INTERNATIONAL STANDARD ISO 1390/III

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Maleic anhydride for industrial use — Methods of test — Part III : Determination of free acidity — Potentiometric method

Anhydride maléique à usage industriel — Méthodes d'essai — Partie III : Détermination de l'acidité libre — 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 1390-1970 and found it technically suitable for transformation. The technical committee, however, divided the recommendation into six parts (ISO 1390, parts I to VI), which therefore replace ISO Recommendation R 1390-1970, to which they are technically identical.

ISO Recommendation R 1390 had been approved by the member bodies of the following countries :

Austria	Iran	South Africa, Rep. of
Belgium	Ireland	Spain
Brazil	Italy	Sweden
Cuba	Korea, Rep. of	Switzerland
Czechoslovakia	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	United Kingdom
Hungary	Portugal	U.S.S.R.
India	Romania	

No member body had expressed disapproval of the Recommendation.

The member bodies of the following countries disapproved the transformation of the Recommendation into an International Standard :

France
Netherlands

Printed in Switzerland

Maleic anhydride for industrial use — Methods of test — Part III : Determination of free acidity — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

1.1 This part of ISO 1390 specifies a potentiometric method for the determination of the free acidity of maleic anhydride for industrial use.

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

1.2 The method is applicable to acids having a dissociation constant not less than 1×10^{-3}.

NOTE — Fumaric acid and other acids having a dissociation constant less than 1×10^{-3} are not determined by this method.

2 PRINCIPLE

Potentiometric titration of the free acidity in a test portion with a standard volumetric triethylamine solution in anhydrous methyl ethyl ketone (butanone).

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade.

3.1 Acetone.

3.2 Maleic acid $[(CHCOOH)_2]$.

3.3 Triethylamine $[(C_2H_5)_3N]$, free from primary and secondary amines, 0,1 N standard volumetric solution in methyl ethyl ketone ($CH_3CH_2COCH_3$) (butanone), previously standardized against the maleic acid (3.2) using the procedure specified in clause 5.

NOTE — Methyl ethyl ketone (butanone) of satisfactory quality may be obtained by treatment with anhydrous calcium chloride, decantation and distillation.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Microburette, of capacity 10 ml, graduated in 0,02 ml or smaller divisions.

4.2 pH meter, fitted with a glass measuring electrode and a calomel reference electrode.

The saturated aqueous solution of potassium chloride in the calomel electrode should be replaced by a saturated solution of potassium chloride in methanol. The calomel electrode should preferably be of the sleeve type with a ground glass joint.

4.3 Electromagnetic stirrer.

5 PROCEDURE

5.1 Weigh, to the nearest 0,01 g, a quantity of the test sample not exceeding 10 g and containing not more than 0,1 g of maleic acid. Transfer this test portion to a dry 150 ml beaker and dissolve in 75 ml of the acetone (3.1).

5.2 Place the glass and calomel electrodes in the solution, stir by means of the electromagnetic stirrer (4.3), cover the beaker to reduce evaporation and titrate potentiometrically with the triethylamine solution (3.3) from the microburette (4.1). On nearing the equivalence point, add the triethylamine solution in 0,02 ml portions, reading the corresponding potential each time.

5.3 If the test portion contains less than 0,006 g of maleic acid, the potential increments Δ_1, Δ_0, and Δ_2 will coincide with the large changes in potential at the start of the titration. Accordingly, if the volume of the triethylamine solution used is less than 0,5 ml, add at least 0,010 g of the maleic acid (3.2) and repeat the determination.

6 EXPRESSION OF RESULTS

6.1 Calculate the increments in potential corresponding to the addition of the triethylamine solution (3.3) in 0,02 ml portions. Let the three largest increments be Δ_1, Δ_0, and Δ_2, with Δ_0 being the largest increment, Δ_1 preceeding Δ_0, and Δ_2 following Δ_0.

Calculate the volume V_1, in millilitres, of the triethylamine solution using the formula

$$V_1 = V_0 + \frac{0{,}02 \times (\Delta_0 - \Delta_1)}{2\Delta_0 - (\Delta_1 + \Delta_2)}$$

where V_0 is the volume, in millilitres, of the triethylamine solution (3.3) added to reach the potential between Δ_0 and Δ_1.

NOTE – This calculation of the volume of the triethylamine solution is not exact. The differences between the theoretical values and the values obtained in this way are negligible, however, as only very small amounts of titrant are added when the equivalence point is approached. For simplicity, this method is preferred.

6.2 The free acidity A, expressed as a percentage by mass of maleic acid $[(CHCOOH)_2]$, is given by the formula

$$A = \frac{(11{,}6 \times V_1) - m_1}{m_0 \times 10}$$

where

m_0 is the mass, in grams, of the test portion (see 5.1);

m_1 is the mass, in milligrams, of the maleic acid (3.2) added (see 5.3);

V_1 is as defined in 6.1.

ANNEX

ISO PUBLICATIONS RELATING TO MALEIC ANHYDRIDE FOR INDUSTRIAL USE

ISO 1390/I – General.

ISO 1390/II – Measurement of colour of molten material.

ISO 1390/III – Determination of free acidity – Potentiometric method.

ISO 1390/IV – Determination of maleic anhydride content – Titrimetric method.

ISO 1390/V – Determination of ash.

ISO 1390/VI – Determination of iron content – 2,2′-Bipyridyl photometric method.