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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 744

SODIUM CARBONATE FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′ – BIPYRIDYL PHOTOMETRIC METHOD

1st EDITION
May 1968

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 744, *Sodium carbonate 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 by the Technical Committee, began in 1951 and led, in 1956, to the adoption of a Draft ISO Recommendation.

In June 1966, this Draft ISO Recommendation (No. 1010) 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 :

Argentina
Austria
Belgium
Brazil
Chile
Czechoslovakia
France
Germany
Hungary
India
Israel
Italy
Japan
Korea, Rep. of
Netherlands
New Zealand
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Switzerland
Turkey
U.A.R.
United Kingdom
U.S.A.
U.S.S.R.
Yugoslavia

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 May 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 744 May 1968

SODIUM CARBONATE FOR INDUSTRIAL USE

DETERMINATION OF IRON CONTENT

2,2′ – BIPYRIDYL PHOTOMETRIC METHOD

1. SCOPE

This ISO Recommendation describes the 2,2′-bipyridyl photometric method for the determination of the iron content of sodium carbonate 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.

Photometric 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*, $d = 1.8$ approximately, 37 % (m/m) or approximately 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 have an iron content not higher than 1 ppm.

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 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, transfer to a 500 ml one-mark volumetric flask and wash. Dilute to the mark. 1 ml of this standard solution contains 2 mg of Fe.

3.7 *Iron standard solution containing 0.2 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 0.2 mg of Fe. The solution should be prepared just before use.

3.8 *Iron standard solution containing 0.01 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.

3.9 *Iron standard solution containing 0.002 g/l of Fe.* Transfer 50.0 ml of the iron standard solution (3.8) to a 250 ml one-mark volumetric flask and dilute to the mark. 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

Ordinary laboratory apparatus.

4.1 *Spectrophotometer,*

or

4.2 *Photoelectric absorptiometer.*

5. PROCEDURE

5.1 Test portion

Weigh to the nearest 0.1 g, 50 ± 1 g of the test sample.*

5.2 Blank test

Place a volume of the hydrochloric acid solution (3.1) equal to that used for the neutralization 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 water bath under a hood. Take up the residue with 2 ml of hydrochloric acid solution (3.1) and 50 ml of water. Boil for a few minutes. Allow to cool to room temperature, transfer to a 250 ml one-mark volumetric flask and wash. Dilute to the mark and mix thoroughly. Then perform the development of the colour reaction following the same procedure as described in clause 5.4.2.

5.3 Preparation of calibration curve

5.3.1 *Preparation of the standard matching solutions* for photometric measurements with a 40 mm 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 quantities of standard iron solution (3.8) indicated in the following Table :

Standard iron solution (3.8)	Corresponding to
ml	μg of iron
0 (1)	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

(1) Compensation solution

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* See clause 2.2 of ISO Recommendation R 739, *Sodium carbonate for industrial use – Preparation and storage of test samples.*