INTERNATIONAL STANDARD ISO 5443

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Sodium hexafluorosilicate for industrial use — Determination of iron content — 1,10-Phenanthroline spectrophotometric method

Hexafluorosilicate de sodium à usage industriel — Dosage du fer — Méthode spectrophotométrique à la phénanthroline-1,10

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FOREWORD

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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 5443 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1976.

It has been approved by the member bodies of the following countries :

Belgium
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Romania
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Switzerland
Thailand
Turkey
United Kingdom
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Yugoslavia

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Sodium hexafluorosilicate for industrial use — Determination of iron content — 1,10-Phenanthroline spectrophotometric method

WARNING — Sodium hexafluorosilicate is poisonous if taken internally. Breathing of the dust should be avoided. Contact with eyes and skin should be prevented and operators should wash thoroughly after handling the material and should wear a respirator and goggles when handling the powdered material.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 1,10-phenanthroline spectrophotometric method for the determination of the iron content of sodium hexafluorosilicate for industrial use.

The method is applicable to products having iron contents, expressed as Fe, between 0,000 4 and 0,1 % (*m*/*m*).

2 REFERENCE

ISO 5444, *Sodium hexafluorosilicate for industrial use — Determination of loss in mass at* 105 °C.

3 TEST SAMPLE

Use the residue from the determination of the loss in mass at 105 °C (see ISO 5444) to prepare the test sample.

4 PRINCIPLE

Decomposition of the hexafluorosilicate of a test portion and elimination of silica by evaporation with hydrofluoric and sulphuric acids. Dissolution of the residue in hydrochloric acid and reduction of the trivalent iron with hydroxylammonium chloride. Formation of a complex between iron(II) and 1,10-phenanthroline in a buffered medium and spectrophotometric measurement of the coloured complex at a wavelength of about 510 nm.

5 REAGENTS

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

5.1 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m*/*m*) solution.

5.2 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m*/*m*) solution.

5.3 Hydrofluoric acid, ρ approximately 1,13 g/ml, about 40 % (*m*/*m*) solution.

5.4 Hydrochloric acid, approximately 4 N solution.

5.5 Ammonium hydroxide, approximately 4 N solution.

5.6 Ammonium acetate, 200 g/l solution.

Dissolve 200 g of ammonium acetate trihydrate ($CH_3COONH_4.3H_2O$) in water and dilute to about 900 ml. Add the quantity of the hydrochloric acid solution (5.4), or the ammonium hydroxide solution (5.5), necessary to bring the pH of the solution, after dilution (1 + 10), to between 5,8 and 6,0. Dilute to 1 000 ml and mix.

5.7 Sodium citrate, dihydrate [$NaCOO\text{-}CH_2\ C(OH)(COONa)\text{-}CH_2\text{-}COONa\text{-}2H_2O$], 100 g/l solution.

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

5.9 1,10-Phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$), 1 g/l solution.

This reagent may be replaced by the corresponding quantity of 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$).

Store the solution away from light. Use only colourless solutions.

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

Dissolve 1,404 3 g, weighed to the nearest 0,000 1 g, of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2SO_4.FeSO_4.6H_2O$] in 200 ml of water. Add 20 ml of the sulphuric acid solution (5.1), cool to ambient temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

1 ml of this standard solution corresponds to 0,200 mg of Fe.

5.11 Iron, standard solution, corresponding to 0,020 g of Fe per litre.

Transfer 50,0 ml of the standard iron solution (5.10) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution corresponds to 20 µg of Fe.

Prepare this solution at the time of use.

5.12 Phenolphthalein, 10 g/l solution in 95 % (*V*/*V*) ethanol.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Platinum crucible, of capacity 30 ml.

6.2 Spectrophotometer, fitted with cells of optical path length 1, 2, 4 or 5 cm.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 5 ± 0,1 g of the test sample dried at 105 °C (see clause 3) into the platinum crucible (6.1).

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used in the determination, but omitting the test portion.

7.3 Preparation of the test solution

Add 10 ml of the hydrofluoric acid solution (5.3) and 1 ml of the sulphuric acid solution (5.1) to the crucible containing the test portion (7.1). Heat the crucible and contents on a hot-plate in a fume cupboard, avoiding excessive heating and ensuring that the mixture does not boil. When no more hydrofluoric acid is evolved, allow to cool and add 5 ml of the hydrofluoric acid solution. Heat as before to expel hydrofluoric acid and finally heat more strongly until copious white fumes are expelled. Allow to cool and add 2 ml of the hydrochloric acid solution (5.2). Warm to dissolve the residue and transfer the solution quantitatively to a 100 ml one-mark volumetric flask, if necessary filtering, washing the filter with water and collecting the washings in the flask. Dilute to the mark and mix.

7.4 Preparation of the calibration graph

7.4.1 *Preparation of standard colorimetric solutions* for spectrophotometric measurements with cells of optical path length 1, 2, 4 or 5 cm

According to the expected content, place the volumes of the standard iron solution (5.11) shown in the following table in a series of 100 ml one-mark volumetric flasks.

7.4.2 *Colour development*

To each flask, add 1 ml of the hydroxylammonium chloride solution (5.8) and allow to stand for 2 to 3 min. Add 10 ml of the sodium citrate solution (5.7), followed by 10 ml of the 1,10-phenanthroline solution (5.9), mixing after each addition, and dilute to about 70 ml. Add 10 ml of the ammonium acetate solution (5.6), dilute to the mark, mix and allow to stand for 15 to 30 min.

Expected iron content, % (*m*/*m*)					
0,002 to 0,1		0,001 to 0,05		0,000 4 to 0,02	
Standard iron solution (5.11)	Corresponding mass of Fe	Standard iron solution (5.11)	Corresponding mass of Fe	Standard iron solution (5.11)	Corresponding mass of Fe
ml	µg	ml	µg	ml	µg
0*	0	0*	0	0*	0
2,50	50	1,00	20	0,50	10
5,00	100	3,00	60	1,00	20
10,00	200	5,00	100	2,00	40
15,00	300	7,00	140	3,00	60
20,00	400	9,00	180	4,00	80
25,00	500	11,00	220	5,00	100
		13,00	260		
Optical path length of spectrophotometer cell, cm					
1		2		4 or 5	

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* Compensation solutions.

7.4.3 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements using the spectrophotometer (6.2) at a wavelength of about 510 nm and in cells of optical path length appropriate to the expected iron content (see the table in 7.4.1), after having adjusted the instrument to zero absorbance against water.

7.4.4 *Plotting the graph*

Deduct the absorbance of the compensation solution from that of each standard colorimetric solution and plot a graph having, for example, the numbers of micrograms of iron (Fe) contained in 100 ml of standard colorimetric solution as abscissae and the corresponding values of absorbance as ordinates.

7.5 Determination

7.5.1 *Colour development*

According to the expected iron content, place an exactly measured volume, not greater than 60 ml, of the test solution (7.3) in a 100 ml one-mark volumetric flask. Add a few drops of the phenolphthalein solution (5.12) and neutralize by the addition of the ammonium hydroxide solution (5.5). Add 3 to 5 drops of the hydrochloric acid solution (5.4) and 1 ml of the hydroxylammonium chloride solution (5.8) and allow to stand for 2 to 3 min. Add 10 ml of the sodium citrate solution (5.7), followed by 10 ml of the 1,10-phenanthroline solution (5.9) and dilute to about 70 ml, if necessary. Add 10 ml of the ammonium acetate solution (5.6) dilute to the mark, mix and allow to stand for 15 to 30 min.

7.5.2 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements on the test solution (7.3) and on the blank test solution (7.2), after colour development, by means of the procedure specified in 7.4.3.

8 EXPRESSION OF RESULTS

By means of the calibration graph (7.4.4), determine the masses of Fe corresponding to the absorbance of the aliquot portions of the test and blank test solutions taken for the colour development.

The iron content, expressed as a percantage by mass of Fe, is given by the formula

$$\frac{m_1 - m_2}{m_0 \times 10^6} \times D \times 100 = \frac{m_1 - m_2}{m_0} \times D \times 10^{-4}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in micrograms, of Fe found in the aliquot portion of the test solution taken for colour development;

m_2 is the mass, in micrograms, of Fe found in the corresponding aliquot portion of the blank test solution;

D is the ratio of the volume, in millilitres, of the test solution to the volume, in millilitres, of the aliquot portion taken for the colour development.

9 TEST REPORT

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HEXAFLUOROSILICATE FOR INDUSTRIAL USE

ISO 4281 — Free acidity and sodium hexafluorosilicate content.

ISO 5440 — Determination of phosphate content — Molybdovanadate spectrophotometric method.

ISO 5443 — Determination of iron content — 1,10-Phenanthroline spectrophotometric method.

ISO 5444 — Determination of loss in mass at 105 °C.

ISO 5915 — Determination of particle size distribution — Sieving method.

ISO 6229 — Determination of free silica content — Gravimetric method.