# INTERNATIONAL STANDARD 1389/VII

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# Phthalic anhydride for industrial use — Methods of test — Part VII : Determination of maleic anhydride content — Polarographic method

*Anhydride phtalique à usage industriel — Méthodes d'essai — Partie VII : Dosage de l'anhydride maléique — Méthode polarographique*



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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 VII : Determination of maleic anhydride content — Polarographic method

## 1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1389 specifies a polarographic method for the determination of the maleic anhydride content of phthalic anhydride for industrial use.

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



## 2 PRINCIPLE

Dissolution of a test portion in acetone. Removal of 1,4-naphthaquinone present in the solution by extraction with benzene. Polarographic measurement of the aqueous solution.



## 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 Benzene.**

**3.3 Nitrogen,** free from oxygen.

**3.4 Hydrochloric acid,** approximately 0,2 N solution.

**3.5 Maleic anhydride,** standard solution in acetone, corresponding to 0,200 g of maleic anhydride per litre.

Weigh, to the nearest 0,000 1 g, 0,020 0 g of maleic anhydride ($C_4H_2O_3$), dissolve in the acetone (3.1), make up to volume in a 100 ml one-mark volumetric flask with the same acetone, and mix.

1 ml of this standard solution contains 0,200 mg of maleic anhydride.

**3.6 Maleic anhydride,** standard solution in acetone, corresponding to 0,020 g of maleic anhydride per litre.

Introduce 10,0 ml of the standard maleic anhydride solution in acetone (3.5) into a 100 ml one-mark volumetric flask, dilute to the mark with the acetone (3.1) and mix.

1 ml of this standard solution contains 0,020 mg of maleic anhydride.

Prepare this solution immediately prior to use.

## 4 APPARATUS

Ordinary laboratory apparatus and

**4.1 Polarograph.**

**4.2 Separating funnel,** of capacity 300 ml.

## 5 PROCEDURE

### 5.1 Preparation of the calibration graph

#### 5.1.1 *Preparation of standard matching solutions*

Into a series of seven 100 ml beakers, introduce the volumes of the standard maleic anhydride solution (3.5) shown in table 1.

TABLE 1

| Standard maleic anhydride solution (3.5) | Corresponding mass of maleic anhydride |
|---|---|
| ml | mg |
| 3,0 | 0,600 |
| 5,0 | 1,000 |
| 7,0 | 1,400 |
| 10,0 | 2,000 |
| 12,0 | 2,400 |
| 15,0 | 3,000 |
| 20,0 | 4,000 |

Into a second series of five 100 ml beakers, introduce the volumes of the standard maleic anhydride solution (3.6) shown in table 2.

TABLE 2

| Standard maleic anhydride solution (3.6) | Corresponding mass of maleic anhydride |
|---|---|
| ml | mg |
| 1,0 | 0,020 |
| 2,0 | 0,040 |
| 5,0 | 0,100 |
| 10,0 | 0,200 |
| 20,0 | 0,400 |

Treat the contents of each of the twelve beakers as follows :

Dilute to about 20 ml with the acetone (3.1), add 50 ml of the hydrochloric acid solution (3.4) and evaporate to about 25 ml on a boiling water bath.

After cooling, filter through a filter paper and wash with about 20 ml of water, collecting the filtrate and washings in a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

### 5.1.2 *Polarographic measurements*

Transfer to a polarographic cell and subject to a slow stream of the nitrogen (3.3) for 10 to 15 min to remove all oxygen. Record the polarogram of each of the standard matching solutions (5.1.1) at potentials from −0,4 to −0,8 V. Read the value of the average diffusion current for each standard matching solution.

### 5.1.3 *Plotting of the graph*

Plot a graph having, for example, the values, in milligrams, of the quantities of maleic anhydride contained in 100 ml of standard matching solutions (5.1.1) as abscissae, and the corresponding values of average diffusion current as ordinates.

## 5.2 Determination

### 5.2.1 *Test portion*

Weigh, to the nearest 0,001 g, about 0,5 g of the test sample into a 200 ml conical flask.

### 5.2.2 *Preparation of the test solution*

Add to the conical flask containing the test portion (5.2.1) 20 ml of the acetone (3.1). After dissolution of the test portion, add 50 ml of the hydrochloric acid solution (3.4) and evaporate to about 25 ml on a boiling water bath. bath (4.3).

After cooling, filter through a filter paper and wash with about 20 ml of water. Transfer the filtrate and the washings quantitatively to the separating funnel (4.2) and extract twice with 25 ml portions of the benzene (3.2) to remove 1,4-naphthaquinone. Transfer the aqueous solution to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

### 5.2.3 *Polarographic measurement*

Carry out the polarographic measurement of the test solution following the procedure specified in 5.1.2.

NOTE – The temperature and rate of fall of the mercury drops should be the same as used in preparing the calibration graph.



## 6 EXPRESSION OF RESULTS

By means of the calibration graph (5.1.3), determine the mass of maleic anhydride corresponding to the value of the average diffusion current for the test solution.

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

$$B = \frac{100\,m_1}{1\,000\,m_0} = \frac{m_1}{10\,m_0}$$

where

$m_0$ is the mass, in grams, of the test portion (5.2.1);

$m_1$ is the mass, in milligrams, of maleic anhydride found in the test solution (5.2.2).

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.

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