INTERNATIONAL STANDARD 1389/IX

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Phthalic anhydride for industrial use — Methods of test — Part IX : Determination of impurities oxidizable in the cold by potassium permanganate — Iodometric method

Anhydride phtalique à usage industriel — Méthodes d'essai —
Partie IX : Détermination des matières oxydables à froid par le permanganate de potassium —
Méthode iodométrique

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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

Printed in Switzerland

Phthalic anhydride for industrial use — Methods of test — Part IX : Determination of impurities oxidizable in the cold by potassium permanganate — Iodometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1389 specifies an iodometric method for the determination of impurities oxidizable in the cold by potassium permanganate in phthalic anhydride for industrial use.

This method is not specific for maleic anhydride, for which the polarographic method given in part VII (see the annex) should be used.

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

2 PRINCIPLE

Oxidation of unsaturated organic acids and anhydrides in a test portion with an excess of cold standard volumetric potassium permanganate solution in the presence of sulphuric acid, followed by iodometric determination of the permanganate remaining.

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 Potassium iodide (KI).

3.3 Potassium permanganate, 0,1 N standard volumetric solution.

3.4 Sodium thiosulphate, 0,1 N standard volumetric solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Burette, of capacity 50 ml, graduated in 0,05 ml or smaller divisions.

5 PROCEDURE

Weigh, to the nearest 0,005 g, about 5 g of the test sample, add 100 ml of water and heat gently until the test portion is dissolved. Cool rapidly to room temperature. (Phthalic anhydride is completely soluble under these conditions when hot; a precipitate forms on cooling but this in no way interferes with the rest of the determination.)

After cooling, add 3 ml of the sulphuric acid solution (3.1), cool again to 20 ± 1 °C and add 25,0 ml of the potassium permanganate solution (3.3). Mix thoroughly and leave to stand at 20 ± 1 °C for 5 min. Add 1 g of the potassium iodide (3.2) and titrate the liberated iodine with the sodium thiosulphate solution (3.4) from the burette (4.1) (the colour change is very sensitive and does not require the addition of starch as indicator, except with coloured solutions).

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

6 EXPRESSION OF RESULTS

The content of oxidizable matter, expressed as a percentage by mass of maleic anhydride ($C_4H_2O_3$), is given by the formula

$$\frac{0{,}098 \times (V_0 - V_1)}{m}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used in the blank test;

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (3.4) used in the determination;

m is the mass, in grams, of the test portion.

NOTE — If the concentration of the standard volumetric solutions used is not exactly as specified in the list of reagents, an appropriate correction should be made.