INTERNATIONAL STANDARD 994

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Potassium hydroxide for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

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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 994 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in September 1971.

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

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 994-1969, *Potassium hydroxide for industrial use — Determination of iron content — 2,2'-bipyridyl spectrophotometric method.*

Printed in Switzerland

Potassium hydroxide for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

1 SCOPE

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of the iron content of potassium hydroxide for industrial use.

This method is more sensitive and more widely used than the 2,2'-bipyridyl method which was specified in ISO/R 994.

2 FIELD OF APPLICATION

The method is applicable to products having iron contents equal to or greater than 0,3 mg/kg.

3 PRINCIPLE

Reduction of the trivalent iron by hydroxylammonium chloride, followed by the formation of a bivalent iron/1,10-phenanthroline complex in a buffered system. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

4 REAGENTS

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

4.2 Ammonia solution, ρ approximately 0,91 g/ml, about 25 % (*m/m*) NH_3 solution or approximately 13 N, with a maximum iron content of 0,2 mg/kg.

4.3 Hydroxylammonium chloride ($NH_2OH.HCl$), 10 g/l solution.

4.4 Buffer solution, pH 4,9.

Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in about 500 ml of water. Add 240 ml of glacial acetic acid (ρ approximately 1,05 g/ml, 99 to 100 % (*m/m*) solution or approximately 17,4 N) to the solution and dilute to 1 000 ml.

4.5 Bromine water, saturated at room temperature.

4.6 1,10-Phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$), 2,5 g/l solution.

This product may be replaced by 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$).

4.7 Iron standard solution, corresponding to 0,200 g of Fe per litre.

Dissolve 1,404 3 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$], weighed to the nearest 0,0001 g, in 200 ml of water. Add 20 ml of sulphuric acid, ρ approximately 1,84 g/ml, cool to room temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

4.8 Iron standard solution, corresponding to 0,010 g of Fe per litre.

Transfer 25,0 ml of the iron standard solution (4.7) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 0,010 mg of Fe.

4.9 Methyl orange, 0,5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, or

5.2 Photoelectric absorptiometer, fitted with filters giving maximum transmission between 500 and 520 nm.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,1 g, in a weighing bottle fitted with a ground glass lid, a mass of test sample[1] (solid or liquid) containing between 35 and 40 g of KOH.

1) See ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

6.2 Blank test

Pour 25 ml of water and a volume of the hydrochloric acid solution (4.1) identical to that used to neutralize the test portion (see 6.4.1) into a 600 ml beaker. Add 40 ml of the ammonia solution (4.2), 5 drops of the methyl orange solution (4.9) and then neutralize with the ammonia solution (4.2). Add the hydrochloric acid solution (4.1) drop by drop until the colour changes to red, and then an excess of 2 ml of this acid. Add 5 ml of the bromine water (4.5) to remove the colour of the indicator, boil for 5 min, cool to room temperature, transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix. Proceed as described in 6.4.2.

6.3 Preparation of the calibration curve

6.3.1 *Preparation of the standard matching solution,* for photometric measurements with a 5 cm cell.

Into a series of five 100 ml one-mark volumetric flasks, transfer the quantities of the iron standard solution (4.8) indicated in the following table :

Iron standard solution (4.8)	Corresponding mass of Fe
ml	mg
0*	0
2,5	0,025
5,0	0,050
10,0	0,100
15,0	0,150

* Compensation solution

Add to each flask 0,5 ml of the hydrochloric acid solution (4.1) and the amount of water necessary to make up the volume to about 50 ml. Then add 5 ml of the hydroxylammonium chloride solution (4.3), 5 ml of the 1,10-phenanthroline hydrochloride solution (4.6) and 25 ml of the buffer solution (4.4).

Dilute to the mark, mix and wait for 10 min.

6.3.2 *Photometric measurements*

Carry out the photometric measurements with the spectrophotometer (5.1) at a wavelength of about 510 nm, or with the photoelectric absorptiometer (5.2), fitted with suitable filters, after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Plotting the calibration curve*

Prepare a chart having, for example, the iron (Fe) contents in milligrams per 100 ml of standard matching solution as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 1 000 ml beaker. Add 120 ml of water and neutralize cautiously with the hydrochloric acid solution (4.1) in the presence of 5 drops of the methyl orange solution (4.9). Add an excess of 2 ml of this acid, then 5 ml of the bromine water (4.5). Boil for 5 min, cool to room temperature, transfer quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

6.4.2 *Colour development*

Transfer 50,0 ml of the test solution (6.4.1) to a 100 ml one-mark volumetric flask. Add 5 ml of the hydroxylammonium chloride solution (4.3), 5 ml of the 1,10-phenanthroline hydrochloride solution (4.6) and 25 ml of the buffer solution (4.4). Dilute to the mark, mix and wait for 10 min.

6.4.3 *Photometric measurement*

Measure the absorbance of the solution (6.4.2) as described in 6.3.2, after having adjusted the instrument to zero absorbance against the blank test solution (6.2).

7 EXPRESSION OF RESULTS

By reference to the calibration curve (6.3), determine the quantity of Fe corresponding to the value of the absorbance measured.

The iron content (Fe) is given, in milligrams per kilogram, by the formula

$$m_1 \times \frac{250}{50} \times \frac{1\,000}{m_0} = \frac{5\,000\,m_1}{m_0}$$

where

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

m_1 is the mass, in milligrams, of Fe found in the aliquot portion of the test solution.

8 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 those to which reference is made, or regarded as optional.