INTERNATIONAL STANDARD 2992

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

Ammonium sulphate for industrial use — Determination of iron content — 2,2′-bipyridyl photometric method

Sulfate d'ammonium à usage industriel — Dosage du fer — Méthode photométrique au 2,2′-bipyridyle

First edition — 1974-04-01

UDC 661.522 : 546.72 : 543.42

Ref. No. ISO 2992-1974 (E)

Descriptors : ammonium compounds, ammonium sulphate, chemical analysis, determination of content, iron, photometric analysis.

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.

International Standard ISO 2992 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in November 1972.

It has been approved by the Member Bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Israel	Sweden
Bulgaria	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Romania	U.S.S.R.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

Printed in Switzerland

Ammonium sulphate for industrial use — Determination of iron content — 2,2′-bipyridyl photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 2,2′-bipyridyl photometric method for the determination of the iron content of ammonium sulphate for industrial use. The method is applicable to iron (Fe) contents equal to or greater than 0,000 1 % (*m/m*).

2 PRINCIPLE

Reduction of trivalent iron by hydroxylammonium chloride. Formation of the complex between divalent iron and 2,2′-bipyridyl in a buffered medium (pH value between 4,5 and 6).

Photometric measurement of the coloured complex at a wavelength of about 522 nm.

3 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

3.1 Hydrochloric acid, approximately N solution.

3.2 Hydroxylammonium chloride, 100 g/l solution.

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in water, dilute to 100 ml and mix.

3.3 Ammonium acetate, 300 g/l solution.

Dissolve 30 g of ammonium acetate (CH_3COONH_4) in water, dilute to 100 ml and mix.

3.4 2,2′-bipyridyl, 10 g/l solution in hydrochloric acid.

Dissolve 1 g of 2,2′-bipyridyl in 10 ml of the hydrochloric acid solution (3.1), dilute to 100 ml and mix.

3.5 Standard iron solution corresponding to 2 g of Fe per litre.

Weigh, to the nearest 0,001 g, 7,030 g of ammonium iron(II) sulphate hexahydrate and place in a beaker of suitable capacity. Add 50 ml of a 100 g/l sulphuric acid solution and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 2 mg of Fe.

3.6 Standard iron solution corresponding to 0,2 g of Fe per litre.

Introduce 50,0 ml of the standard iron solution (3.5) into a 500 ml one-mark volumetric flask, add 5 ml of a 100 g/l sulphuric acid solution, dilute to the mark and mix.

1 ml of this standard solution contains 0,2 mg of Fe.

3.7 Standard iron solution corresponding to 0,01 g of Fe per litre.

Introduce 50,0 ml of the standard iron solution (3.6) into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

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

Prepare this solution immediately before it is required for use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer, fitted with appropriate filters.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,1 g, about 10 g of the test sample.

5.2 Blank test

Carry out, at the same time as the determination and following the same procedure, a blank test using the same quantities of all the reagents as used for the determination.

5.3 Preparation of calibration curve

5.3.1 *Preparation of the standard matching solutions,* relating to measurements carried out with an optical path length of 1 cm.

Into a series of eleven 100 ml one-mark volumetric flasks, introduce the volumes of the standard iron solution (3.7) shown in the following table.

Standard iron solution (3.7)	Corresponding mass of iron
ml	μg
0*	0
5,0	50
10,0	100
15,0	150
20,0	200
25,0	250
30,0	300
35,0	350
40,0	400
45,0	450
50,0	500

* Compensation solution

Add to each flask the quantity of water necessary to attain a volume of about 50 ml, then successively, mixing after each addition, 2 ml of the hydrochloric acid solution (3.1), 2 ml of the hydroxylammonium chloride solution (3.2) and, after 5 min, 5 ml of the ammonium acetate solution (3.3) and 1 ml of the 2,2′-bipyridyl solution (3.4). Dilute to the mark, mix and allow to stand for 10 min.

5.3.2 *Photometric measurements*

Carry out the photometric measurements using the spectrophotometer (4.1), at a wavelength of about 522 nm, or the photoelectric absorptiometer (4.2), fitted with appropriate filters, after having adjusted the instrument to zero absorbance against the compensation solution.

5.3.3 *Preparation of calibration chart*

Draw a curve having, for example, the numbers of micrograms of iron (Fe) contained in 100 ml of standard matching solutions as abscissae and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 *Preparation of the test solution*

Add 2 ml of the hydrochloric acid solution (3.2) to the test portion (5,1), dilute to the mark in a 50 ml one-mark volumetric flask and mix.

5.4.2 *Colour development*

According to the iron content, take an aliquot portion of the test solution (5.4.1) containing between 50 and 500 μg of Fe and introduce into a 100 ml one-mark volumetric flask. If necessary, dilute to about 50 ml and then add, successively, mixing after each addition, 2 ml of the hydrochloric acid solution (3.1), 2 ml of the hydroxylammonium chloride solution (3.2) and, after 5 min, 5 ml of the ammonium acetate solution (3.3) and 1 ml of the 2,2′-bipyridyl solution (3.4). Dilute to the mark, stir and allow to stand for 10 min.

5.4.3 *Photometric measurements*

Carry out the photometric measurements on the solution (5.4.2) and the blank test according to the procedure described in 5.3.2, after having adjusted the instrument to zero absorbance against water.

6 EXPRESSION OF RESULTS

By means of the calibration chart (see 5.3.3), determine the quantity of iron corresponding to the values of the photometric measurements.

The iron content, expressed as a percentage by mass of Fe, is given by the formula

$$\frac{(m_1 - m_2) \times D}{m_0 \times 10\,000}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in micrograms, of iron found in the aliquot portion of the test solution taken for the colour development;

m_2 is the mass, in micrograms, of iron found in a corresponding aliquot portion of the blank test solution;

D is the ratio between the volume of the test solution and the aliquot portion taken for the colour development.

7 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

ANNEX

This document forms part of the following series on methods of test for ammonium sulphate for industrial use :

ISO 2992 – *Determination of iron content – 2,2'-bipyridyl photometric method.*

ISO 2993 – *Determination of free acidity – Titrimetric method.*

ISO 2994 – *Determination of matter insoluble in water – Gravimetric method.*

ISO 3332 – *Determination of ammoniacal nitrogen content – Volumetric method after distillation.*[1)]

ISO 3333 – *Determination of copper content – Zinc dibenzyldithiocarbamate photometric method.*[1)]

1) At present at the stage of draft.

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