solution (4.2) and 10 ml of the acetone (4.3). Cool to ambient temperature, then introduce the test portion (7.1).

Fit the flask (5.1) to the column (5.2). Fill the sleeve fitted to the outside of the column with water at ambient temperature so as to regulate the rate of distillation and permit separation of the methyl formate formed. The water in the sleeve is not renewed or cooled during the test. Fit the thermometer (5.3) to the top of the Vigreux column (5.2) and also the adaptor (5.4), of which the outlet tube to the atmosphere is partly filled with 3 ml of water.

Place 50 ml of the sodium hydroxide solution (4.4) in the conical flask (5.5) and place the flask under the adaptor (5.4), so that the end of the latter is beneath the surface of the liquid.

Gently heat the bottom of the flask (5.1), so that the methyl formate produced separates slowly in the column (5.2).

When the distillation of the formate is complete (which takes approximately 45 min), close the tap of the adaptor and continue to heat gradually until 5 ml of acetone (measured in the adaptor) is distilled, at approximately 56 °C. The vapour from this acetone displaces any methyl formate that may remain in the apparatus.

Open the tap of the adaptor again and then rinse the condenser (5.2) and the whole of the adaptor (5.4) with water; do not rinse the column. Add the washings to the sodium hydroxide solution in the conical flask (5.5) and immediately stopper the latter. Shake the contents of the conical flask and, after waiting for at least 20 min, back-titrate with the sulphuric acid standard volumetric solution (4.5) in the presence of the phenolphthalein solution (4.6).

7.3 Blank test

Carry out a blank test using exactly the same procedure (including the conditions of distillation and rinsing) with the same quantities of reagents but replacing the test portion by water.

8 EXPRESSION OF RESULTS

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

$$\frac{3,2 \times (V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of the sulphuric acid standard volumetric solution (4.5) used in the blank test;

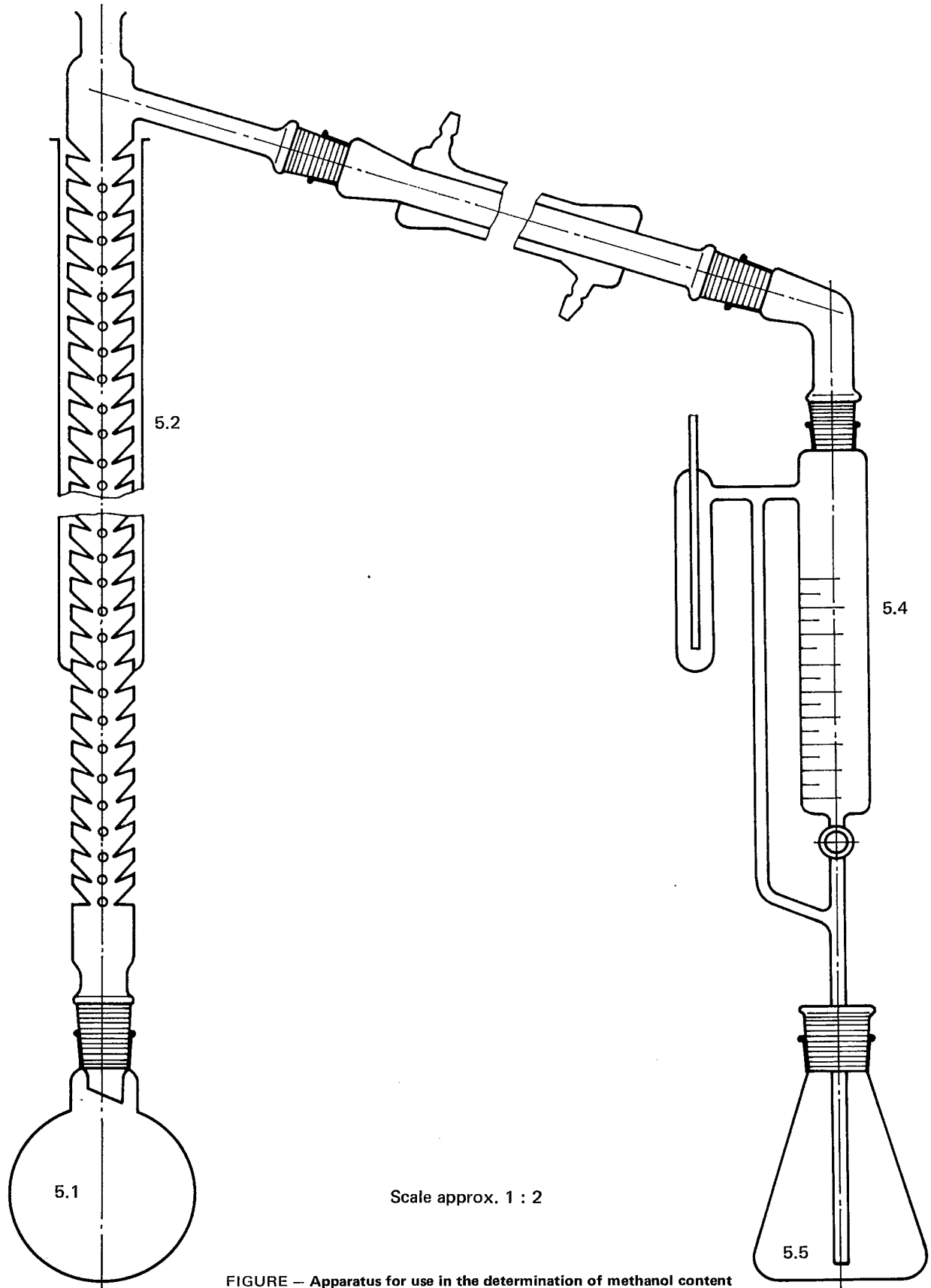
V_2 is the volume, in millilitres, of the sulphuric acid standard volumetric solution (4.5) used for the determination;

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

9 TEST REPORT

The test report shall include the following particulars :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard, or regarded as optional.



Scale approx. 1 : 2

FIGURE - Apparatus for use in the determination of methanol content