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Methods for chemical analysis of borosilicate glasses

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

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Methods for chemical analysis of borosilicate glasses

1 Scope This Japanese Industrial Standard specifies methods for chemical analysis of borosilicate glasses.

Remarks 1 The borosilicate glasses specified in this Standard mean the glasses made of silicon dioxide, boron trioxide, aluminium oxide and sodium oxide as principal components, used for physical and chemical experimental appliances, cooking appliances, shield-beam type electric lamp for automobiles, etc.

2 The normative references are as follows.

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 rule for test methods of reagents*

JIS K 8005 *Reference materials for volumetric analysis*

JIS K 8885 *Silicon dioxide*

JIS Z 8401 *Rules for rounding off of numerical values*

2 General matters General matters common to chemical analysis methods shall be in accordance with JIS K 0050, JIS K 0115, JIS K 0121 and JIS K 8001.

3 Analysis items Analysis items specified in this Standard shall be as follows:

- (1) Silicon dioxide (SiO_2)
- (2) Aluminium oxide (Al_2O_3)
- (3) Total iron content (as Fe_2O_3)
- (4) Titanium dioxide (TiO_2)
- (5) Zirconium oxide (ZrO_2)
- (6) Sodium oxide (Na_2O)
- (7) Potassium oxide (K_2O)
- (8) Boron trioxide (B_2O_3)
- (9) Chlorine (Cl)
- (10) Total arsenic content (as As_2O_3)

4 Methods for sampling and treating of sample

4.1 A necessary quantity of analysis sample shall be quickly pulverized at each analysis

and reduced to 250 μm max. Immediately thereafter, it shall be weighed⁽¹⁾.

Note ⁽¹⁾ When precise results are required, the loss of weight due to ignition of the pulverized sample is to be compared to the loss of weight due to ignition of the non-pulverized sample.

4.2 When the analysis sample is weighed, the sample shall be carefully and sufficiently agitated to present an average composition. Further, it shall be confirmed that foreign matters are not mixed in.

4.3 For weighing the analysis sample, a chemical balance shall be used and the sample shall be accurately weighed to the nearest 0.1 mg.

5 Organizing methods of analysis values

5.1 The analysis shall, as a rule, be done twice repeatedly on the same sample. When their difference is larger than the values given in Table 1, the sample shall be analyzed again.

Table 1 Tolerances of analysis value (%)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	ZrO ₂	Na ₂ O	K ₂ O	B ₂ O ₃	Cl	As ₂ O ₃
0.17	0.05	0.003	0.003	0.005	0.08	0.007 ⁽²⁾ 0.030 ⁽³⁾	0.18 ⁽⁴⁾ 0.15 ⁽⁵⁾	0.005	0.03 ⁽³⁾

Notes ⁽²⁾ The tolerance in the case of the atomic absorption method.

⁽³⁾ The tolerance in the case of the flame method.

⁽⁴⁾ The tolerance in the case of the direct method.

⁽⁵⁾ The tolerance in the case of the silicon dioxide filtrate method.

5.2 For analysis, the blank test shall be carried out through all operations, and the content rate shall be corrected.

5.3 The analysis result shall be expressed in percentages and the average of 5.1 shall be rounded off as follows in accordance with JIS Z 8401.

- (1) The average values for silicon dioxide and boron trioxide shall be rounded off to one decimal place.
- (2) The average values for aluminium oxide and sodium oxide shall be rounded off to two decimal places.
- (3) The average values for other components except for silicon dioxide, boron trioxide, aluminium oxide and sodium oxide shall be rounded off to three decimal places. However, for potassium oxide and total arsenic content, the average values shall be rounded off to two significant digits.

6 Method for determination of silicon dioxide

6.1 Division of methods The method for determination of silicon dioxide shall be in accordance with aggregation and gravimetric absorptiometry combined method.

6.2 Aggregation and gravimetric absorptiometry combined method

6.2.1 Summary A sample is fused with sodium carbonate, dissolved in hydrochloric acid. Silicic acid is aggregated by adding polyethylene oxide. Thereafter, it is filtered. The precipitate is ignited and weighed. Then, hydrofluoric acid is added to it and silicon dioxide is volatilized. Thereafter, it is ignited and weighed again. The amount of silicon dioxide is obtained from its loss. A portion of the filtrate is taken and the residual silicon dioxide amount is determined by molybdate blue absorptiometry. The sum of both amounts shall be taken as the total amount of silicon dioxide.

6.2.2 Reagents The reagents shall be as follows:

- (1) **Hydrochloric acid (1+1, 1+4, 1+50)**
- (2) **Hydrofluoric acid (46 mass %)**
- (3) **Hydrofluoric acid (1+9)** Hydrofluoric acid from which hydrosilicofluoric acid is removed by adsorption by passing it through Cl-type strong basic anion exchange column (plastics chromato-column) is used.
- (4) **Sulfuric acid (1+1)**
- (5) **Boric acid solution (40 g/L)**
- (6) **Sodium carbonate (anhydrous)**
- (7) **Ammonium molybdate solution** 10 g of ammonium molybdate (tetrahydrate) is dissolved with water and diluted to 100 ml. If necessary, it is filtered and stored in a polyethylene bottle. If molybdic acid is deposited in the bottle during storage, the solution shall be newly prepared.
- (8) **Tartaric acid solution (100 g/L)** It is stored in a polyethylene bottle.
- (9) **Ascorbic acid solution (50 g/L)** It is preserved in a polyethylene bottle at a cold dark place. Those which one month or longer has elapsed after preparation shall not be used.
- (10) **Polyethylene oxide solution** 0.1 g of polyethylene oxide is added little by little to 200 ml water while mixing it by stirring to be dissolved in. It is stored in a polyethylene bottle. Those which two weeks have elapsed after preparation shall not be used.
- (11) **Silicon dioxide standard solution (0.5 mg SiO₂/ml)** Silicon dioxide specified in JIS K 8885 is ignited and allowed to cool in a desiccator. Thereafter, 0.500 g thereof is weighed into a platinum crucible and mixed with 1.5 g of sodium carbonate (anhydrous). Thereafter, they are fused by heating. After standing to cool, the melt is dissolved with water, transferred into a 1 000 ml volumetric flask, diluted with water to the marked line and stored in a polyethylene bottle.

6.2.3 Weighing amount of sample 0.50 g of the sample shall be weighed.

6.2.4 Operation The determination operation shall be carried out as follows:

- (1) Weigh the sample on a platinum evaporating dish (for example, No. 75) and mix it with 1.5 g of sodium carbonate (anhydrous). Thereafter, first heat them at a low temperature, gradually raising the temperature, and then fuse them by ignition at about 1 000 °C⁽⁶⁾.
- (2) Cover it with a watch glass, allow it to cool, add 20 ml of hydrochloric acid (1+1) and dissolve it by heating in a water bath. Thereafter, continue to heat it for about 20 min.
- (3) Remove the watch glass without washing it with water⁽⁷⁾, add about 0.05 g of powdery filter paper and sufficiently mix them by stirring. Thereafter, add 10 ml of polyethylene oxide solution, sufficiently mix them by stirring and let it stand for about 5 min.
- (4) Filter it by using a filter paper (Class 5 B) and wash it with hot hydrochloric acid (1+50) several times. Thereafter, sufficiently wash it with hot water. Receive the filtrate and the washings in a 300 ml beaker and use them for determination of residual silicon dioxide from step (7) on.
- (5) Put the precipitate into a platinum crucible (for example No. 30) together with the filter paper, add one drop of sulfuric acid (1+1) and incinerate the filter paper by heating at a low temperature. Thereafter, ignite it at $1\,100 \pm 50$ °C for about 1 h. After standing to cool in a desiccator, measure the mass and repeat the ignition until a constant weight is obtained.
- (6) Then, moisten it with water, add 3 drops of sulfuric acid (1+1) and about 10 ml of hydrofluoric acid (46 mass %), heat them on a sand bath and evaporate them to dryness. Ignite it at $1\,100 \pm 50$ °C for about 5 min and let it stand to cool in a desiccator. Thereafter, weigh the mass.
- (7) After cooling the filtrate and the washings of (4), transfer them into a 250 ml measuring flask and dilute it with water to the marked line. Accurately take 10 ml of this solution into a 100 ml polyethylene beaker. Store the remainder to use it for determination of boron trioxide.
- (8) Add 2 ml of hydrofluoric acid (1+9) and let it stand for about 10 min. Thereafter, add 50 ml of boric acid solution.
- (9) Add 2 ml of ammonium molybdate solution, stir it to mix and let it stand for about 10 min. After adding 5 ml of tartaric acid solution, add 2 ml of ascorbic acid solution, transfer it into a 100 ml measuring flask, dilute it with water to the marked line and let it stand for about 30 min.
- (10) Take a portion of this solution into the absorption cell of a photometer and measure the absorbance at near 650 nm in wavelength.

Notes ⁽⁶⁾ When the melt becomes transparent, heating is to be stopped because when the fusing time is too long, it becomes difficult to be dissolved with hydrochloric acid.

⁽⁷⁾ The watch glass is washed at the operation of (4).

6.2.5 Preparation of a working curve Accurately dilute silicon dioxide standard solution with water to ten times the volume, take stepwise 0 ml to 6 ml thereof (0 mg to 0.30 mg as silicon dioxide) into each 100 ml polyethylene beaker, add 1 ml of hydrochloric acid (1+4), follow the procedures in 6.2.4 (8) to (10) and prepare the relation curve between the absorbance and the amount of silicon dioxide to the working curve.

6.2.6 Calculation The residual silicon dioxide amount shall be obtained by the working curve prepared in 6.2.5 and the content of silicon dioxide in the sample shall be calculated from the following formula:

$$\text{SiO}_2 = \frac{(m_1 - m_2) + a \times \frac{250}{10}}{m} \times 100$$

where, SiO_2 : silicon dioxide content (mass %)

m_1 : mass of 6.2.4 (5) (g)

m_2 : mass of 6.2.4 (6) (g)

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

m : weighing amount of sample of 6.2.3 (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-zinc back titration method.

7.2 EDTA-zinc back titration method

7.2.1 Summary A sample is decomposed by heating with hydrofluoric acid and sulfuric acid and is evaporated to dryness. It is dissolved in hydrochloric acid at a constant volume. A portion thereof is taken, excess EDTA solution is added to it, with the pH regulated to 5.5, and aluminium-EDTA chelate is produced by boiling. After cooling, it is titrated with zinc standard solution by Xylenol orange as an indicator. Sodium fluoride is added, then they are boiled and cooled. Thereafter, EDTA corresponding to free aluminium, titanium and zirconium is titrated with zinc standard solution. The amount of aluminium oxide is calculated by subtracting the amounts of titanium dioxide and zirconium oxide therefrom.

7.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1+1)
- (2) Nitric acid (1+1)
- (3) Hydrofluoric acid (46 mass %)
- (4) Sulfuric acid (1+1)
- (5) Aqueous ammonia (1+9)
- (6) Sodium fluoride
- (7) Buffer solution (5.5 in pH) 50 g of sodium acetate (trihydrate) is dissolved with

water, added with 2.5 ml of acetic acid and diluted with water to 1 000 ml.

- (8) **0.01 mol/L zinc standard solution** Accurately weigh 0.66 g of zinc specified in JIS K 8005 to the nearest 0.1 mg, add to it about 20 ml of water and 5 ml of nitric acid (1+1) and decompose by heating on a water bath. After cooling, it is transferred into a 1 000 ml measuring flask, diluted with water to the marked line and stored in a polyethylene bottle. The amount of aluminium oxide per 1 ml of this solution is calculated from the following formula:

$$f = \frac{G \times P}{0.6539 \times 100} \times 0.0005098$$

where, f : aluminium oxide amount per 1 ml of 0.01 mol/L zinc standard solution (g)

G : weighing amount of zinc (g)

P : purity of zinc (mass %)

- (9) **EDTA solution (0.01 mol/L)** 3.73 g of disodium ethylenediaminetetraacetate (dihydrate) is dissolved with water to 1 000 ml and stored in a polyethylene bottle.
- (10) **Xylenol orange solution (1 g/L)** The sample preparation method and storing method are in accordance with 4.4 Table 8 of JIS K 8001.
- (11) **Methyl orange solution (1 g/L)**

7.2.3 Weighing amount of sample 1.0 g of sample shall be weighed.

7.2.4 Operation The determination operation shall be carried out as follows:

- (1) Weigh a sample onto a platinum evaporation dish (for example No. 75), moisten it with about 1 ml of water, add 5 ml of sulfuric acid (1+1) and 10 ml of hydrofluoric acid, sufficiently mix it by stirring with platinum wire or the like, carefully decompose it by heating on a sand bath and evaporate it by heating until white fumes of sulfuric acid start to be generated.
- (2) After standing to cool, add 5 ml of hydrofluoric acid and sufficiently mix them by stirring. Evaporate it by heating again on the sand bath and generate white fumes of sulfuric acid for about 5 min.
- (3) After standing to cool, wash the inside wall of the evaporation dish with a small amount of water, heat it again on the sand bath and evaporate it to dryness.
- (4) After standing to cool, add 20 ml of hydrochloric acid (1+1) and about 30 ml of water, mix them by stirring and dissolve soluble salts by heating. After cooling, transfer it into a 200 ml measuring flask and dilute it with water to the marked line.

This solution is used as sample solution (A) for determination of aluminium oxide, total iron content, titanium dioxide and zirconium oxide, and for determination of the sodium oxide and potassium oxide by flame method or atomic absorption method.

- (5) Accurately take 25 ml of the sample solution (A) into a 300 ml beaker, add 15 ml of EDTA solution, 100 ml of hot water and one drop of Methyl orange solution, drop aqueous ammonia (1+9) until the colour of solution turns orange, add 15 ml of buffer solution and boil it for 5 min.