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JAPANESE INDUSTRIAL STANDARD

**Methods for chemical
analysis of Soda-Lime-
Magnesia-Silica glasses**

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Methods for chemical analysis of
Soda-Lime-Magnesia-Silica glasses

R 3101-1991

1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of soda-lime-magnesia-silica glasses.

Remarks 1. The soda-lime-magnesia-silica glasses mean the glasses of which main components are silicon dioxide, sodium oxide, calcium oxide, and magnesium oxide, and including aluminium oxide, iron oxide, titanium dioxide, potassium oxide, sulfur trioxide, and arsenic oxide.

These methods are applicable to those including 2.0 % or under each of barium oxide, boron oxide and fluorine, other than the components given above.

2. Applicable Standards to this Standard are shown in the following:

JIS K 0050 General Rules for Chemical Analysis

JIS K 0115 General Rules for Molecular Absorptiometric Analysis

JIS K 0121 General Rules for Atomic Absorption Spectrochemical Analysis

JIS K 8001 General Rules of Testing Methods for Reagents

JIS K 8005 Standard Substances for Volumetric Analysis

JIS K 8121 Potassium Chloride

JIS K 8150 Sodium Chloride

JIS K 8885 Silicon Dioxide

JIS Z 8401 Rules for Rounding off of Numerical Values

JIS Z 8801 Test Sieves

2. General matters

General matters in common with the methods for chemical analysis shall be in accordance with JIS K 0050, JIS K 0115, and JIS K 0121.

3. Analytical items

The analytical items specified in this Standard shall be as given in the following:

Silicon dioxide (SiO_2)Aluminium oxide (Al_2O_3)

Total iron content (as Fe_2O_3)

Titanium dioxide (TiO_2)

Calcium oxide (CaO)

Magnesium oxide (MgO)

Sodium oxide (Na_2O)

Potassium oxide (K_2O)

Sulfur trioxide (SO_3)

Total arsenic content (as As_2O_3)

4. Sampling method and handling method

4.1 The analytical sample shall be weighed out from those which have been pulverized so that the total amount can pass through 250 μm sieve of JIS Z 8801, dried for about 2 h in an air bath of 105 to 110°C, and immediately after leaving cooled in a desiccator.

4.2 In weighing out the analytical sample, care shall be taken so that it presents the mean composition by mixing well with stirring, and confirmation shall be made so that no foreign substance is intermingled.

4.3 The sample shall be weighed out correctly to the nearest 0.1 mg, by using a chemical balance.

Remarks: Since the powders of soda-lime-magnesia-silica glass absorb moisture, carbon dioxide and others during pulverization and preservation, a necessary amount for analysis shall be pulverized on each occasion.

Furthermore, in the case where accurate results are required, correction shall be made with comparing the ignition losses at 1000°C of the pulverized sample and non-pulverized sample.

5. Arrangement of analysis value

5.1 Analysis shall generally be carried out twice or more on the same sample, and when the difference in results is greater than the numerical value given in the permissible deviation (%) of Table 1, retest shall be carried out, and the mean value of those of within permissible deviation shall be obtained.

Table 1. Permissible deviation on analysis values (%)

SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3	As_2O_3
0.3	0.05	0.006	0.003	0.10	0.10	0.10 ⁽¹⁾ 0.30 ⁽²⁾	0.05 ⁽¹⁾ 0.05 ⁽²⁾	0.03	0.03

Notes ⁽¹⁾ In the case of gravimetric method

⁽²⁾ In the case of flame analysis and atomic absorption spectro-chemical analysis.

5.2 In the analysis, contents (%) shall be corrected with carrying out blank tests throughout the total procedures.

5.3 The analytical results shall be expressed in percentage, and be rounded off as given in the following in accordance with JIS Z 8401.

- (1) For silicon dioxide, one place of decimal
- (2) For total iron content and titanium dioxide, three places of decimals
- (3) For other components excluding silicon dioxide, total iron content and titanium dioxide, two places of decimals

6. Methods for determination of silicon dioxide

6.1 Division of methods The methods for determination of silicon dioxide shall be in accordance with either one of the following:

- (1) Combined method of dehydration-gravimetric method and absorptiometry
- (2) Combined method of aggregation-gravimetric method and absorptiometry

6.2 Combined method of dehydration-gravimetric method and absorptiometry

6.2.1 Summary The sample is melted with sodium carbonate, and dissolved in hydrochloric acid, and after silicic acid has been dehydrated by evaporation to dryness, soluble salts are dissolved with hydrochloric acid and filtered. The precipitates are ignited and weighed, and after silicon dioxide has been volatilized by adding hydrofluoric acid, ignited again and weighed, and the main amount of silicon dioxide is obtained from the ignition loss. The filtrate is taken separately and the residual amount of silicon dioxide is obtained by molybdate blue absorptiometry. The sum of both is considered to be the total amount of silicon dioxide.

6.2.2 Reagents The reagents shall be in accordance with the following:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1, 1 + 4, 1 + 50)
- (3) Hydrofluoric acid (46 %)
- (4) Hydrofluoric acid (1 + 9) That has adsorption-eliminated of silico hydrofluoride passing through Cl type strong basic anion exchange column (plastic-made chromato tube) is used.
- (5) Sulfuric acid (1 + 1)
- (6) Boric acid solution (4 w/v %)
- (7) Sodium carbonate (anhydride)
- (8) Ammonium molybdate solution Dissolve 10 g of ammonium molybdate (tetrahydrate) with water to make up to 100 ml. If required, filter, and preserve in a plastic bottle. When any molybic acid has precipitated during preservation, prepare newly.

- (9) Tartaric acid solution Dissolve 10 g of tartaric acid with water to make up to 100 ml, and preserve in a plastic bottle.
- (10) Reducing agent solution Ascorbic acid solution (5 w/v %). Preserve this in a plastic bottle at a cool and dark place. That has lapsed one month or over after preparation should preferably not be used.
- (11) Standard silicon dioxide solution (0.05 mg SiO₂/ml) Ignite anhydrous silicic acid made by precipitation (JIS K 8885), after cooling weigh out 0.100 g into a platinum crucible, and after it has been mixed with 1 g of sodium carbonate (anhydrous), heat to fuse. After cooling dissolve with water and transfer into a one mark volumetric flask of 1000 ml, dilute up to the marked line and preserve in a plastic bottle. Dilute to two times correctly with water on each occasion of use.

6.2.3 Mass of sample to be weighed out For the sample, a mass of 0.50 g shall be weighed out.

6.2.4 Operation The determining operation shall be carried out in accordance with the following procedures:

- (1) Weigh out the sample in a platinum dish (for example, No. 75). After this has been mixed with 1.5 g of sodium carbonate (anhydride), at first heat at a low temperature, and increase temperature gradually, and ignite to approximately 1000°C to fuse.
- (2) After leaving cool with covered with a watch glass, add 20 ml of hydrochloric acid (1 + 1), heat to dissolve on a water bath, remove the watch glass by washing with small quantity of water, and successively evaporate to dryness. In the meantime, smash deposited salts to powder with a glass rod of which end has been flattened, from time to time.
- (3) After leaving cool, add 5 ml of hydrochloric acid, leave alone for approximately 1 min, add 20 ml of hot water and heat for approximately 5 min on a water bath to dissolve soluble salts.
- (4) Filter using filter paper (Class 5 B), wash several times with hot hydrochloric acid (1 + 50), and further wash sufficiently with hot water. Receive the filtrate and washings in a beaker (300 ml), and use for the determination of the residual silicon dioxide.
- (5) Put the precipitate into a platinum crucible (for example, No. 30) together with filter paper, add one drop of sulfuric acid (1 + 1), and after the filter paper has been incinerated by heating at a low temperature, ignite for approximately 1 h at 1100 ± 50°C. After leaving cool in a desiccator, weigh the mass, and repeat ignition until it becomes constant mass.
- (6) Moisten impure silicon dioxide with water, add 3 drops of sulfuric acid (1 + 1) and approximately 10 ml of hydrofluoric acid, heat on a sand bath, and evaporate to dryness. Ignite at 1100 ± 50°C for approximately 5 min, and weigh the mass after cooling in a desiccator.

- (7) After cooling, transfer the filtrate and washings of (4) into a one mark volumetric flask of 250 ml, and dilute up to the marked line with water. Separately take 10 ml of this solution correctly into a plastic beaker (100 ml).
- (8) Add 2 ml of hydrofluoric acid (1 + 9), and after this has been left alone for approximately 10 min, add 50 ml of boric acid solution.
- (9) Add 2 ml of ammonium molybdate solution and mix by stirring, and leave alone for 10 min. After 5 ml of tartaric acid solution has been added, add 2 ml of ascorbic acid solution, transfer into a one mark volumetric flask of 100 ml, dilute up to the marked line with water, and leave alone for 30 min.
- (10) Take a part of this solution into the absorption cell of a photometer, and measure absorbance at a wavelength of about 650 nm.

6.2.5 Preparation of working curve Take 0 to 6 ml of standard silicon dioxide solution (0 to 0.3 mg as silicon dioxide) stepwise into plastic beakers (100 ml), add 1 ml of hydrochloric acid (1 + 4), hereafter operate in accordance with 6.2.4 (8) to (10), prepare the relationship curve between the absorbances and the amounts of silicon dioxide, and consider this to be the working curve.

6.2.6 Calculation Obtain the amount of residual silicon dioxide from the working curve which has been prepared in 6.2.5, and calculate the content of silicon dioxide in the sample in accordance with the following formula:

$$\text{Silicon dioxide (\%)} = \frac{(w_1 - w_2) + a \times \frac{250}{10}}{W} \times 100$$

where, w_1 : mass of 6.2.4 (5) (g)

w_2 : mass of 6.2.4 (6) (g)

a : amount of residual silicon dioxide of 6.2.4 (10) (g)

W : weighed out mass of sample of 6.2.3 (g)

6.3 Combined method of aggregation-gravimetric method and absorptiometry

6.3.1 Summary The sample is melted with sodium carbonate and boric acid, and is dissolved with hydrochloric acid, and after silicic acid has been aggregated by adding polyethyleneoxide, subjected to filtration. The precipitates are ignited and weighed, and next, silicon dioxide has been volatilized by adding hydrofluoric acid, this is ignited again and weighed, and the main amount of silicon dioxide is obtained from its ignition loss. Then the filtrate is taken separately to obtain the residual amount of silicon dioxide by means of the molybdate blue absorptiometry. The sum of both is considered to be the total amount of silicon dioxide.

6.3.2 Reagents The reagents shall be in accordance with the following:

- (1) Boric acid
- (2) Polyethylene-oxide solution (0.05 %) Add 0.1 g of polyethyleneoxide into 200 ml of water, bit by bit, while stirring to dissolve, and preserve in a plastic bottle. That which has lapsed two weeks after preparation should preferably not be used.
- (3) Other reagents used shall be the same as 6.2.2 (2) to (11).

6.3.3 Mass of sample to be weighed out For the sample, a mass of 0.5 g shall be weighed out.

6.3.4 Operation The determining operation shall be carried out in accordance with the following procedures:

- (1) Weigh out the sample into a platinum dish (for example, No. 75), and after this has been mixed with 1.5 g of the sodium carbonate (anhydride) and 0.2 g of the boric acid, heat at a low temperature at first, and increase temperature gradually, and ignite at about 1000°C to melt ⁽³⁾.

Note ⁽³⁾ Stop heating when the melt has become transparent. Too long melting time leads to cause difficulty to be melted with hydrochloric acid.

- (2) Cover with a watch glass, and after leaving cool, add 20 ml of hydrochloric acid (1 + 1), and after this has been heated to dissolve on a water bath, continue heating for about 20 min.
- (3) Remove the watch glass without washing with water ⁽⁴⁾, add a small amount of powdered filter paper and after it has been stirred well to mix, add 10 ml of polyethylene oxide solution and stir well to mix, and leave alone for 5 min.

Note ⁽⁴⁾ Washing of the watch glass is carried out at the time of operation ⁽⁴⁾.

- (4) Filter using filter paper (Class 5 B), and after this has been washed several times with hot hydrochloric acid (1 + 50), wash with hot water sufficiently. Receive the filtrate and washings into a beaker (300 ml), and use for determination of the residual silicon dioxide.
- (5) Put the precipitates into a platinum crucible (for example, No. 30) together with the filter paper, add one drop of sulfuric acid (1 + 1), and after the filter paper has been incinerated by heating at a low temperature, ignite at 1100 ± 50°C for about 1 h. After leaving cool in a desiccator weigh the mass, and repeat ignition until it becomes constant mass.
- (6) Moisten the impure silicon dioxide with water, add 3 drops of sulfuric acid (1 + 1) and approximately 10 ml of hydrofluoric acid, heat on a sand bath and evaporate to dryness. Ignite at 1100 ± 50°C for 5 min, and after leaving cool in a desiccator, weigh the mass.

- (7) After cooling, transfer the filtrate and washings of (4) into a one mark volumetric flask of 250 ml, and dilute up to the marked line with water. Separately take 10 ml of this solution exactly into a plastic beaker (100 ml), and operate in accordance with 6.2.4 (8) to (10).

6.3.5 Preparation of working curve Operate in the same manner as 6.2.5.

6.3.6 Calculation Obtain the amount of residual silicon dioxide from the working curve which has been prepared in 6.3.5, and calculate the content of silicon dioxide in the sample in accordance with the following formula:

$$\text{Silicon dioxide (\%)} = \frac{(w_1 - w_2) + a \times \frac{250}{10}}{W} \times 100$$

where, w_1 : mass of 6.3.4 (5) (g)

w_2 : mass of 6.3.4 (6) (g)

a : residual amount of silicon dioxide of 6.3.4 (7) (g)

W : weighed out mass of sample of 6.3.3 (g)

7. Method for determination of aluminium oxide

7.1 Division of methods The method for determination of aluminium oxide shall be in accordance with EDTA titration.

7.2 EDTA titration

7.2.1 Summary The sample is heated and decomposed with hydrofluoric acid and sulfuric acid, and after this has been evaporated to dryness, allowed to be constant volume by dissolving with hydrochloric acid. A part of this is taken out, to this an excessive amount of EDTA solution is added, pH is adjusted to 5.5, aluminium-EDTA chelate is allowed to complete by boiling, and after cooling, titration is carried out with zinc standard solution using xylenol orange as indicator. Next, sodium fluoride is added and is boiled, and after cooling, the EDTA equivalent to substitution-isolated aluminium and titanium is titrated with zinc standard solution. Then the amount of aluminium oxide is calculated by subtracting the amount of titanium dioxide.

7.2.2 Reagent The reagents shall be in accordance with the following:

- (1) Hydrochloric acid (1 + 1)
- (2) Nitric acid
- (3) Hydrofluoric acid (46 %)
- (4) Sulfuric acid (1 + 1)
- (5) Aqueous ammonia (1 + 9)
- (6) Sodium fluoride

- (7) Buffer solution (pH 5.5) Dissolve 50 g of sodium acetate (trihydrate) with water, add 2.5 ml of acetic acid, and dilute to 1000 ml with water.
- (8) M/100 zinc standard solution Weigh out 0.66 g of zinc of standard reagent for volumetric determination (JIS K 8005) exactly to the nearest 0.1 mg, add approximately 20 ml of water and 5 ml of nitric acid (1 + 1), heat to decompose on a water bath, after cooling transfer into a one mark volumetric flask of 1000 ml, and dilute up to the marked line with water. Preserve in a plastic bottle. The amount of aluminium oxide per 1 ml of this solution is to be calculated from the following formula:

$$f = \frac{G \times P}{0.6537 \times 100} \times 0.0005098$$

where, f : the amount of aluminium oxide per 1 ml of M/100 zinc standard solution (g)

G : weighed out mass of zinc (g)

P : purity of zinc (%)

- (9) EDTA solution (M/100) Dissolve 3.73 g of disodium ethylenediaminetetraacetate (dihydrate) with water to make up to 1000 ml, and preserve in a plastic bottle.
- (10) Xylenol orange solution (0.1 w/v %) Dissolve 0.1 g of 3, 3-bis [N, N-di-(carboxymethyl)-aminomethyl]-*o*-cresol sulfophthalein with 100 ml of water, put into a brown bottle and preserve in a cool and dark place. Prepare newly on each 1 to 2 months.
- (11) Methyl orange solution (0.1 w/v %)

7.2.3 Mass of sample to be weighed out For the sample, a mass of 1.0 g shall be weighed out.

7.2.4 Operation The determining operation shall be carried out in accordance with the following procedures:

- (1) Weigh out the sample in a platinum dish (for example, No. 75), moisten with approximately 1 ml of water, add 4 ml of sulfuric acid (1 + 1) and 10 ml of hydrofluoric acid, stir well to mix with a platinum wire or the like, heat to decompose on a sand bath carefully, and heat to evaporate until white fume of sulfuric acid begins to generate.
- (2) After leaving cool, wash inner wall with a small amount of water, add 1 ml of nitric acid and 5 ml of hydrofluoric acid, and after this has been stirred well to mix, heat to evaporate on the sand bath again, and allow to generate white fume of sulfuric acid for about 5 min.
- (3) After leaving cool, wash inner wall with a small amount of water, heat on the sand bath again, and evaporate to dryness.