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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1913

FORMIC ACID FOR INDUSTRIAL USE

DETERMINATION OF LOW CONTENTS OF VOLATILE ACIDS OTHER THAN FORMIC ACID (LESS THAN 0.5 % (m/m) CALCULATED AS ACETIC ACID)

VOLUMETRIC METHOD

1st EDITION

January 1971

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BRIEF HISTORY

The ISO Recommendation R 1913, *Formic acid for industrial use – Determination of low contents of volatile acids other than formic acid (less than 0.5 % (m/m), calculated as acetic acid) – Volumetric method,* 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. 1913, which was circulated to all the ISO Member Bodies for enquiry in February 1970. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

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

ISO Recommendation R 1913 January 1971

FORMIC ACID FOR INDUSTRIAL USE

DETERMINATION OF LOW CONTENTS OF VOLATILE ACIDS OTHER THAN FORMIC ACID (LESS THAN 0.5 % (m/m) CALCULATED AS ACETIC ACID)

VOLUMETRIC METHOD

INTRODUCTION

This ISO Recommendation supplements ISO Recommendation R 731, *Formic acid for industrial use – Methods of test*, which describes the following methods :

- determination of total acidity,
- determination of acids other than formic acid (see Note in section 1),
- limit test for inorganic chlorides,
- limit test for inorganic sulphates,

and ISO Recommendation R 1707, *Formic acid for industrial use – Determination of iron content – 2,2′-bipyridyl photometric method.*

A laboratory sample with a mass of not less than 250 g is necessary to carry out the whole series of tests described in the three documents.

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a volumetric method for the determination of low contents of volatile acids other than formic acid in formic acid for industrial use.

The method, as described, is applicable to formic acid containing less than 0.5 % (m/m) of other acids, calculated as acetic acid.

NOTE. A method is described in ISO Recommendation R 731, applicable to formic acid containing between 0.5 and 6.0 % (m/m) of other acids, calculated as acetic acid. Consideration is to be given to a gas-liquid chromatographic method.

2. PRINCIPLE

Decomposition of most of the formic acid by concentrated sulphuric acid and of the remainder by chromic acid.

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

3. REAGENTS

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

3.1 *Sulphuric acid*, concentrated solution, ρ 1.84 (g/ml), approximately 96 % (m/m) or 36 N.

3.2 *Chromic acid* solution.
Dissolve 100 g of chromic trioxide (CrO_3) in 100 ml of water.

3.3 *Potassium hydroxide,* N solution.

3.4 *Sodium hydroxide,* 0.05 N standard volumetric solution.

3.5 *Phenolphthalein,* 1 g/l ethanolic solution.
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 opposite, with ground glass joints, consisting of the following items :

(A) *Two-necked round-bottomed flask,* capacity 250 ml.

(B) *Magnetic stirring bar,* totally enclosed in borosilicate glass or 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 device.*

(E) *Water-cooled reflux condenser.*

(F) *Bubbler tube.*

(G) *Dropping funnel,* capacity 100 ml.

(H) *Pressure regulator.*

The flask (A) is connected to the 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 to be 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 (F).

4.2 *Steam distillation apparatus,* with a distillation flask of capacity 1000 ml.

5. SAMPLING

Follow the principles given in ISO Recommendation R ...*. Additionally, place the laboratory sample in a clean, airtight, 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 the risk of contaminating the contents thereby in any way.

6. PROCEDURE

6.1 Test portion

Place in the dropping funnel (G) about 30 g of the laboratory sample weighed by difference to the nearest 0.1 g.

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* Under study.

Dimensions in millimetres

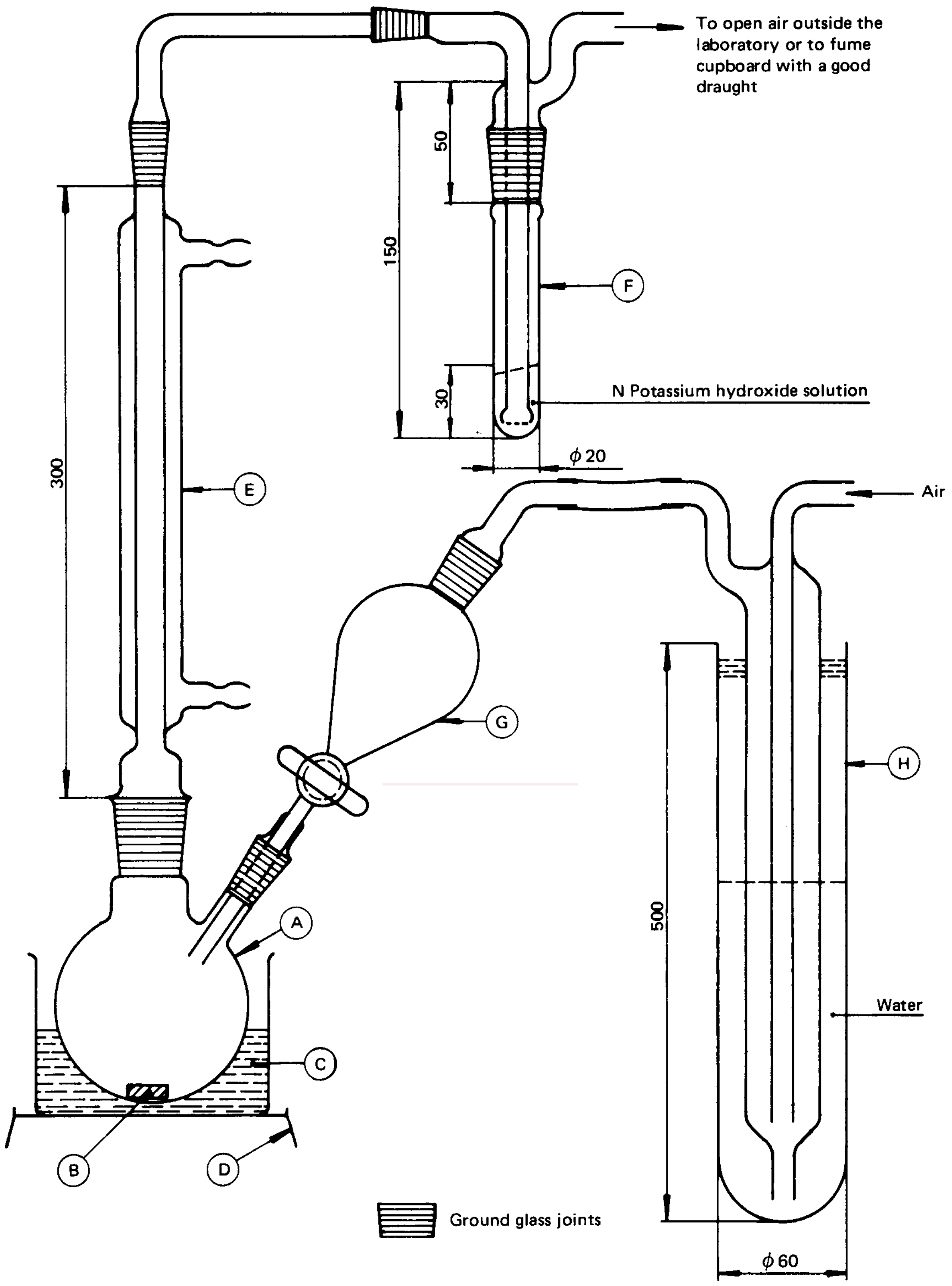

FIGURE – Apparatus for decomposition of formic acid (see clause 4.1)