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UREA FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2'-BIPYRIDYL PHOTOMETRIC METHOD

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BRIEF HISTORY

The ISO Recommendation R 1595, *Urea for industrial use – Determination of iron content – 2,2' -bipyridyl photometric 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).

Work on this question led to the adoption of Draft ISO Recommendation No. 1595 which was circulated to all the ISO Member Bodies for enquiry in February 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Iran	Sweden
Brazil	Israel	Switzerland
Canada	Italy	Thailand
Chile	Netherlands	Turkey
Czechoslovakia	Peru	U.A.R.
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia
Greece	Romania	

The following Member Body opposed the approval of the Draft :

United Kingdom

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided to accept it as an ISO RECOMMENDATION.

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UREA FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′-BIPYRIDYL PHOTOMETRIC METHOD

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a 2,2′-bipyridyl photometric method for the determination of the iron (Fe) content of urea for industrial use, applicable to contents greater than or equal to 0.000 05 %.

2. PRINCIPLE

Ignition of the material at 800 °C and fusion of the residue with anhydrous potassium hydrogen sulphate. Solution in hydrochloric acid and reduction of trivalent iron by means of hydroxylammonium chloride.

Formation of a bivalent iron-2,2′-bipyridyl complex in the presence of a buffer solution (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 equal purity should be used in the test.

3.1 *Potassium hydrogen sulphate*, anhydrous.

3.2 *Hydrochloric acid*, approximately N solution.

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

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

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

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

3.5 *2,2′-bipyridyl*, 10 g/l in hydrochloric acid solution.

Dissolve 1 g of 2,2′-bipyridyl in 10 ml of the hydrochloric acid solution (3.2) and dilute to 100 ml.

3.6 *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 and transfer quantitatively to a 500 ml one-mark volumetric flask.

Dilute to the mark and mix.

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

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

Transfer 50.0 ml of the iron standard solution (3.6) to a 500 ml one-mark volumetric flask, add 5 ml of 100 g/l sulphuric acid solution and dilute to the mark.

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

The solution should be prepared just before use.

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

Transfer 50.0 ml of the iron standard solution (3.7) to a 1000 ml one-mark volumetric flask and dilute to the mark.

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

The solution should be prepared just before use.

4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Platinum dish*, flat-bottomed, about 50 mm in diameter and 25 mm high.

4.2 *Spectrophotometer*, or

4.3 *Photoelectric absorptiometer.*

5. PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.1 g, about 100 g of the test sample*.

5.2 Blank test

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

5.3 Preparation of calibration curve

5.3.1 *Preparation of the standard colorimetric solutions* for photometric measurements with a 1 cm cell. Into each of a series of eleven 100 ml one-mark volumetric flasks, place respectively the quantities of standard iron solution (3.8) indicated in the following table :

Volume of standard iron solution (3.8)	Corresponding mass of iron (Fe)
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 volumetric flask an amount of water sufficient to bring the volume to approximately 50 ml, then add 2 ml of hydrochloric acid solution (3.2) and 2 ml of the hydroxylammonium chloride solution (3.3), stirring after each addition. Allow to stand for 5 minutes, then add 5 ml of the ammonium acetate solution (3.4) and 1 ml of the 2,2′-bipyridyl solution (3.5). Dilute to the mark, mix thoroughly and allow to stand for 10 minutes.

* For this determination, the residue from the ash determination may be used (See ISO Recommendation R 1594, *Urea for industrial use – Determination of ash – Gravimetric method*).

5.3.2 *Photometric measurement*. Carry out the photometric measurement with the spectrophotometer (4.2) at a wavelength of about 522 nm, or with the photoelectric absorptiometer (4.3) with a suitable filter, adjusting the instrument to zero absorbance against the compensation solution .

5.3.3 *Preparation of the calibration chart*. Prepare a calibration chart having, for example, the iron (Fe) content in micrograms per 100 ml of the standard colorimetric solution as abscissae and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 *Preparation of sample solution*. Heat the dish (4.1) over a small flame and place a little of the test portion in it. When it has melted, add the remainder of the test portion in small amounts, waiting after each addition until all the product has melted.

When a solid grey mass has been obtained, transfer the dish containing the material to an electric furnace controlled at about 300 °C and steadily raise the temperature to about 800 °C and continue heating until the residue is completely ignited.

Remove the dish from the furnace and allow to cool. Add to the dish 1 ± 0.01 g of potassium hydrogen sulphate (3.1) and melt over a flame. Keep in the molten state for 10 minutes and then allow to cool. Dissolve the melt in 2 ml of the hydrochloric acid solution (3.2) and about 10 ml of water, heating gently to dissolve. Transfer quantitatively to a 50 ml one-mark volumetric flask (filtering if necessary), dilute to the mark and mix.

5.4.2 *Colour development*. Transfer to a 100 ml one-mark volumetric flask an aliquot portion of the sample solution (5.4.1) containing between 50 and 500 μg of Fe.

If necessary, dilute to about 50 ml and successively add, mixing after each addition, 2 ml of the hydrochloric acid solution (3.2) and 2 ml of the hydroxylammonium chloride solution (3.3). Allow to stand for 5 minutes, then add 5 ml of the ammonium acetate solution (3.4) and 1 ml of the 2,2′ -bipyridyl solution (3.5). Dilute to the mark, mix and allow to stand for 10 minutes.

5.4.3 *Photometric measurement*. Carry out the photometric measurements on both the sample and blank solutions, following the same procedure as indicated in clause 5.3.2, after adjusting the instrument to zero absorbance against water.

6. EXPRESSION OF RESULTS

By means of the calibration chart (see clause 5.3.3) determine the amount of iron corresponding to the photometric measurements. The iron (Fe) content is given, as a percentage by mass, by the following formula :

$$\frac{(m_1 - m_2) \times D \times 100}{m_0}$$

where

m_1 is the mass, in grammes, of iron found in the aliquot portion of the sample solution;

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

D is the ratio of the volume of the sample solution to that of the aliquot portion taken for the colour development;

m_0 is the mass, in grammes, of the test portion.

7. TEST REPORT

Give 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 ISO Recommendation or regarded as optional.

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