

STATE STANDARD OF THE RUSSIAN FEDERATION

Sodium silicate INSTANT

TECHNICAL TERMS

RUSSIAN STATE STANDARD

Moscow

STATE STANDARD OF THE RUSSIAN FEDERATION

Sodium silicate INSTANT

specifications

Soluble sodium silicate. Specifications

GOST R

50418-92

Date of introduction 01.01.94

This standard applies to soluble sodium silicate, produced in the form of lumps and granules intended for the manufacture of liquid glass in accordance with GOST 13078.

1. TECHNICAL REQUIREMENTS

1.1. Soluble sodium silicate, must meet the requirements of this standard and technological documentation, approved in the established order.

1.2. On physical and chemical parameters of sodium silicate, must comply with the standards specified in the table.

1.3. chemical composition standards are set based on the calcined product.

1.4. Sodium silicate should be made pieces the size of not less than 10 and not more than 150 mm. Admitted pieces smaller than 10 and more than 150 mm, if their total weight does not exceed 40% of the batch weight and 30% in the production of liquid glass, used for welding materials. The maximum size of the pieces of sodium silicate - 200 mm.

2. ACCEPTANCE

2.1. Sodium silicate accepted in batches. Party consider any number of homogeneous in its product quality indicators, drawn up as a single document containing:

2.2. In each batch of sodium silicate determine its appearance, the mass fraction of silicon dioxide, sodium oxide and silicate module. The mass fraction of iron oxide and aluminum oxide, calcium oxide and sulfur manufacturer determines from the customer's request.

2.3. To verify compliance with product quality requirements of this standard, a sample of the total weight of not less than 2 kg.

2.4. If test results are unsatisfactory at least one of the indicators to repeat the test on the indicator at twice the sample from the same lot. repeated test results apply to the entire batch.

3. METHODS OF ANALYSIS

3.1. Sample selection

3.1.1. Spot samples of equal weight are taken with forceps or stainless steel scoop from three different places total weight of not less than 2 kg. Selected single samples are combined, mixed and cut by quartering to obtain a combined sample weight of at least 750

3.1.2. The combined sample was divided into two equal parts, one of which is subjected to the tests, and the other is placed in a clean, dry plastic jar with a lid or cork and stored for three months in case of differences in the quality assessment. On the bank adhesive label indicating the product name, batch number, date of sampling and sample number.

3.1.3. The selected part of the test sample cut by quartering to 20-25 g, wipe or wash the pieces with ethyl alcohol according to GOST 18300 (1-2 cm³ per sample), fast ground in an agate, chalcedony and quartz, a mortar to a state of powder, 12-15 grams of which placed in a beaker for weighing, close tightly with a lid and put in a desiccator. It prevents mechanical abrasion tests. The resulting powder is intended for all subsequent tests.

3.2. General requirements for methods of analysis

For weighing samples analyzed, residues after drying and calcination, chemical reagents used for the preparation of standard and titrant used laboratory scale according to GOST 24104 with a weighing error of no more than 0.0002 g and a maximum weighing limit of 200 grams, the weight D-2-210 GOST 7328. The weighing result is recorded with an accuracy of four decimal figures.

Reagents for preparation of buffer solutions and support is weighed on a laboratory scale in accordance with GOST 24104 with an error of less than 0.01 g and a maximum weighing limit of 1 kg. Use weights G-4-1110 according to GOST 7328. The weighing result is recorded to the nearest second decimal.

For the analysis of used chemical reagents qualification of not less than "pure for analysis" and distilled water in accordance with GOST 6709.

For filtration use decalcified paper filters.

.. B "diluted 1: 1, 1: 2", etc. expression digits indicate the first volume part of acid or ammonia, and the second - the bulk of the water. If the concentration or dilution of the acid or the aqueous ammonia solution is not specified, is meant a concentrated acid or concentrated ammonia solution.

To analyze the volumetric glassware used in accordance with GOST 1770 of not less than 2 grade accuracy. To measure the volume of aliquots of solutions of samples, standard and volumetric solutions using volumetric glassware on the normative and technical documentation (pipettes, burettes). Measuring the volume of support and buffer solutions is carried out measuring glassware according to GOST 1770 (cylinders, beakers).

The expression "hot water" or "hot solution" denotes that the fluid temperature is 60-80 ° C and "hot water" or "warm solution" - the liquid temperature is 40-50 ° C.

To construct the calibration curves requires a minimum of five points, which should be distributed evenly over the measuring range, and the minimum and maximum measurement values establish the

limits of the measuring range. Calibration curves checked at least once every 6 months, as well as when replacing reagents or photocolormeter.

Accurate solution concentration is calculated to four decimal places as a result of at least three parallel determinations.

3.3. Appearance of sodium silicate is determined visually.

3.4. Determination of mass fraction LOI

3.4.1. Equipment

Laboratory muffle furnace with heating to a temperature of 500 ° C.

Porcelain crucibles with lids number 3 and 4 in accordance with GOST 9147.

3.4.2. analysis

In the pre-calcined and weighed porcelain crucible placed 0.8-1.0 g of sodium silicate powder. The crucible with the sample cover lid 3/4 (hereinafter avoid splashing), calcined by gradually increasing the temperature in an electric muffle furnace and kept at a temperature of 400-420 ° C for 30-40 min. The crucible with the sample is cooled in a desiccator and weighed. Repeat baking to constant weight.

3.4.3. Processing of results

The weight proportion LOI (X) as a percentage calculated by the formula

where m_1 - weight loss after calcination sample, g;

m - mass of sample sodium silicate, city

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.3%.

The total relative error in the analysis of $\pm 0,4\%$ at a confidence level of $P = 0.95$.

3.5. Determination of the mass fractions of silicon dioxide (gravimetric method)

3.5.1. The essence of the method

The method is based on the decomposition of the sample by dissolution in hot water, double dehydration silicic acid in hydrochloric acid medium, separation and calcination of a precipitate, distilling off the silicon tetrafluoride by treatment with hydrofluoric acid.

3.5.2. The reagents, solutions, equipment

Hydrochloric acid in accordance with GOST 3118, x. h., diluted 1:99.

Sulphuric acid in accordance with GOST 4204, x. h, diluted 1: 1.

Acid hydrofluoric according to GOST 10484, s. h.

Ammonia water according to GOST 3760, x. h.

Ethyl alcohol in accordance with GOST 18300 premium.

Crucibles of platinum number 100-7 or 100-8 number and cover a platinum crucible № 101-7 or 101-8 number in accordance with GOST 6563.

Laboratory muffle furnace with a heating temperature of 1000-1100 ° C.

Porcelain cup number 4 in accordance with GOST 9147.

Cups for weighing in accordance with GOST 25336.

3.5.3 Analysis

0.8-1.0 g of sodium silicate powder is weighed in a weighing beaker and carefully poured into a porcelain cup. Glass for weighing immediately weighed and the mass of sample is calculated by mass difference.

A weighed sample was treated in a cup of hot water of 75-100 cm³, is placed on the tiles, with a watch glass cover and gently boil while stirring for 15-30 minutes to dissolve the drug.

When determining silica silicate with low modulus of sample silicate can be dissolved in a porcelain dish on a boiling water bath. Then the cup through the spout into the solution while stirring with a glass rod frequently administered dropper 25 cm³ of hydrochloric acid, and the cup should be covered with a watch glass. Without removing the glass cup with the contents was placed in a boiling bath for 10-15 minutes after which the watch glass is removed and it is washed with water and the cup. The resulting solution a total volume of 150 cm³ together with sludge precipitated silica gel is evaporated to near dryness on a boiling water bath or under infrared lamp. The resultant solid was triturated gently pestikoobraznoy to a powder with a glass rod, which should be in the cup. Heating on a steam bath or under infrared lamp is continued until complete disappearance of the odor of hydrogen chloride. The completeness of removal of hydrogen chloride is also checked with a stick dipped ammonia. For a more complete dehydration of the silica cup in a water bath kept 2 more hours.

The dried precipitate is moistened and cooled hydrochloric acid 3.4 cm³ and covering with a watch glass dish, incubated for 15-20 min, after which it is poured into hot water 70-75cm³. The solution with the precipitate was stirred with a glass rod to give a precipitate cup stand (not more than 10 min) and then decanted to filter "white ribbon". The precipitate was washed 3-4 times by decantation with small portions of hot water, bringing it to the filter and then the filter is 8-10 times with warm dilute (1:99) solution of hydrochloric acid.

The silicic acid was recovered again by evaporating the resulting filtrate together with the washings in the same porcelain dish on a boiling water bath to dryness, and repeating a previous operations, except for decanting washing.

Both the filter with the precipitate of silicic acid is dried to slightly damp, carefully wrapped inside edge of the filter, closing this sediment cone and tightly stacked up in the pre-calcined and weighed platinum crucible.

The contents of the crucible and ashed calcined in an electric furnace at a laboratory temperature of 1000-1100 ° C to constant weight.

The calcined precipitate is treated with a few drops of diluted (1: 1) sulfuric acid and 7.5 cm³ of hydrofluoric acid. The resulting mixture was evaporated to dryness on a hotplate. Then the dried salt residue was calcined by gradually raising the temperature to 1000-1100 ° C in a muffle furnace and within 15-20 minutes. After cooling in a desiccator, with the remainder of the crucible is weighed.

3.5.4. Processing of results

The weight proportion of silica (X1) in percentage is calculated by the formula

where m_1 - mass of the crucible with the residue of silicon dioxide, g;

m_2 - mass of the crucible with the residue after treatment with hydrofluoric acid, in grams;

m - mass of the sample of sodium silicate, g;

x - mass fraction of loss on ignition (§ 3.4.), %.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.3%.

The total relative error in the analysis of $\pm 0,45\%$ at a confidence level of $P = 0.95$.

3.6. Determination of the mass fraction of silicon dioxide (combined method)

3.6.1. The essence of the method

The essence of the method lies in the gravimetric determination of the bulk silicon dioxide. The filtrate determine the content silica photolorimetry the blue color of the reduced kremnemolibdenovogo complex. The weight proportion of silicon dioxide is defined as the sum of the results of gravimetric and photolorimetric methods.

3.6.2. The reagents, solutions, equipment

Reagents, solutions, equipment -. 3.5.2 of Claim and

Photoelectric colorimeter in accordance with GOST 12083, or a spectrophotometer.

Silica according to GOST 9428.

Ascorbic acid, with a mass fraction of 5% solution.

Ammonium molybdenum-acidic according to GOST 3765, with a mass fraction of 5% solution.

Salt of the oxide of iron and ammonium double sulfuric acid (Mohr's salt) in accordance with GOST 4208, with a mass fraction of 4% solution.

Thiourea according to GOST 6344, with a mass fraction of 5% solution.

Copper (II) of sulfuric acid 5 water (copper sulphate) in accordance with GOST 4165, with a mass fraction of 0.4% solution.

Sodium carbonate anhydrous 83 according to GOST.

Hydrochloric acid according to GOST 3118, diluted 1: 1 and the molar concentration of solution (HCl) = 0.5 mol / dm³.

3.6.3. Preparation for analysis

I reductants mixture is prepared by mixing equal volumes of Mohr salt solution and ascorbic acid. The mixture was stored in a dark place is not more than 7 days.

II reducing mixture is prepared by mixing equal volumes of solutions of thiourea and sulfuric acid copper.

To prepare a standard solution containing 0.25 mg / cm³ of SiO₂, weighed 0.0625 g of finely powdered silica, previously calcined at a temperature of 1000-1100 ° C to constant weight, and fused in a platinum crucible, with 1 g of anhydrous sodium carbonate. The melt is leached with water, the resulting solution was placed in a volumetric flask of 250 cm³, made up to the mark with water and mix. The solution was stored in a plastic container.

3.6.4. analysis

The bulk of the silica obtained by a single release in accordance with para. 3.5.3. The filtrate with the washings are collected in a volumetric flask of 250 cm³, the volume is adjusted to the mark with water and mixed thoroughly. An aliquot of the sample solution in an amount of 25 cm³ or 50 placed in a volumetric flask of 100 cm³ were added 10 cm³ of distilled water, 4 cm³ of an acidic solution of ammonium molybdenum. After 10 minutes, add 20 cm³ of hydrochloric acid of 1: 1 and 2 cm³ of the mixture of reducing solution (I or II), mixed well and the volume adjusted solution with water to the mark. comparison solution containing no SiO₂, prepared in conjunction with the test. The solutions were allowed to stand for 20 minutes.

The absorbance of the sample solution against a reference solution at photoelectrocolorimeter measured using a red filter (λ = 650 nm) in cells with a layer thickness of 5 cm. The content of SiO₂ is determined by the calibration curve.

3.6.5. Building a graded schedule

The flasks with 100 cm³ burette metered standard solution containing 0.25; 0.5; 0.75; 1.0; 1.25 mg SiO₂. At the same time prepare a comparison solution containing no SiO₂. Each solution was added to 20 cm³ of water and 5 cm³ of hydrochloric acid solution of concentration c (HCl) = 0.5 mol / dm³ solution of 4 cm³ molybdenum acid ammonium. After 10 min, a solution of 20 cm³ of hydrochloric acid (1: 1) solution and 2 cm³ of the mixture of reducing agents. Bring the solution volume up to the mark with water and mixed thoroughly. The solutions were allowed to stand for 20 minutes.

Absorbance solutions was measured as described in Sec. 3.6.4. The data obtained are building the calibration curve.

3.6.6. Processing of results

The weight proportion of silica in the filtrate (after a single deposition) (X₂) as a percentage calculated by the formula

where m₃ - mass of silica, found on the calibration curve, mg;

m - mass of sample sodium silicate, g;

V - total volume of permeate solution cm³;

V₁ - volume of the aliquot part taken for fotokolorimetrirovaniya cm³

x - mass fraction of loss on ignition (§ 3.4.), %.

The overall weight proportion of silica (X₃) as a percentage calculated by the formula

$$X_3 = X_1 + X_2,$$

wherein X_1 - mass fraction of silica after a single allocation of claim 3.5.4..

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.3%.

The total relative error in the analysis of $\pm 0,45\%$ at a confidence level of $P = 0.95$.

3.7. Determination of the mass fractions of silicon dioxide (volumetric method)

3.7.1. The essence of the method

The method is based on the titrimetric determination of the amount of sodium hydroxide (equivalent of silicic acid) which is formed in the neutralized solution after separation of the precipitate in the presence of sodium hexafluorosilicate ethanol.

3.7.2. Apparatus, reagents, solutions

A cup of glassy carbon.

Platinum Spatula number 209-2 in accordance with GOST 6563.

Laboratory muffle furnace with a heating temperature of at least 600°C .

Burette of normative-technical documentation with a capacity of 25 cm³.

Sodium fluoride according to GOST 4463, x. h., dehydrated at 600°C and cooled in a desiccator.

Rectified ethyl alcohol Technical GOST 18300 premium and mass fraction of 50% solution.

Hydrochloric acid in accordance with GOST 3118, x. h., solutions with a molar concentration $(\text{HCl}) = 0.1 \text{ mol / dm}^3$ and $c(\text{HCl}) = 1 \text{ mol / dm}^3$.

Sodium hydroxide according to GOST 4328, x. h., solutions of molar concentration $c(\text{NaOH}) = 1 \text{ mol / dm}^3$, with $(\text{NaOH}) = 0,1 \text{ mol / dm}^3$.

Methyl red indicator TU 6-09-5169-84, preparation of the solution in accordance with GOST 4919.1.

Distilled water in accordance with GOST 6709.

3.7.3. analysis

5.4 g of sodium hydroxide in the cup is weighed glassy gently and quickly (3-5 min) was melted and cooled.

1.9-2.0 g of sodium silicate powder is weighed in a weighing beaker and carefully poured into a cup to the surface of the melt solidified, trying to distribute a sample of the spatula across the surface of the sodium hydroxide. Glass for weighing -vzveshivayut again and determine the mass difference between the mass of sample sample. The dish was warmed on a hotplate, continuously stirring the mixture with a spatula, and then melted at a temperature of $400-500^\circ\text{C}$ for 3-5 min. Cup is then cooled, the melt is dissolved in hot water and transferred quantitatively to beaker 300-400 cm³ beaker is then heated to

melt completely dissolved. Introduced into the solution a few drops of methyl red and the solution acidified with hydrochloric acid, giving a 2.1 drop in excess, and the solution should be clear.

The solution is cooled and transferred into a volumetric flask of 250 cm³ and the volume is adjusted with water to the mark and the solution is stirred. 50 cm³ of the prepared solution was placed in a conical flask of 250 cm³ was added 0.3 cm³ of methyl red and neutralized with sodium hydroxide solution of concentration 1 mol / dm³ to transition to a yellow red color, and then add a solution of hydrochloric acid concentration of 0.1 mol / dm³ to move the yellow color of the solution in the red.

To the neutralized solution was added (5,0 ± 0,1) g of sodium fluoride, stirred until dissolution, added precisely measured volume of a solution of hydrochloric acid concentration of 1 mol / dm³ (V1) until the yellow color transition to the red solution and the excess of acid 5 cm³. Then added to a solution of 50 cm³ of ethanol, stirred for 1 min, transferred to a flask of 200 cm³ capacity, diluted with ethanol up to the mark with a mass fraction of 50%, and stirred again. About 100 cm³ of the resulting solution is filtered into a dry beaker through a 300-400 cm³ dry pleated filter "blue ribbon", discarding the first 3-4 cm³.

3.7.4. control experiment

The conical flask was placed 250 cm³ 70 cm³ of water and 0.8-1.0 g of sodium hydroxide. Dissolved, stirring, add 0.3 cm³ of methyl red and neutralized dropwise with hydrochloric acid solution to change the yellow color in the red. Then the first solution was added sodium hydroxide concentration of 1 mol / dm³ until the yellow color of the solution and then hydrochloric acid solution of concentration c (HCl) = 0.1 mol / dm³ solution until the red color.

To the neutralized solution was added (5,0 ± 0,1) g of sodium fluoride, stirring well until dissolved and add accurately measured volume of a solution of hydrochloric acid concentration c (HCl) = 1 mol / dm³ (V3) to transfer the yellow solution red in color and an excess of acid 5 cm³. Then 50 cm³ of ethanol, stirred for 1 min, transferred to a flask of 200 cm³ capacity, diluted with ethanol up to the mark with a mass fraction of 50%, and stirred again. About 100 cm³ of the resulting solution is filtered into a dry beaker through a folded 300-400 cm³ dry filter "blue ribbon", discarding the first 3-4 cm³. 100 cm³ of the filtrate was placed in a conical flask of 250 cm³ was added 0.3 cm³ of methyl red and titrate the excess acid with sodium hydroxide concentration c (NaOH) = 1 mol / dm³ to red color transition in a yellow solution. Note the volume of sodium hydroxide solution, who had gone on titration (V4).

where V1 - volume of hydrochloric acid concentration of exactly 1 mol / dm³ added for determining cm³;

V2 - volume of sodium hydroxide solution concentration of exactly 1 mol / dm³ spent on back titration cm³;

V3 - volume of hydrochloric acid concentration exactly 1 mol / dm³ added to the control experiment, cm³;

V4 - volume sodium hydroxide solution concentration exactly 1 mol / dm³ spent on back titration in a control experiment, cm³;

0.01502 - the mass of silicon dioxide, corresponding to 1 cm³ of hydrochloric acid concentration of exactly 1 mol / dm³, g;

m - mass of the sample, g;

x - mass fraction of loss on ignition (§ 3.4.),%.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them should not exceed the allowable of 0.6%.

When differences in the evaluation of the mass fraction of silica analysis was performed by gravimetric method.

3.8. Determination of the mass fraction of the sum of iron oxide and aluminum oxide (Complexometric method)

3.8.1. The essence of the method

The method is based on the formation of complex compounds of aluminum and iron with Trilon B, subject to an excess. The excess is titrated with Trilon B solution of sulfuric acidic zinc in the presence of a buffer at pH 5.2-5.8 with the indicator xylenol orange. The sample was pre-decomposed with a mixture of hydrofluoric and sulfuric acids.

3.8.2. Apparatus, reagents, solutions

A cup of platinum № 118-3 in accordance with GOST 6563.

Spatula Platinum № 209-11 in accordance with GOST 6563.

Acid hydrofluoric according to GOST 10484, s. h.

Sulphuric acid in accordance with GOST 4204, x. h.

Acetic acid, 61 according to GOST, x. h.

Hydrochloric acid in accordance with GOST 3118, x. h.

Ammonia water according to GOST 3760, x. h, solutions with a mass fraction of 25 and 10%.

Hydroxylamine hydrochloride sour according to GOST 5456, with a mass fraction of 10% solution.

Ammonium acetic acidic in accordance with GOST 3117.

Indicators: paper "Congo" red, Eriochrome Black T, xylenol orange, prepared in accordance with GOST 4919.1, acid chrome dark blue, is prepared as follows: 0.15 g of the indicator is dissolved in 5 cm³ of ammonia buffer solution, added 20 cm³ of ethanol and stirred.

Zinc, GOST 3640

Zinc sulphate GOST 4174, with a solution concentration of $(1 / 2ZnSO_4) = 0.025 \text{ mol} / \text{dm}^3$.

Distilled water in accordance with GOST 6709.

Trilon B (ethylenediaminetetraacetic acid disodium salt) according to GOST 10652, the solution concentration of 0.025 mol / dm³.

Rectified ethyl alcohol Technical GOST 18300 premium.

Acetate buffer solution with pH 5.2-5.8, was prepared as follows: 100 g of acetic acid dissolved in ammonium 300-400 cm³ of water (for better dissolution solution is heated), filtered, and added to 10 cm³ of acetic acid diluted to 1 dm³ and stirred.

Ammonia buffer solution having a pH of 9,5-10,0 prepared as follows: 54 g of ammonium chloride dissolved in 200 cm³ of water, the resulting solution was added 350 cm³ of ammonia solution with a mass fraction of 25% volume of water and the solution adjusted to 1 dm³.

3.8.3. Preparation of volumetric solutions

Trilon B solution concentration of 0.025 mol / dm³, is prepared as follows:

9.31 g Trilon B is dissolved in water in a volumetric flask 1 dm³ capacity, then the volume is brought up to 1 dm³ of water. The solution was stored in plastic or glass, waxed inside vessels. Allowed preparation of a solution of standard-titer Trilon B.

the concentration of zinc salt solution is precisely 0.025 mol / dm³ (to determine the correction coefficient Trilon B solution), is prepared as follows:

1.6345 g of zinc, of freshly purified from steel knife oxide immediately weighed, placed in a porcelain dish and dissolved by heating on a steam bath in a mixture of 100 cm³ of water and 15 cm³ of concentrated nitric acid, covered with a watch glass cup. Then thoroughly washed with water glass, collecting it in the same cup, and the solution was evaporated to 4.3 cm³. The residue from the cup quantitatively transferred by washing the walls of the cup with water in a volumetric flask with a capacity of 1 dm³ water and bring the volume of the solution to the mark. The solution is fit for one month.

Correction factor Trilon B solution concentration of 0.025 mol / dm³ determined by solution of zinc salt as follows:

to 25 cm³ concentration of zinc salt solution precisely 0.025 mol / dm³ was added 5 cm³ ammoniacal buffer solution, 0.1 g of a mixture of the indicator eryochrom T or acidic chromium 10-12 drops of dark blue solution and 70 cm³ of water.

The solution was stirred and titrated from a burette solution of Trilon B to move purple red color to blue (when using eryochrom black-T) or raspberry in the immutable blue (using acid chrome dark blue). Correction factor (K) solution of Trilon B concentration of 0.025 mol / dm³ is calculated according to the formula,
where V - volume solution of Trilon B concentration of 0.025 mol / dm³ spent on titration cm³.

A solution of sulfuric acid zinc concentration of 0.026 mol / dm³, is prepared as follows:

7.2 g zinc sulfuric acid dissolved in water and the solution adjusted to volume with water to 1 dm³.

Ratio of conformity of the solution is determined as follows:

25 cm³ of a sulfuric acid solution of zinc were placed in a titration flask, 70 cm³ of water are added, 5 / cm³ ammoniacal buffer solution, 0.1 g of black display eryochrom mixture and titrate T Trilon B concentration of 0.025 mol / dm³ to move in the blue violet color .

Matching coefficient (K1) of a solution of sulfuric acid zinc concentration of 0.025 mol / dm³ was calculated by the formula.

where V - volume solution of Trilon B concentration of 0.025 mol / dm³ spent on titration cm³;

K - correction factor Trilon B solution concentration of 0.025 mol / dm³.

3.8.4. Preparation of test solution

2.0-2.1 g of sodium silicate are weighed in a platinum gull. At the same time carry out a control experiment for purity reagents. A weighed sample is moistened with water and mixed with 2 cm³ 10.7 cm³ of sulfuric acid hydrofluoric acid. The mixture was first evaporated in a water bath until removal of hydrofluoric acid, then transferred on an electric hot plate and heated to release copious fumes of sulfuric acid. When the contents of the cup will become a syrupy mass, frozen on cooling, heating is stopped.

Thereafter, the cup was poured 10-15 cm³ water, 4.3 cm³ of hydrochloric acid, and stirring with a glass rod, the residue salt is completely dissolved with gentle warming on a water bath.

The solution was transferred into a volumetric flask of 100 or 250 cm³ cup rinsed at least three times with hot water and merging water each time into the same flask. Then the flask and its contents cooled to ambient temperature, topped up with water to the mark and mixed.

3.8.5. analysis

50 cm³ of a solution prepared according to paragraph. 3.8.4, dry transfer pipette into the conical flask of 250 cm³ 15 cm³ of the solution added Trilon B, dipped paper "Congo", 30-35 cm³ of water are added, warmed to 50 ° C and was added dropwise aqueous ammonia with a mass fraction of 25% to redness paper and then aqueous ammonia solution with a mass fraction of 10% to a bright red color of the paper. Then add 20 cm³ of acetate buffer solution, 10 cm³ of an acidic solution of hydroxylamine hydrochloride and heated 1-2 min. Thereafter immediately titrated with sulfuric acid zinc xylenol orange indicator 10 drops tentatively added to the solution. Titration is continued until the yellow color of the solution transition to violet-red. Note the volume of the solution of sulfuric acidic zinc consumed for the titration (V₁). In processing the results must take into account the result of the control experiment.

3.8.6. Processing of results

Weight fraction sum of iron oxide and alumina in terms of alumina (X) as a percentage calculated by the formula, where V₂ - volume of the solution of zinc sulfate concentration of 0.025 mol / dm³ spent on titration of blank test, cm³;

V₄ - volume of sulfuric acid solution of zinc concentration of 0.025 mol / dm³ spent on back titration samples cm³;

K₁ - compliance rate solution of sulfuric acidic zinc concentration of 0.025 mol / dm³;

V₃ - the total volume of the sample solution, cm³;

0.001275 - Al₂O₃ mass corresponding to 1 cm³ solution Trilo-B at a concentration of exactly 0,025 mol / dm³, g;

m - mass of the sample, g;

V₄ - the volume of an aliquot of the sample solution, cm³;

x - mass fraction of loss on ignition (§ 3.4.),%.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.05%.

The total relative error analysis ± 5% at a confidence level of P = 0.95.

3.9. Determination of the mass fraction of iron oxide (III)

3.9.1. The essence of the method

The method is based on the formation of iron sulfosalicylate colored in ammoniacal medium yellow (pH 8-11,5) and subsequent fotokolorimetrirovanii colored solution.

3.9.2. Apparatus, reagents, solutions

Photoelectrocolorimeter of normative and technical documentation, or a spectrophotometer.

Hydrochloric acid in accordance with GOST 3118, diluted 1: 1.

Ammonia water according to GOST 3760, with a mass fraction of 10% solution.

Sulfosalicylic acid in accordance with GOST 4478, with a mass fraction of 20% solution.

Methyl red TU 6-09-5169, preparing a solution according to GOST 4919.

Ammonium chloride according to GOST 3773, the concentration of a solution of 1 mol / dm³, is prepared as follows:

53.50 g of ammonium chloride dissolved in 1 dm³ of water and, if necessary, filtered.

Ammonium nitrate as per GOST 22867, with a mass fraction of 2% solution.

Ammonium iron (III) sulfate (1: 1: 2) 12 Water (ferric alum) TU 6-09-5359.

Iron oxide TU 6-09-5346.

3.9.3. Preparation of standard solutions of iron oxide (III)

A solution of the mass concentration of 1 mg / cm³ Fe₂O₃, is prepared as follows:

6.0399 g of ferric alum was dissolved in 100 cm³ of distilled water acidified with 9 cm³ of hydrochloric acid and diluted with water to 1 dm³.

The exact concentration of the solution is adjusted gravimetrically. For this purpose from the burette metered into three glasses with 300 cm³ each of 10, 20, 30 cm³ of standard solution is diluted to 120-150 cm³ of water, heated to boiling and iron hydroxide is precipitated by ammonia in the presence of methyl red. The resulting precipitate was filtered through a "white ribbon", washed with a hot solution of ammonium nitrate in a mass fraction of 2%, which added a few drops of ammonia. Filter the precipitate was placed in a weighed and precalcined platinum crucible, ashed calcined to constant weight at 850 ° C and calculating the mass concentration of solution (mg / cm³ Fe₂O₃).

Allowed to prepare standard solutions of iron oxide (III).

Solution A was prepared as follows:

1 g of dried at (110 ± 5) ° C for 1 hour, the iron oxide is placed in a conical flask of 500 cm³, 100 cm³ poured in HCl 1: 1, and covering a flask heated on a steam bath until complete dissolution. The solution was cooled, transferred to a volumetric flask of 1000 cm³, was adjusted to the mark with water and mix. Solution B mass concentration 0.1 mg / cm³ Fe₂O₃, prepared by dilution with water A solution ten times.

The solution containing a mass concentration of 0.01 mg / cm³ Fe₂O₃ prepared by diluting solution B with water ten times.

3.9.4. Building a graded curve

The flasks with 100 cm³ metered burette 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 cm³ of solution B.

To each flask is added 10 cm³ of solution of ammonium chloride concentration of 1 mol / dm³, 15 cm³ sulfosalicylic acid solution with a mass fraction of 20% ammonia were added dropwise with a solution of mass fraction of 10% until a stable yellow color and additionally 3 cm³. The solutions were mixed and adjusted with water to the mark.

At the same time prepare a comparison solution containing no Fe₂O₃.

The second calibration curve is constructed similarly to the first, but this solution was taken in a mass concentration of 0.01 mg / cm³.

Absorbance was measured at photoelectrocolorimeter solutions using a blue filter (450 nm wavelength) in cuvettes with 30 mm thickness.

The data obtained are building the calibration curve.

optical density value was plotted on the ordinate, and the corresponding amount in milligrams Fe₂O₃ - on the horizontal axis.

To construct the calibration curve of each point is calculated arithmetic mean optical density results of three parallel determinations.

The calibration curve is checked at least once every 6 months, as well as when replacing reagents or photoelectrocolorimeter.

3.9.5. analysis

In a volumetric flask of 100 cm³ transfer 25 cm³ of the test solution prepared in accordance with para. 3.8.4, poured in 10 cm³ of ammonium chloride concentration of 1 mol / dm³, 15 cm³ sulfosalicylic acid solution with mass fraction of 20% ammonia until the first stable yellow coloration and additionally 3 cm³, then diluted with water to the mark. At the same time prepare a comparison solution containing no Fe₂O₃.

3.9.6. Processing of results

According to the optical density obtained using the calibration curve, find the content of Fe₂O₃ in the test solution,

The weight proportion of iron oxide (X5) as a percentage calculated by the formula

where m_1 - Fe_2O_3 weight, found on the calibration curve, mg;

V - total volume, cm^3 ;

m - mass of the sample, g;

V_1 - volume of the aliquot part of the solution taken for photolorimetrovaniya cm^3 ;

x - mass fraction of loss on ignition (§ 3.4.), %.

If necessary, the determination result is corrected for the mass fraction of the iron oxide control experiment determined.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.01%.

The total relative error analysis $\pm 5\%$ at a confidence level of $P = 0.95$.

3:10. Photolorimetric method for determining a mass fraction of iron oxide (III) with 1,10-phenanthroline

3.10.1. The essence of the method

The method is based on the formation of an orange-red iron complex compounds with 1,10-phenanthroline ($\lambda = 508 \text{ nm}$). Ferric iron is reduced pre-hydroxylamine hydrochloride.

3.10.2. The reagents, solutions, equipment

Photoelectric colorimeter of normative and technical documentation, or a spectrophotometer.

Acetic acid, 61 according to GOST.

Hydroxylamine hydrochloride according to GOST 5456, with a mass fraction of 10% solution.

Sodium acetic acid, GOST 199, with a mass fraction of 40% solution.

1,10-phenanthroline, alcohol solution with a mass fraction of 1%.

Rectified ethyl alcohol Technical GOST 18300 premium.

The acetate buffer solution with pH 5.0, is prepared as follows:

38 g acetic acid sodium dissolved in 500 cm^3 of water was added 6.7 cm^3 of acetic acid, made up with water to a volume of 1000 cm^3 and mixed.

Standard solutions of iron oxide (III), prepared as described in Sec. 3.9.3.

Universal indicator paper.

3.10.3. analysis

Volumetric flask of 100 cm^3 25 cm^3 of transfer of the test solution prepared according to para. 3.8.4, 3 cm^3 added hydroxylamine hydrochloride solution added dropwise and acetic acid-sodium solution to pH 3.0 (pH controlled with universal indicator paper). Then add 10 cm^3 buffer, 2 cm^3 solution of 1,10-phenanthroline, made up to the mark with water and mix. After 40 min the absorbance is measured at photoelectrocolorimeter using a green filter with a wavelength $\lambda = 540 \text{ nm}$ cuvettes with 30 mm thickness. The reference solution used as a control experiment, a solution prepared as well as the test solution.

The content of iron oxide (III) in the test solution found in milligram calibration curve.

3.10.4. Construction of the calibration curve

The volumetric flasks is poured on 100 cm^3 of the burette 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 cm^3 of standard solution B mass concentration of 0.01 mg / cm^3 Fe_2O_3 . At the same time prepare the reference solution. Next come, in accordance with para. 3.10.3. According to the obtained values of the optical density of the solutions and known content of iron (III) oxide build a calibration graph. The second calibration curve is constructed similarly to the first with a solution B mass concentration of 0.1 mg / cm^3 Fe_2O_3 .

3.10.5. Processing of results -. 3.9.6 on p.

When differences in the evaluation of the mass fraction of iron oxide analysis was performed photolorimetry method with 1,10-phenanthroline.

3.11. Determination of the mass fraction of calcium oxide (Complexometric method)

3.11.1. The essence of the method

The method is based on direct titration Trilon B sample solution at pH 12 with indicator chrome dark blue or calcein after decomposition of sodium silicate with a mixture of hydrofluoric and sulfuric acids, and dissolving the residue in dilute hydrochloric acid.

3.11.2. Reagents and solutions

Sodium sulfide (sulfide) in accordance with GOST 2053, with a mass fraction of 2% solution (freshly prepared).

Sodium hydroxide according to GOST 4328, x. h., the solution concentration of 2 mol / dm³.

Distilled water in accordance with GOST 6709.

Indicator "Congo" red.

Refined sugar in accordance with GOST 22, with a mass fraction of 2% solution.

Acid Chrome Dark Blue, prepared according to para. 3.8.2.

Indicator calcein.

Indicator Thymolphthalein.

Potassium chloride in accordance with GOST 4234.

Indicator malachite green, an alcoholic solution with a mass fraction of 0.2%:

The indicator calcein mixture: 0.2 g of calcein and 0.165 g Thymolphthalein ground in a porcelain mortar with 20 g of potassium chloride.

Trilon B concentration of 0.025 mol / dm³ solution is prepared by para. 3.8.3. Correction factor of Trilon B is determined by n. 3.8.3.

Potassium hydroxide in accordance with GOST 24363, the concentration of a solution of 5 mol / dm³.

3.11.3. analysis

Preparation for the determination of calcium oxide solution is carried out as described in Sec. 3.8.4. The resulting solution was quantitatively transferred to a conical flask of 250 cm³ was added 2 cm³ of solution of sugar, water was added to a volume of 85-90 cm³, pH paper dipped "Congo", is neutralized with sodium hydroxide to redness paper added 2 cm³ solution of sodium sulphide stirred added 5 cm³ of sodium hydroxide, the solution was incubated for 1-2 minutes, add 8-10 drops of dark blue and titrate the acidic chromium solution with continuous stirring to a solution of Trilon B crimson color transition Unchanged blue solution.

In the case of mixtures with calcein Thymolphthalein titration conical flask with 250 cm³ was placed a solution obtained according to para. 3.6.4, diluted with water to a volume of 80-100 cm³ added 1-2 drops of malachite green and poured dropwise hydroxide solution potassium solution until colorless, then it is added an excess of 10 cm³. To this solution was added a mixture of 30-50 mg and calcein thymolphthalein indicator and titrate from a burette Trilon B solution concentration of 0.025 mol / dm³ to a sharp change of green fluorescence of the solution and transfer it to the violet color. Titration should be conducted on a black background. Note the amount of Trilon B, who had gone to the titration of calcium oxide.

3.11.4. Processing of results

The weight proportion of calcium oxide (X₄) in percentage is calculated by the formula, where V - volume solution of Trilon B concentration of 0.025 mol / dm³ spent on titration cm³;

K - correction factor Trilon B solution concentration of 0.025 mol / dm³;

0.0014 - CaO mass corresponding to 1 cm³ solution of Trilon B concentration of exactly 0,025 mol / dm³, g;

V₁ - volume of the solution of Trilon B concentration of 0.025 mol / dm³ spent on titration of blank test, cm³;

V₃ - the total volume of the sample solution, cm³;

V₄ - the volume of an aliquot of the sample solution, cm³;

m - mass of the sample, g;

x - mass fraction of loss on ignition (§ 3.4.),%.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.05%.

The total relative error analysis ± 2% at a confidence level of P = 0.95.

3.12. Determination of the mass fraction of sulfur oxide (gravimetric method)

3.12.1. The essence of the method

The method is based on the precipitation of sulfate ion as barium sulfuric acid in weakly acidic solution of barium chloride.

3.12.2. Apparatus, reagents, solutions.

The crucible of platinum number 100-8 and the cover to the crucible of platinum number 101-8 in accordance with GOST 6563.

Laboratory muffle furnace with a heating temperature of at least 950 ° C.

Sodium carbonate anhydrous 83 according to GOST, x. h., with the mass fraction of 1% solution.

Rectified ethyl alcohol Technical GOST 18300 premium.

Barium chloride according to GOST 4108, with mass fraction of 1% solution.

Distilled water in accordance with GOST 6709.

Hydrochloric acid in accordance with GOST 3118, x. h., the molar concentration of solution (HCl) = 2 mol / dm³.

Sulphuric acid in accordance with GOST 4204, x. h, diluted (1: 1)..

Acid hydrofluoric according to GOST 10484, s. h.

Silver nitrate according to GOST 1277, with mass fraction of 1% solution.

Methyl orange TU 6-09-5171, preparation of the solution in accordance with GOST 4919.1.

3.12.3. analysis

1.0-1.1 g of sodium silicate was weighed in a platinum crucible, mixed with 3 g of anhydrous sodium carbonate, and the lid is closed crucible is fused in an oven at a temperature of 900-950 ° C. The cooled alloy was treated with small portions of hot water to 100 cm³, the solution was transferred into a 400 cm³ beaker. Glass, covered with a watch glass, put a hot water bath to lighten the supernatant. Thereafter the watch glass is removed, the liquid is filtered-settled at 300-400 cm³ beaker, decanted 3 times with hot sodium carbonate solution with a mass fraction of 1%, is then transferred to a filter and the precipitate was washed 2-3 times. You can also use the filtrate after separation of the silicon dioxide in accordance with paragraph. 3.5.3, or 100 cm³ of the solution in accordance with para. 3.6.4, from which silica is determined photocolometry. The filtrate was diluted to 200 cm³ of water are added 3-5 drops of methyl orange, carefully neutralized with hydrochloric acid until the solution changes color to yellow, red, and then excess give 5-7 drops of hydrochloric acid.

The operation of the neutralization must be carried out at constant stirring from time to time covering the glass of a watch glass.

Slightly acidic solution is heated to boiling and boiled for 5-10 minutes with constant stirring, surging 20 cm³ hot solution of barium chloride, and left in a boiling water bath in a beaker covered with a watch glass for two hours and for 12 hours at room temperature. Sulfuric acid precipitate barium filtered

through a double filter (blue ribbon) and washed with hot water until the washing waters in the absence of chlorine ion (test with silver nitrate). Filter the precipitate was placed in a weighed and precalcined platinum crucible, gently dried, ashed, calcined in a muffle furnace at a temperature of 30-40 min 800-850 ° C to constant weight.

The resulting precipitate was treated with 1-2 drops of sulfuric acid solution and 5-6 drops of hydrofluoric acid. The mixture was evaporated to dryness on the air bath. Solids again calcined in a muffle oven for 30-40 minutes at a temperature 800-850 ° C and after cooling the crucible in a desiccator with sulfuric acid precipitate barium weighed.

3.12.4. Processing of results

Mass fraction of sulfur oxide (X7) in percentage is calculated by the formula

where m - the mass of the sample, g;

m_1 - mass of sediment sulfur-sour barium, g;

x - mass fraction of loss on ignition (§ 3.4.);

0.343 - weight conversion factor of sulfur-sulfur sour barium oxide.

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.05%.

The total relative error in the analysis of $\pm 10\%$ at a confidence level of $P = 0.95$.

3.13. Determination of the mass fraction of sulfur oxide (VI) (photocolorimetric method)

3.13.1. The essence of the method

The method is based on the measurement of opalescence sulfur-sour barium formed during the interaction of barium ions with sulfate ions.

3.13.2. The reagents, solutions, equipment

Photoelectric colorimeter of normative and technical documentation, or a spectrophotometer.

Sodium-sulfur sour anhydrous according to GOST 4166.

Standard solution containing sulfate ion, is prepared in accordance with GOST 4166. Before applying the appropriate dilution, a solution of 0.1 mg / cm³.

Barium chloride according to GOST 4108.

Glycerol.

Hydrochloric acid in accordance with GOST 3118.

Sodium chloride in accordance with GOST 4233.

A component reagent is prepared as follows:

60 g of sodium chloride and 25 g of barium chloride dissolved in 300 cm³ of water and 300 cm³ of glycerol is added 5 cm³ of hydrochloric acid and stirred well.

3.13.3. analysis

The volumetric flask was placed 100 cm³ 10 cm³ reagent solution and the composite is brought to the label after separation of the filtrate silicic acid in accordance with clause 3.5.3 or 3.6.4. At the same time prepare a comparison solution containing no sulfate ion.

After 30 minutes, the optical density of the sample solution with respect to the reference solution photoelectrocolorimeter using a blue filter with wavelengths $\lambda = 480-490$ nm cuvettes with 30 mm thickness. Upon receipt of the value of optical density, using the calibration curve, find the content of sulfate ion in milligrams.

3.13.4. Building a graded schedule

The volumetric flasks were placed 100 cm³ of solutions containing 0.1; 0.2; 0.3; 0.4; 0.5 mg of sulfate ion, distilled water was adjusted to 25 volumes of solutions and stirred cm³.

At the same time prepare a comparison solution containing no sulfate ion. To each solution is added 10 cm³ of reactant compound, made up to the mark with water and mix.

The absorbance of the solutions is measured in accordance with para. 3.13.3. The data obtained are building the calibration curve.

3.13.5. Processing of results

Mass fraction of sulfur oxide (VI) (X7) in percentage is calculated by the formula where m_1 - mass of the ion sulfate found on the calibration curve, mg;

0,833 - recalculation coefficient sulfate ion to the sulfur oxide (VI);

V - total volume of the solution see "

V₁ - the volume of an aliquot of the sample solution taken for fotokolorimetrirovaniya cm³;

m - mass of the sample, g

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.05%.

The total relative error of the method $\pm 10\%$ at a confidence level of $P = 0.95$.

When differences in the evaluation of the mass fraction of sulfur oxide analysis was performed by gravimetric method.

3.14. Determination of the mass fraction of sodium oxide (acidimetric method)

3.14.1 The essence of the method

The method is based on acidimetric titration, obtained after repeated treatment with hot water of sodium silicate solution.

3.14.2. Apparatus, reagents, solutions

Hydrochloric acid according to GOST 311-8, x. h., the molar concentration of solution (HCl) = 0.1 mol / dm³.

Ethanol rektiftskovanny technical GOST 18300 premium.

Distilled water in accordance with GOST 6709.

Methyl orange TU 6-09-5171, the solution is prepared in accordance with GOST 4919.1.

Phenolphthalein TU 6-09-5360, the solution is prepared in accordance with GOST 4919.1.

3.14.3. Analysis

Weigh 0,19-0,21 g sodium silicate, is placed in a beaker of 150-200 cm³, treated with 25 cm³ of water, heated to boiling and boiling water for 2-3 minutes, allowed to settle. After that the clear solution is poured into a conical flask of 250 cm³.

Remaining in the glass again treated precipitate to completely dissolve small portions of 10 cm³ of hot water. Heated again and the clear solution is poured into the same Erlenmeyer flask. The completeness of the dissolution of the mass sample is checked to phenolphthalein.

After cooling, the liquid in the flask is titrated with a solution of hydrochloric acid in the presence of 3-4 drops of methyl orange to yellow color transition in pale pink.

3.14.4. Processing of results

The weight proportion of sodium oxide (X₈) is calculated as a percentage by the formula where V - volume of hydrochloric acid solution with a molar concentration (HCl) = 0.1 mol / dm³, used for titration of consumed cm³;

m - mass of sample, g;

x - mass fraction of loss on ignition (§ 3.4.);

0.0031 - weight of sodium oxide, corresponding to 1 cm³ of hydrochloric acid molar concentration of exactly 0.1 mol / dm³ of

For the analysis result is the arithmetic average of the results of two parallel determinations, the absolute difference between them does not exceed the allowable of 0.4%.

The total relative error analysis ± 5% at a confidence level of P = 0.95.

3.15. Determination of silicate module

Silicate module - the ratio of the silica moles to the number of moles of sodium oxide.

Lime module (X₉) is calculated according to the formula,

Where in X₁ - mass fraction of silicon dioxide (. paragraphs 3.5, 3.6 or 3.7);

X₈ - mass fraction of sodium oxide (paragraph 3.10.);

1.0323 - molecular weight ratio of sodium oxide to the molecular weight of the silica.

4. TRANSPORTATION AND STORAGE

- 4.1. Sodium silicate is transported in bulk without packaging.
- 4.2. Sodium silicate is transported by all modes of transport in covered vehicles in accordance with goods transportation rules, operating in this mode of transport and technical conditions of loading and securing of cargo. Upon request of sodium silicate is transported in gondola cars and on the platforms.
- 4.3. Sodium silicate should be kept in closed spaces with hard flooring.

5. GARRANTY

- 5.1. The manufacturer guarantees the sodium silicate with this standard under the conditions of transportation and storage.
- 5.2. Warranty period of storage of soluble sodium silicate - 3 months. from the date of manufacture.

INFORMATION INFORMATION

1. DEVELOPED AND INTRODUCED State "Soyuzstroyaterialov 'Association

DEVELOPERS

EV Sobolev, Cand. tehn. Sciences; VE Golubev, PhD. tehn. Sciences; EI Raevskaya, PhD. tehn. Sciences; EA Didenko, PhD. Chem. Sciences; RP Borisov

2 approved and put into force by the State Standard of Russia from 2.12.92 number 1523

3. The test period - 1999

The frequency of inspection - 5 years

4. INSTEAD GOST 13079-81