UDC 661.25 : 620.1

Ref. No. : ISO/R 915-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 915

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′—bipyridyl spectrophotometric method

1st EDITION
December 1968

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 915, *Sulphuric acid and oleum for industrial use – Determination of iron content – 2,2'–bipyridyl spectrophotometric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Based on detailed work on this question carried out by the Technical Committee, a Draft ISO Recommendation was adopted in 1965.

In June 1967, this Draft ISO Recommendation (No. 1185) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	India	Spain
Belgium	Iran	Switzerland
Brazil	Ireland	Thailand
Chile	Italy	Turkey
Cuba	Japan	U.A.R.
Czechoslovakia	Netherlands	United Kingdom
France	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia
Hungary	Romania	
ICAITI*	South Africa, Rep. of	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

* Instituto Centroamericano de Investigación y Tecnologia Industrial (Costa Rica, Guatemala, Honduras, Nicaragua, El Salvador, Panama).

ISO Recommendation R 915 December 1968

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′–bipyridyl spectrophotometric method

1. SCOPE

This ISO Recommendation describes a 2,2′–bipyridyl spectrophotometric method for the determination of the iron content of sulphuric acid and oleum for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of iron content, expressed as Fe greater than 1 p.p.m.

3. PRINCIPLE

Evaporation of the test portion, taking up by means of hydrochloric acid and reduction of iron (III) by means of hydroxylammonium chloride.

Formation of an iron (II) 2,2′–bipyridyl complex in a buffered medium (pH value between 4.5 and 6).

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

4. REAGENTS

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

4.1 *Hydrochloric acid*, approximately N solution.

4.2 *Hydroxylammonium chloride*, 100 g/l solution.

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

4.3 *Ammonium acetate*, 300 g/l solution.

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

4.4 *2,2′–bipyridyl*, 10 g/l hydrochloric acid solution.

Dissolve 1 g of 2,2′–bipyridyl in 10 ml of approximately N hydrochloric acid solution and dilute to 100 ml.

4.5 *Iron standard solution*, containing 2.00 g/l of Fe.

Weigh, to the nearest 1 mg, 7.022 g of iron (II) ammonium sulphate hexahydrate and place in a beaker of suitable capacity. Add 50 ml of 100 g/l sulphuric acid solution (about 2 N) and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 2.00 mg of Fe.

4.6 *Iron standard solution*, containing 0.20 g/l of Fe.

Transfer 50.0 ml of the iron standard solution (4.5) to a 500 ml one-mark volumetric flask, add 5 ml of 100 g/l sulphuric acid solution (about 2 N), dilute to the mark and mix thoroughly.

1 ml of this standard solution contains 0.20 mg of Fe.

The solution should be prepared just before use.

4.7 *Iron standard solution*, containing 0.010 g/l of Fe.

Transfer 50.0 ml of the iron standard solution (4.6) to a 1000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

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

The solution should be prepared just before use.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Weighing pipette*, capacity 60 ml approximately, with ground-glass stopper.

5.2 *Spectrophotometer*.

6. PROCEDURE

6.1 Test portion

Fill the weighing pipette (5.1) with the test sample and weigh by difference to the nearest 10 mg, a test portion of approximately 50 g. Transfer the test portion to a beaker of suitable capacity (250 ml, for example).

6.2 Blank test

At the same time as the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the test.

6.3 Preparation of calibrated curve

6.3.1 *Preparation of standard matching solutions* for the spectrophotometric measurement with 1 cm cell. Into each of a series of eleven 100 ml one-mark volumetric flasks, place the quantities of standard iron solution (4.7) indicated in the following Table :

Standard iron solution (4.7)	Corresponding to Fe
ml	µg
0[1)]	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

1) Compensation solution

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of the hydrochloric acid solution (4.1), 2 ml of the hydroxylammonium chloride solution (4.2) and after 5 minutes, 5 ml of the ammonium acetate solution (4.3) and 1 ml of the 2,2′–bipyridyl solution (4.4). Dilute to the mark, mix thoroughly and wait 10 minutes.

6.3.2 *Spectrophotometric measurement*. Carry out the measurement on the spectrophotometer (5.2) at a wavelength of about 522 nm, adjusting the instrument to zero optical density using as reference the compensation solution.

6.3.3 *Preparation of the calibration chart*. Prepare a calibration chart having for example the iron content in microgrammes per 100 ml of the standard matching solution as abscissae and the corresponding values of optical density as ordinates.

6.4 Determination

6.4.1 *Preparation of sample solution*. Place the beaker containing the test portion (6.1) on a sand bath and carefully evaporate to dryness.

Cool, take up with 2 ml of the hydrochloric acid solution (4.1), 25 ml of water and heat to facilitate the dissolution. Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and filter if necessary.

6.4.2 *Colour development*. Transfer an aliquot of the sample solution (6.4.1) containing between 50 and 500 µg of iron, to a 100 ml one-mark volumetric flask. Dilute to approximately 50 ml if necessary, then add successively 2 ml of the hydrochloric acid solution (4.1), 2 ml of the hydroxylammonium chloride solution (4.2) and, after 5 minutes, 5 ml of the ammonium acetate solution (4.3) and 1 ml of the 2,2'-bipyridyl solution (4.4). Dilute to the mark, mix and wait 10 minutes.

6.4.3 *Spectrophotometric measurement*. Carry out the spectrophotometric measurement according to the procedure of clause 6.3.2, adjusting the instrument to zero optical density using as reference the blank test solution (6.2).

7. EXPRESSION OF RESULTS

By reference to the calibration chart (see clause 6.3.3), determine the iron content corresponding to the spectrophotometric measurement.

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

$$\frac{A \times 100 \times 100}{V \times E}$$

where

A is the mass, in grammes, of iron determined in the aliquot of the sample solution,

V is the volume, in millilitres, of the sample solution taken for the colour reaction,

E is the mass, in grammes, of the test portion.