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INTERNATIONAL STANDARD ISO 731/VII

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Formic acid for industrial use — Methods of test — Part VII : Determination of low contents of other volatile acids — Titrimetric method after distillation

Acide formique à usage industriel — Méthodes d'essai —
Partie VII : Détermination de faibles teneurs en autres acides volatils — Méthode titrimétrique après distillation

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1913-1971 and found it technically suitable for transformation. Number 1913, however, has been changed to 731/VII. International Standard ISO 731/VII therefore replaces ISO Recommendation R 1913-1971, to which it is technically identical.

ISO Recommendation R 1913 had been approved by the member bodies of the following countries :

Australia	India	Spain
Austria	Iran	Sweden
Belgium	Israel	Switzerland
Czechoslovakia	Japan	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Portugal	U.S.A.
Greece	Romania	U.S.S.R.
Hungary	South Africa, Rep. of	

No member body had expressed disapproval of the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

Formic acid for industrial use — Methods of test — Part VII : Determination of low contents of other volatile acids — Titrimetric method after distillation

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 731 specifies a titrimetric method after distillation for the determination of low contents of volatile acids other than formic acid in formic acid for industrial use.

The method is applicable to products containing less than 0,5 % (*m/m*) of other volatile acids, expressed as acetic acid.

NOTE — The method specified in part III (see the annex) is applicable to formic acid containing between 0,5 and 6,0 % (*m/m*) of other acids, expressed as acetic acid. Consideration is to be given to a gas-liquid chromatographic method.

This document should be read in conjuction with part I (see the annex).

2 PRINCIPLE

Decomposition of most of the formic acid in a test portion by sulphuric acid and of the remainder by chromic acid.

Steam distillation of acetic acid and/or other volatile acids and titration of the distillate with standard volumetric sodium hydroxide solution in the presence of phenolphthalein as indicator.

3 REAGENTS

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

3.1 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution or approximately 36 N.

3.2 Chromic acid solution.

Dissolve 100 g of chromium(VI) oxide (CrO_3) in 100 ml of water.

3.3 Potassium hydroxide, approximately 1 N solution.

3.4 Sodium hydroxide, 0,05 N standard volumetric solution.

3.5 Phenolphthalein, 1 g/l in 95 % (*V/V*) ethanol.

Dissolve 0,1 g of phenolphthalein in 100 ml of 95 % (*V/V*) ethanol.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Apparatus for decomposition of formic acid, as shown in the figure, with ground glass joints and consisting of the following components :

A — *Round-bottomed flask*, with one central and one angled side neck, of capacity 250 ml.

B — *Magnetic stirring bar*, totally enclosed in borosilicate glass or polytetrafluoroethylene (PTFE), and capable of withstanding concentrated sulphuric acid at 100 °C and hot chromic acid.

C — *Boiling water bath*.

D — *Electric heater incorporating a magnetic stirrer*.

E — *Water-cooled reflux condenser*.

F — *Bubbler tube*.

G — *Dropping funnel*, of capacity 100 ml.

H — *Pressure regulator*.

The flask (A) is connected to the reflux condenser (E) and the dropping funnel (G). The reflux condenser (E) is connected by means of a glass tube to a bubbler tube (F), which is filled to a depth of about 30 mm with the potassium hydroxide solution (3.3). The dropping funnel (G) is connected by a rubber tube to the pressure regulator (H). The pressure regulator is filled with water to obtain a pressure sufficiently high to overcome the resistance of the bubbler tube (F).

4.2 Steam distillation apparatus, with a distillation flask of capacity 1 000 ml.

5 PROCEDURE

5.1 Test portion

In the dropping funnel (G), place about 30 g of the laboratory sample, weighed by difference to the nearest 0,1 g.

5.2 Determination

Introduce 45 ml of the sulphuric acid solution (3.1) into the flask (A). Fill the bubbler tube (F) with the potassium

hydroxide solution (3.3) to a depth of about 30 mm and fill the pressure regulator (H) with water. Assemble the apparatus and heat on the boiling water bath (C) whilst stirring with the magnetic stirrer (B/D). Add the test portion (5.1) at a rate of 1 drop per 3 to 4 s into the heated sulphuric acid solution. The decomposition of the formic acid begins, forming carbon monoxide which passes through the bubbler tube.

WARNING — It is essential that an effective outlet be provided from the bubbler tube to the open air outside the laboratory or to a fume cupboard with a good draught to ensure complete removal of the toxic carbon monoxide.

When all the test portion has been added, continue heating the contents of the flask (A) for 15 min on the boiling water bath (C).

Allow to cool to about 50 °C, then rinse the contents of the bubbler tube (F) through the reflux condenser (E) into the flask (A), using 45 ml of water.

WARNING — Take care during this operation since a base and water are being added to a concentrated acid.

Disconnect the pressure regulator (H) from the dropping funnel (G) and introduce 10 ml of the chromic acid solution (3.2) into the dropping funnel. Add this solution, drop by drop, to the flask (A) until gas is no longer evolved and the colour of the solution indicates a distinct excess (about 5 ml) of chromic acid. Heat the contents of the flask for 15 min on the boiling water bath (C).

Disconnect the apparatus, transfer the contents of the flask (A) quantitatively to the steam distillation apparatus (4.2) with 50 ml of water in two or three portions, and distil fractions of about 150 ml into 300 ml conical flasks until about 150 ml remains in the steam distillation flask. Close each conical flask and protect the contents from absorption of carbon dioxide by means of a soda-lime tube.

Allow the contents of each flask to cool and then titrate with the sodium hydroxide solution (3.4) in the presence of a few drops of the phenolphthalein solution (3.5).

6 EXPRESSION OF RESULTS

The content of volatile acids other than formic acid, expressed as a percentage by mass of acetic acid (CH_3COOH), is given by the formula

$$\frac{V \times 0{,}003\,0 \times 100}{m} = \frac{0{,}30 \times V}{m}$$

where

V is the total volume, in millilitres, of the standard volumetric hydroxide solution (3.4) used in titrating the contents of all the conical flasks;

m is the mass, in grams, of the test portion (5.1);

0,003 0 is the mass, in grams, of acetic acid corresponding to 1 ml of exactly 0,05 N sodium hydroxide solution.

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.

Dimensions in millimetres

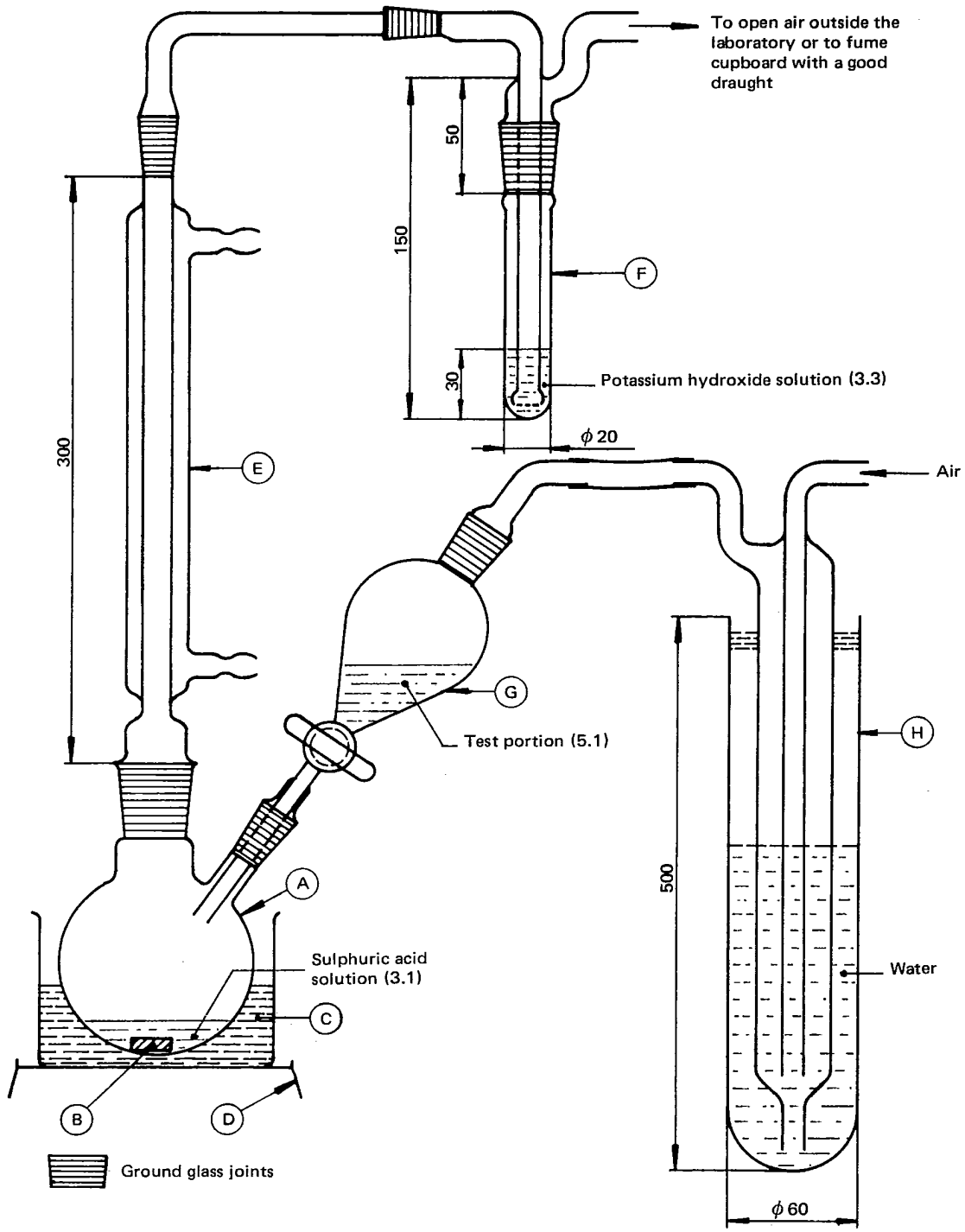

FIGURE — **Apparatus for decomposition of formic acid (4.1)**

ANNEX

ISO PUBLICATIONS RELATING TO FORMIC ACID FOR INDUSTRIAL USE

ISO 731/I – General.

ISO 731/II – Determination of total acidity – Titrimetric method.

ISO 731/III – Determination of content of other acids – Potentiometric method.

ISO 731/IV – Visual limit test for inorganic chlorides.

ISO 731/V – Visual limit test for inorganic sulphates.

ISO 731/VI – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 731/VII – Determination of low contents of other volatile acids – Titrimetric method after distillation.