INTERNATIONAL STANDARD 731/III

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

Formic acid for industrial use — Methods of test — Part III : Determination of content of other acids — Potentiometric method

Acide formique à usage industriel — Méthodes d'essai — Partie III : Dosage des autres acides — Méthode potentiométrique

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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 731-1968 and found it technically suitable for transformation. ISO/R 731 has, however, been subdivised into seven parts. International Standard ISO 731/III replaces clause 4 of ISO Recommendation R 731-1968, to which it is technically identical.

ISO Recommendation R 731 had been approved by the member bodies of the following countries :

Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Bulgaria	Israel	Spain
Chile	Italy	Switzerland
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
France	Netherlands	U.S.S.R.
Germany	New Zealand	Yugoslavia
Greece	Poland	
Hungary	Portugal	

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

U.S.A.

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 III : Determination of content of other acids — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 731 specifies a potentiometric method for the determination of the content of acids other than formic acid, in formic acid for industrial use.

The method is applicable to products containing 0,5 to 6,0 % (*m/m*) of other acids, expressed as acetic acid.

NOTE — The method specified in Part VII (see the annex) is applicable to formic acid containing less than 0,5 % (*m/m*) of other volatile acids, expressed as acetic acid.

This document should be read in conjunction with Part I (see the annex).

2 PRINCIPLE

Quantitative oxidation of the formic acid in a test portion with excess mercury(II) oxide. Potentiometric titration of the residual acids with standard volumetric sodium hydroxide solution.

3 REAGENTS

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

3.1 Mercury(II) oxide (HgO).

3.2 Acetic acid, approximately 0,5 % (*V/V*) solution.

Dilute 5 ml of glacial acetic acid, ρ approximately 1,05 g/ml, to 1 000 ml.

3.3 Sodium hydroxide, 0,1 N standard volumetric solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Two conical flasks, with ground glass necks, of capacity 250 ml.

4.2 Two water-cooled reflux condensers, with ground glass joints to fit the flasks (4.1).

4.3 pH meter, with glass measuring electrode and calomel reference electrode.

5 PROCEDURE

5.1 Test portion

5.1.1 *For expected content of acids other than formic acid less than 2* % (m/m)*, expressed as acetic acid*

Weigh, to the nearest 0,01 g, about 5 g of the laboratory sample.

5.1.2 *For expected content of acids other than formic acid equal to or greater than 2* % (m/m)*, expressed as acetic acid*

Weigh, to the nearest 0,005 g, about 2 g of the laboratory sample.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure but omitting the test portion.

5.3 Preparation of test solution

Place the test portion (5.1) in one of the conical flasks (4.1). Add exactly 5,0 ml of the acetic acid solution (3.2) and an amount of the mercury(II) oxide (3.1) calculated on the basis of 5,5 g of mercury(II) oxide per gram of formic acid present in the test portion. Then add sufficient water to bring the total volume to about 30 ml.

Fit the flask with one of the condensers (4.2) and heat gently for 10 min. Fairly strong evolution of carbon dioxide from the solution containing the test portion occurs. Reflux gently for 30 min and rinse the condenser with 20 to 25 ml of water. Cool the flask to ambient temperature and pour the contents, without filtering, into a 250 ml beaker. Rinse the flask into the beaker with 20 to 25 ml of water, and transfer the washings to the beaker.

5.4 Titration

Stir the solution vigorously, preferably using a magnetic stirrer, throughout the titration. Titrate the residual acids with the sodium hydroxide solution (3.3) using the pH meter (4.3). The end-point of the titration is at pH 8,6 for the determination and at pH 8,3 for the blank test (5.2).

Test portions requiring more than 10 to 15 ml of the sodium hydroxide solution do not give very sharp end-points and a period of vigorous stirring is necessary in order to obtain a stable final pH.