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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 983

SODIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2'-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

1st EDITION
February 1969

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BRIEF HISTORY

The ISO Recommendation R 983, *Sodium hydroxide 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).

Work on this question led, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1092) 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	Ireland	Spain
Belgium	Israel	Switzerland
Chile	Italy	Thailand
Cuba	Japan	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	Portugal	Yugoslavia
India	Romania	
Iran	South Africa, Rep. of	

One Member Body opposed the approval of the Draft :

U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

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ISO Recommendation R 983 February 1969

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DETERMINATION OF IRON CONTENT

2,2'-BIPYRIDYL SPECTROPHOTOMETRIC METHOD

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1. SCOPE

This ISO Recommendation describes the 2,2'-bipyridyl spectrophotometric method for the determination of the iron content of sodium hydroxide for industrial use.

2. PRINCIPLE

Preliminary reduction of trivalent iron by means of hydroxylammonium chloride. Formation of a bivalent iron-2,2'-bipyridyl complex in the presence of ammonium acetate.
Spectrophotometric measurement of the coloured complex at a wavelength of about 522 nm.

3. REAGENTS

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

3.1 *Hydrochloric acid,* approximately $d = 1.19$, 38 % (m/m) or 12 N solution.

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,* 200 g/l solution.
Ammonium acetate used for the solution should not have an iron content higher than 1 part per million.

3.5 *2,2'-bipyridyl,* 1 g/l hydrochloric acid solution.
Dissolve 1 g of 2,2'-bipyridyl, weighed to the nearest 0.2 mg, in 20 ml of the hydrochloric acid solution (3.2) and dilute to 1000 ml.

3.6 *Iron standard solution,* containing 2.000 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, then 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.

3.7 *Iron standard solution,* containing 0.200 g/l of Fe.

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

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

Transfer 50.0 ml of the standard solution (3.7) 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.

3.9 *Iron standard solution,* containing 0.0020 g/l of Fe.

Transfer 50.0 ml of the standard solution (3.8) to a 250 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

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

The solution should be prepared just before use.

3.10 *Litmus paper.*

4. APPARATUS

4.1 *Ordinary laboratory apparatus.*

4.2 *Spectrophotometer,* or

4.3 *Photoelectric absorptiometer.*

5. PROCEDURE

5.1 Test portion

In a weighing bottle of approximately 150 ml capacity, fitted with a ground glass stopper, weigh to the nearest 0.1 g a mass of the test sample (solid or liquid)* containing between 35 and 40 g of NaOH.

5.2 Blank test

Place a volume of the hydrochloric acid solution (3.1) equal to that used for the neutralization of the test portion in the preparation of the sample solution (see clause 5.4.1) in a silica dish of suitable capacity (100 ml, for example). Evaporate to dryness on a boiling water bath under a hood. Take up the residue with approximately 2 ml of the hydrochloric acid solution (3.1) and approximately 50 ml of water. Again evaporate to dryness on a boiling water bath. Take up again with approximately 2 ml of the hydrochloric acid solution (3.1) and 50 ml of water. Transfer quantitatively to a 250 ml one-mark volumetric flask. Allow to cool, dilute to the mark and mix thoroughly.

Then carry out the development of the colour reaction following the procedure described in clause 5.4.2.

* See ISO Recommendation R 977, *Sodium hydroxide for industrial use – Preparation and storage of test sample,* clause 2.2.

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5.3 Preparation of calibration curve

5.3.1 *Preparation of the standard matching solutions* for spectrophotometric measurement with a 4 cm cell.

5.3.1.1 CASE OF MEDIUM IRON CONTENTS. Into each of a series of eleven 100 ml one-mark volumetric flasks, place respectively the volumes of standard iron solution (3.8) indicated in the following table :

Volume of standard iron solution (3.8)	Corresponding mass of iron
ml	µg
0 *	0
1.00	10
2.00	20
3.00	30
4.00	40
5.00	50
6.00	60
7.00	70
8.00	80
9.00	90
10.00	100

* Compensating solution

5.3.1.2 CASE OF LOW IRON CONTENTS. Into each of a series of eleven 100 ml one-mark volumetric flasks, place respectively the volumes of standard iron solution (3.9) indicated in the following table :

Volume of standard iron solution (3.9)	Corresponding mass of iron
ml	µg
0 *	0
1.00	2
2.00	4
3.00	6
4.00	8
5.00	10
6.00	12
7.00	14
8.00	16
9.00	18
10.00	20

* Compensating solution

Add to each volumetric flask an amount of water sufficient to reach approximately 50 ml, and then 2.0 ml of hydrochloric acid solution (3.2) and 2.0 ml of the hydroxylammonium chloride solution (3.3), stirring after each addition; after leaving to stand for 1 minute, add 10.0 ml of the ammonium acetate solution (3.4) and 2.0 ml of the 2,2'-bipyridyl solution (3.5). Dilute to the mark, mix thoroughly and wait 10 minutes.

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5.3.2 *Spectrophotometric measurement.* Carry out the spectrophotometric measurement using either the spectrophotometer (4.2) at a wavelength of about 522 nm, or the photoelectric absorptiometer (4.3) with a suitable filter, adjusting the instrument to zero optical density against the compensating solution.

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

5.4 Determination

5.4.1 *Preparation of sample solution.* Place the test portion (5.1) in a beaker of suitable capacity (600 ml for example). If the product is solid, dissolve in approximately 150 ml of water, stirring to obtain a solution. If the product is liquid, dilute to approximately 150 ml.

Then neutralize the solution with the hydrochloric acid solution (3.1), added carefully in the presence of the litmus paper (3.10) as indicator. Then add a 2 ml excess of the hydrochloric acid solution (3.1). Boil for a few minutes. Cool to room temperature, then transfer quantitatively to a 250 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

5.4.2 *Colour development.* Transfer 25.0 ml of the sample solution (see clause 5.4.1) to a 100 ml one-mark volumetric flask and 25.0 ml of the blank test solution (see clause 5.2) to a similar flask. Dilute to approximately 50 ml, add 2.0 ml of the hydroxylammonium chloride solution (3.3) to each flask, allow to stand for 1 minute, and then add 10.0 ml of the ammonium acetate solution (3.4) and 2.0 ml of the 2,2'-bipyridyl solution (3.5). Mix thoroughly after each addition. Dilute to the mark, again mix thoroughly and wait 10 minutes.

5.4.3 *Spectrophotometric measurement.* Carry out the spectrophotometric measurement following the same procedure as described in clause 5.3.2, adjusting the instrument to zero optical density using as reference the blank test solution.

6. EXPRESSION OF RESULTS

By reference to the calibration chart (see clause 5.3.3), read the iron content corresponding to the spectrophotometric measurement.

The iron content, expressed as iron (III) oxide (Fe_2O_3), is given as a percentage, by mass, by the following formula :

$$\frac{A \times 250 \times 100}{25 \times E} \times 1.4297 = 1429.7 \times \frac{A}{E}$$

where

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

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

1.4297 is the conversion factor for Fe to Fe_2O_3.

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