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Sodium and potassium silicates for industrial use — Determination of iron content — 1,10-Phenanthroline photometric method

Silicates de sodium et de potassium à usage industriel — Dosage du fer — Méthode photométrique à la 1,10-phénanthroline

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FOREWORD

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

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No Member Body expressed disapproval of the document.

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Sodium and potassium silicates 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 iron content of sodium and potassium silicates for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having iron contents greater than 2 mg/kg.

3 PRINCIPLE

Prior reduction of the trivalent iron by hydroxylammonium chloride. Formation of the divalent iron/1,10-phenanthroline complex in a buffered medium. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, approximately 2 N solution.

4.2 Hydroxylammonium chloride ($NH_2OH.HCl$), 100 g/l solution.

4.3 Buffer solution, pH 4,9.

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

4.4 Bromine water, saturated at room temperature.

4.5 1,10-Phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$), 2,5 g/l solution.

This reagent may be replaced by 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$), 2,5 g/l solution.

4.6 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 0,000 1 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.

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

Transfer 25,0 ml of the standard iron solution (4.6) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

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

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

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Spectrophotometer or

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

5.3 Platinum crucible, with lid. Upper diameter about 30 mm, height about 30 mm.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, in a weighing bottle fitted with a ground glass closure, a quantity of the test sample corresponding to approximately 5 g of the anhydrous product.

6.2 Blank test

Pour 25 ml of water, a volume of the hydrochloric acid solution (4.1) 15 ml in excess of that used to neutralize the test portion (see 6.4.1), 5 drops of the methyl orange solution (4.8) and 5 ml of the bromine water (4.4) (to decolorize the indicator) into a 600 ml beaker. Boil for 5 min, cool to room temperature and transfer

quantitatively to a 250 ml one-mark volumetric flask. Dilute to the mark, mix, and then proceed as specified in 6.4.2.

6.3 Preparation of the calibration curve

6.3.1 *Preparation of the standard colorimetric solutions*, relating to photometric measurements carried out with cells of 4 cm or 5 cm optical path length.

Into a series of five 100 ml one-mark volumetric flasks, transfer the quantities of the standard iron solution (4.7) indicated in the following table :

Standard iron solution (4.7)	Corresponding mass of Fe
ml	mg
0*	–
2,5	0,025
5,0	0,050
10,0	0,100
15,0	0,150

* Compensation solution.

Add 5 ml of the hydrochloric acid solution (4.1) and the amount of water required to dilute to about 50 ml to each flask. Then add 5 ml of the hydroxylammonium chloride solution (4.2), 5 ml of the 1,10-phenanthroline hydrochloride solution (4.5) and 25 ml of the buffer solution (4.3). Dilute to the marks, 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, in each case, adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the numbers of milligrams of iron (Fe) contained in 100 ml of the standard colorimetric solutions on the abscissa and the corresponding values of absorbance on the ordinate.

6.4 Determination

6.4.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 600 ml beaker. Add 175 ml of water and neutralize cautiously with the hydrochloric acid solution (4.1) in the presence of 5 drops of the methyl orange solution (4.8). Add an excess of 15 ml of this acid and then add 5 ml of the bromine water (4.4). Boil for 5 min, cool to room temperature, transfer quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

NOTE – If the resultant solution is turbid, discard it and weigh, to the nearest 0,01 g, a new test portion, corresponding to 1 g of the anhydrous product. Place it in a platinum crucible, previously cleaned by heating in a furnace and rinsing with hydrochloric acid and with water. Add 5 g of potassium sodium carbonate and heat slowly until the mix is fused.

After cooling, dissolve in 100 ml of hot water, added in portions, and transfer quantitatively to a 600 ml beaker. Dilute to 150 ml and proceed according to the instructions given in 6.4.1 from "neutralize cautiously". Adjust the blank test (6.2) by also adding 5 g of potassium sodium carbonate.

6.4.2 *Colour development*

Transfer not more than 50,0 ml of the test solution (6.4.1), containing less than 0,150 mg of iron, to a 100 ml one-mark volumetric flask. Add 5 ml of the hydroxylammonium chloride solution (4.2), 5 ml of the 1,10-phenanthroline hydrochloride solution (4.5) and 25 ml of the buffer solution (4.3). Dilute to the mark, mix and wait 10 min.

6.4.3 *Photometric measurement*

Carry out the photometric measurement on the solution (6.4.2) as specified in 6.3.2, after having adjusted the instrument to zero absorbance against the blank test solution (6.2).

7 EXPRESSION OF RESULTS

By means of the calibration chart (6.3.3), determine the quantity of Fe corresponding to the value of the absorbance measured.

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

$$m_1 \times \frac{250}{V} \times \frac{1\,000}{m_0} = \frac{250\,000\,m_1}{Vm_0}$$

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;

V is the volume, in millilitres, of test solution (6.4.1) taken for the determination.

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 regarded as optional.