INTERNATIONAL STANDARD 2124

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Sodium and potassium silicates for industrial use — Determination of silica content — Titrimetric method

First edition — 1972-06-15

UDC 661.31/.32 : 546.284-31

Ref. No. ISO 2124-1972 (E)

Descriptors : chemical analysis, determination of content, potassium silicates, silicon dioxide, sodium silicates, volumetric analysis.

Price based on 2 pages

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 2124 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in March 1971 by the Member Bodies of the following countries:

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

No Member Body expressed disapproval of the document.

Printed in Switzerland

Sodium and potassium silicates for industrial use — Determination of silica content — Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a simple and rapid titrimetric method for the determination of the silica content of sodium and potassium silicates for industrial use.

2 REFERENCES

ISO/R 645, *Statistical vocabulary and symbols — First series of terms and symbols — Part 1 : Statistical vocabulary.*

ISO 2122, *Sodium and potassium silicates for industrial use — Preparation of solution of products not easily soluble in boiling water and determination of matter insoluble in water.* (At present at the stage of Draft).

3 PRINCIPLE

Neutralization of the alkalinity of a test portion, previously dissolved, with methyl red as indicator.

Addition of sodium fluoride, which reacts with the silicic acid, forming an equivalent quantity of sodium hydroxide.

Addition of a measured volume of standard volumetric hydrochloric acid solution in excess, followed by ethanol.

Filtration of the silicic compound thus obtained, which is insoluble under these conditions.

Titration of the excess of acid in the filtrate with standard volumetric sodium hydroxide solution with methyl red as indicator.

4 REACTIONS

$$H_2SiO_3 + 6\,NaF + H_2O \longrightarrow Na_2SiF_6 + 4\,NaOH$$

$$NaOH + HCl \longrightarrow NaCl + H_2O.$$

5 REAGENTS

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

5.1 Sodium fluoride, anhydrous, heated at 600 °C and cooled in a desiccator.

5.2 Ethanol, approximately 95 % (V/V) which may be denatured with ether.

5.3 Ethanol, approximately 50 % (V/V) which may be denatured with ether.

5.4 Hydrochloric acid, N standard volumetric solution.

5.5 Hydrochloric acid, approximately 0.1 N solution.

5.6 Sodium hydroxide, N standard volumetric solution.

5.7 Methyl red, 1 g/l solution in 95 % (V/V) ethanol.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Burette, 25 ml, graduated in 0.05 ml.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0.1 mg, a quantity of laboratory sample corresponding to 2.0 ± 0.3 g of SiO_2.

NOTE — In the case of products not easily soluble, use an equivalent quantity of the solution prepared according to the method described in ISO 2122.

7.2 Blank test

Place in a 250 ml conical flask, 70 ml of water, 5.00 ml of the sodium hydroxide solution (5.6) and 0.30 ml of the methyl red solution (5.7). Add, by means of a graduated pipette, approximately 4.5 ml of the hydrochloric acid solution (5.4) and neutralize with the hydrochloric acid solution (5.5) until the colour of the indicator turns from yellow to red. Then add 5 ± 0.1 g of the sodium fluoride (5.1) to the solution thus neutralized. Stir to aid dissolution of this product, then add a precisely measured volume (V_3) of the hydrochloric acid solution (5.4) corresponding to an excess of about 5 ml after the colour change to red.

Then add 50 ml of the ethanol (5.2), stir for about 1 min, transfer quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark with ethanol (5.3) and mix. Filter a little more than 100 ml on a folded, dry, close grained filter, discarding the first few millilitres. Take 100.0 ml of the filtrate and place in a 250 ml conical flask; add 0.30 ml of the methyl red solution (5.7) and titrate the excess of acid with the sodium hydroxide solution (5.6), contained in the burette (6.1), until the colour changes from red to yellow.

7.3 Determination

Place the test portion (7.1) in a 250 ml one-mark volumetric flask, if necessary dissolving it in water, dilute to the mark and mix.

Take 50.0 ml of this dilute solution immediately and place in a 250 ml conical flask. Add 0.30 ml of the methyl red solution (5.7). Neutralize with the hydrochloric acid solution (5.4) the total alkalinity less about 1 mg equivalent (if necessary, carry out a preliminary titration of this alkalinity on another 50 ml sample) and neutralize with hydrochloric acid solution (5.5) until the colour of the indicator changes from yellow to red. Then add to the neutralized product 5 ± 0.1 g of the sodium fluoride (5.1). Stir to aid dissolution of this product and then add a precisely measured volume (V_1) of the hydrochloric acid solution (5.4) corresponding to an excess of about 5 ml after the colour changes to red. Add 50 ml of the ethanol (5.2), stir for about 1 min, transfer to a 200 ml one-mark volumetric flask, dilute to the mark with the ethanol (5.3) and mix.

Filter a little more than 100 ml on a folded, dry, close grained filter, discarding the first few millilitres. Take 100.0 ml of the filtrate and place in a 250 ml conical flask; add 0.30 ml of the methyl red solution (5.7) and titrate the excess of acid with the sodium hydroxide solution (5.6) contained in the burette (6.1) until the colour changes from red to yellow.

8 EXPRESSION OF RESULTS

8.1 Formula for calculation

Silica content is given, as a percentage by mass, by the formula :

$$[(V_1 - 2V_2) - (V_3 - 2V_4)] \times 0.015\,02 \times \frac{250}{50} \times \frac{100}{m}$$

$$= [(V_1 - 2V_2) - (V_3 - 2V_4)] \times \frac{7.510}{m}$$

where

V_1 is the volume, in millilitres, of the hydrochloric acid solution (5.4) added for the determination (7.3);

V_2 is the volume, in millilitres, of the sodium hydroxide solution (5.6) used for the back titration in the determination;

V_3 is the volume, in millilitres, of the hydrochloric acid solution (5.4) added for the blank test (7.2);

V_4 is the volume, in millilitres, of the sodium hydroxide solution (5.6) used for the back titration in the blank test;

0.015 02 is the mass, in grams, of SiO_2 corresponding to 1 ml of the hydrochloric acid solution (5.4);

m is the mass, in grams, of the test portion.

8.2 Repeatability[1)]

The results obtained by this method are exact to ± 0.3 % in absolute value.

9 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.

1) **repeatability** : Closeness of agreement between successive results obtained with the same method on identical test material and under the same conditions (same operator, same apparatus, same laboratory and same time). (Definition taken from ISO/R 645.)