H-20-03

INTERNATIONAL STANDARD ISO 1390/VI

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

Maleic anhydride for industrial use — Methods of test — Part VI : Determination of iron content — 2,2′-Bipyridyl photometric method

Anhydride maléique à usage industriel — Méthodes d'essai — Partie VI : Dosage du fer — Méthode photométrique au bipyridyle-2,2′

First edition — 1977-06-01

UDC 661.73 : 620.1 : 543.8 **Ref. No. ISO 1390/VI-1977 (E)**

Descriptors : maleic anhydride, tests, chemical analysis, determination, colouring, solidification point, acidity, ash, iron.

Price based on 3 pages

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

1390 PT VI-77 4851903 0002695 7

Maleic anhydride for industrial use — Methods of test — Part VI : Determination of iron content — 2,2′-Bipyridyl photometric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1390 specifies a 2,2′-bipyridyl photometric method for the determination of the iron content of maleic anhydride for industrial use.

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

2 PRINCIPLE

Dissolution, in hot hydrochloric acid, of the residue from the determination of ash of a test portion (see part V). Reduction, by hydroxylammonium chloride, of the trivalent iron contained in the solution thus obtained. Formation of the coloured complex iron(II) -2,2′-bipyridyl in a buffered medium. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

3 REAGENTS

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

3.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

3.2 Nitric acid, approximately 4 N solution.

3.3 Urea (NH_2—CO—NH_2) solution.

Dissolve 100 g of urea in 100 ml of water.

3.4 Hydroxylammonium chloride (NH_2OH·HCl), 100 g/l solution.

3.5 Ammonium acetate (CH_3COONH_4), 500 g/l solution.

3.6 2,2′-Bipyridyl, 5 g/l hydrochloric solution.

Dissolve 0,5 g of 2,2′-bipyridyl in 100 ml of approximately 1 N hydrochloric acid solution.

3.7 Iron, standard solution corresponding to 0,100 g of Fe per litre.

Weigh, to the nearest 0,000 1 g, 0,702 2 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2 SO_4$·$FeSO_4$·$6H_2O$], dissolve in 50 ml of approximately 3 N sulphuric acid solution, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of Fe.

3.8 Iron, standard solution corresponding to 0,010 g of Fe per litre.

Place, in a 1 000 ml one-mark volumetric flask, 100 ml of the standard iron solution (3.7), dilute to the mark and mix.

1 ml of this standard solution contains 10 μg of Fe.

Prepare this solution immediately before use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer, fitted with filters giving a maximum transmission between 500 and 520 nm.

4.3 Stirrer of platinum wire.

5 PROCEDURE

5.1 Preparation of calibration graph

5.1.1 *Preparation of the standard colorimetric solutions* for photometric measurements carried out with cells of 4 or 5 cm optical path length

Into a series of seven 100 ml one-mark volumetric flasks, place the volumes of the standard iron solution (3.8) shown in the following table :

Standard iron solution (3.8)	**Corresponding mass of iron**
ml	μg
0*	0
2,0	20
4,0	40
7,0	70
10,0	100
15,0	150
20,0	200

* Blank test of reagents for calibration graph.

5.1.2 *Colour development*

Treat the contents of each flask as follows :

Add 20 ml of the nitric acid solution (3.2), 2 ml of the urea solution (3.3) and 2 ml of the hydroxylammonium chloride solution (3.4). Mix and allow to stand for 2 min. Then add 30 ml of the ammonium acetate solution (3.5) and 5 ml of the 2,2′-bipyridyl solution (3.6). Dilute to the mark and mix.

5.1.3 *Photometric measurements*

Using the spectrophotometer (4.1), at a wavelength of about 510 nm, or the photoelectric absorptiometer (4.2) fitted with suitable filters, carry out the photometric measurement of each standard colorimetric solution, after having adjusted the instrument to zero absorbance against the solution for the blank test of the reagents for the calibration graph.

5.1.4 *Plotting of the graph*

Plot a graph having, for example, as abscissae, the values, expressed in micrograms, of the quantities of iron (Fe) contained in 100 ml of standard colorimetric solution (5.1.1) and, as ordinates, the corresponding measured values of the absorbance.

5.2 Determination

5.2.1 *Preparation of test solution*

To the platinum or silica dish containing the residue from the determination of ash (see part V), add 5 ml of the hydrochloric acid solution (3.1). Heat the dish on a boiling water bath, stirring with the platinum stirrer (4.3) until complete dissolution of the residue. Allow to cool and transfer the solution quantitatively to a 100 ml one-mark volumetric flask.

5.2.2 *Colour development*

Carry out the colour development of the test solution (5.2.1) as specified in 5.1.2, but omitting the addition of 20 ml of the nitric acid solution (3.2).

5.2.3 *Photometric measurement*

After colour development of the test solution, carry out the photometric measurement as specified in 5.1.3, after having adjusted the instrument to zero absorbance against water.

NOTE – As an alternative to the measurement of absorbance, the test solution, prepared in accordance with 5.2.1 and 5.2.2, may be compared visually with a series of standard colorimetric solutions prepared under similar conditions, and its iron content deduced from this comparison.

6 EXPRESSION OF RESULTS

By reference to the calibration graph (5.1.4), determine the mass of iron corresponding to the absorbance of the test solution.

The iron content, expressed in milligrams of iron (Fe) per kilogram, is given by the formula

$$\frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion taken for the determination of ash (see part V);

m_1 is the mass, in micrograms, of iron found in the test solution (5.2.1).

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.