INTERNATIONAL STANDARD

ISO 1389/V

Phthalic anhydride for industrial use — Methods of test — Part V : Determination of free acidity — Potentiometric method

Anhydride phtalique à usage industriel — Méthodes d'essai —
Partie V : 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 1389-1970 and found it technically suitable for transformation. The technical committee, however, divided the recommendation into eleven parts (ISO 1389, parts I to XI), which therefore replace ISO Recommendation R 1389-1970, to which they are technically identical.

ISO Recommendation R 1389 had been approved by the member bodies of the following countries :

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

Phthalic anhydride for industrial use — Methods of test — Part V : Determination of free acidity — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1389 specifies a potentiometric method for the determination of the free acidity of phthalic anhydride for industrial use.

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

2 PRINCIPLE

Potentiometric titration of the free acidity in a test portion with a standard volumetric solution of triethylamine in anhydrous butanone.

3 REAGENTS

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

3.1 Acetone.

3.2 Phthalic acid $[1,2\text{-}(COOH)_2C_6H_4]$.

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

NOTE — 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.

Replace the saturated aqueous potassium chloride solution in the calomel electrode 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 0,150 g of phthalic 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 (4.2) 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 0,008 g of phthalic 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 phthalic 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 phthalic acid $[1,2\text{-}(COOH)_2C_6H_4]$, is given by the formula

$$A = \frac{(16,6 \times V_1) - m_1}{m_0 \times 10}$$

where

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

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

V_1 is as defined in 6.1.

ANNEX

ISO PUBLICATIONS RELATING TO PHTHALIC ANHYDRIDE FOR INDUSTRIAL USE

ISO 1389/I — General.

ISO 1389/II — Measurement of colour of molten material.

ISO 1389/III — Measurement of colour stability.

ISO 1389/IV — Measurement of colour after treatment with sulphuric acid.

ISO 1389/V — Determination of free acidity — Potentiometric method.

ISO 1389/VI — Determination of phthalic anhydride content — Titrimetric method.

ISO 1389/VII — Determination of maleic anhydride content — Polarographic method.

ISO 1389/VIII — Determination of ash.

ISO 1389/IX — Determination of impurities oxidizable in the cold by potassium permanganate — Iodometric method.

ISO 1389/X — Determination of 1,4-naphthaquinone content — Colorimetric method.

ISO 1389/XI — Determination of iron content — 2,2'-Bipyridyl photometric method.