

JIS

JAPANESE INDUSTRIAL STANDARD

**Methods for Chemical
Analysis of Feldspar**

JIS M 8853—1976

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

JAPANESE INDUSTRIAL STANDARD
Methods for Chemical Analysis of Feldspar

J I S
M 8853-1976
(Reaffirmed: 1990)

1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of feldspar for ceramic industry.

2. General Matters

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

3. Analytical Items

Analytical items specified in this Standard shall be as follows:

Ignition loss (Ig. loss)
 Silicon dioxide (SiO₂)
 Aluminium oxide (Al₂O₃)
 Iron (III) oxide (Fe₂O₃)
 Titanium dioxide (TiO₂)
 Sodium oxide (Na₂O)
 Potassium oxide (K₂O)

4. Sampling and Treating of Sample

4.1 For an analytical sample, carefully sufficiently mix by stirring the sample so as to represent the average composition and further, confirm non mixing of foreign matters.

4.2 For the analytical sample, spread about 5 g of the sample of which all mass has passed the 105 µm test sieve of JIS Z 8801, thinly on a flat weighing bottle (50 mm in diameter). After drying in a air bath at 105 to 110°C for 2 h, reserve in a desiccator, and weigh out therefrom.

4.3 For weighing out of the analytical sample, use a chemical balance, and weigh to the nearest 0.1 mg.

5. Arranging of Analytical Value

5.1 Perform analysis, as a rule, two or more times on the same sample. When the difference is larger than the numerical value shown by the tolerance (%) of Table 1, analyze again, and calculate the mean value of those within the tolerance.

Table 1. Tolerance on Analytical Value (%)

Chemical composition	Ig. loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	Na ₂ O	K ₂ O
Tolerance	0.08	0.3	0.15	0.006	0.003	0.14	0.25

Applicable Standards:

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 8005-Standard Substances for Volumetric Analysis
 JIS K 8885-Silicon Dioxide
 JIS Z 8401-Rules for Rounding off of Numerical Values
 JIS Z 8801-Test Sieves

5.2 On analysis, perform a blank test over all the operation, and correct the content.

5.3 Express the analytical result by percentage and round off in accordance with JIS Z 8401 as follows:

- (1) To one place of decimal for silicon dioxide.
- (2) To three places of decimals for iron (III) oxide and titanium dioxide.
- (3) To two places of decimals for other components excepting above-mentioned three components.

6. Method for Determination of Ignition Loss

6.1 Classification of Method The method for determination of ignition loss shall be in accordance with a gravimetric method.

6.2 Gravimetric Method

6.2.1 Summary Weigh the loss when a sample is ignited at 1000 to 1050°C to constant mass.

6.2.2 Weighing out Amount of Sample Weigh out 1 g of the sample.

6.2.3 Operation Carry out the determining operation in accordance with the following procedures:

- (1) Weigh out the sample into a platinum crucible (for instance, number 30), cover it with opening a gap slightly, heat initially at a low temperature, raise gradually the temperature, and ignite lastly in an electric furnace at 1000 to 1050°C for about 30 min. After standing to cool in a desiccator, weigh the mass.
- (2) Repeat ignition for each 15 min, and obtain the loss when the sample becomes constant in mass.

6.2.4 Calculation Calculate the ignition loss in the sample in accordance with the following formula.

$$\text{Ignition loss (\%)} = \frac{w}{W} \times 100$$

where, w : loss (g)

W : weighed out amount of sample (g)

7. Method for Determination of Silicon Dioxide

7.1 Classification of Methods The method for determination of silicon dioxide shall be in accordance with either one of the following methods.

- (1) Dehydrating gravimetry absorptiometry joint method
- (2) Flocculation gravimetry absorptiometry joint method

7.2 Dehydrating Gravimetry Absorptiometry Joint Method

7.2.1 Summary Fuse a sample with sodium carbonate, dissolve in hydrochloric acid, and dehydrate silicic acid by evaporation to dryness. Thereafter, dissolve soluble salts in hydrochloric acid, and filter. Ignite the precipitate, weigh, and add hydrofluoric acid. After volatilizing silicon dioxide, ignite again, weigh, and obtain the amount of principal silicon dioxide from its loss. Separately take the filtrate, and obtain the amount of residual silicon dioxide by a molybdenum blue absorptiometry. Take the sum of both as total amount of silicon dioxide.

7.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1, 1 + 4, 1 + 50)
- (3) Hydrofluoric acid (46 %)

- (4) Hydrofluoric acid (1 + 9) Hydrofluoric acid from which hydrosilicofluoride is removed by adsorption by passing through a C 1 type strong basic ion exchange column (a plastic pipe is used as the chromatopipe). Reserve in a plastic bottle.
- (5) Sulfuric acid (1 + 1)
- (6) Boric acid solution (4 w/v %) Reserve in a plastic bottle.
- (7) Sodium carbonate (anhydride)
- (8) Ammonium Molybdate Solution Dissolve 10 g of ammonium molybdate (tetrahydrate) in water to make 100 ml. Filter as occasion demands, and reserve in a plastic bottle. However, when molybdic acid is deposited during reservation, prepare freshly.
- (9) Tartaric acid solution Dissolve 10 g of tartaric acid in water to make 100 ml, and reserve in a plastic bottle.
- (10) Ascorbic acid solution (5 w/v %) Put in a plastic bottle, and reserve in a cold dark place. It is preferable not to use the ascorbic acid solution for which one month has elapsed after preparation.
- (11) Standard silicon dioxide solution (0.05 mg SiO₂/ml) Ignite anhydrous silicic acid (sedimentating) (JIS K 8885), and allow to stand to cool in a desiccator. Weigh out 0.100 g therefrom into a platinum crucible, mix with 1 g of sodium carbonate (anhydride), and fuse by heating. After standing to cool, dissolve in water, transfer to a 1000 ml measuring flask, dilute up to the marked line with water, and reserve in a plastic bottle (0.1 mg SiO₂/ml). Dilute accurately two times with water each service.

7.2.3 Weighing out Amount of Sample Weigh out 0.50 g of the sample.

7.2.4 Operation Carry out the determining operation in accordance with the following procedures:

- (1) Weigh out a sample onto a platinum dish (for instance, number 75). After mixing with 1.5 g of sodium carbonate (anhydride), heat initially at a low temperature, raise gradually the temperature, and fuse by igniting at about 1100°C.
- (2) Cover with a watch glass. After standing to cool, add 20 ml of hydrochloric acid (1 + 1), dissolve by heating on a water bath, wash the watch glass, remove, and successively dry up by evaporation. Crush deposited salts with a glass rod of which a top is flattened occasionally during that time, and reduce to powder.
- (3) After standing to cool, add 5 ml of hydrochloric acid, allow to stand for about 1 min, add 20 ml of hot water, and dissolve soluble salts by heating on a water bath for about 5 min.
- (4) Add a suitable amount of powdery filter paper. After mixing by stirring, filter by using filter paper (class 5 B), wash several times with hot hydrochloric acid (1 + 50) and further, sufficiently wash with hot water. Receive the filtrate and washings with a beaker (300 ml), and reserve it for determination of residual silicon dioxide.
- (5) Put the precipitate together with the filter paper into a platinum crucible (for instance, number 30), add one drop of sulfuric acid (1 + 1), incinerate the paper by initially heating at a low temperature, and ignite at 1100 ± 50°C for about 1 h. After standing to cool in a desiccator, weigh the mass, and repeat ignition to constant mass.
- (6) Moisten impure silicon dioxide with water, add three drops of sulfuric acid (1 + 1) and about 10 ml of hydrofluoric acid, and evaporate to dryness by heating on a sand bath. Ignite at 1100 ± 50°C for about 5 min. After standing to cool in a desiccator, weigh the mass.
- (7) Transfer the filtrate and washings of (4) to a 250 ml measuring flask after cooling, dilute up to the marked line with water, and separately take accurately 10 ml of this solution into a plastic beaker (100 ml).

- (8) Add 2 ml of hydrofluoric acid (1 + 9). After standing to cool for about 10 min, add 50 ml of boric acid solution, and dilute to about 70 ml with water.
- (9) Add 2 ml of ammonium molybdate solution, mix by stirring, and allow to stand for 10 min. After adding 5 ml of tartaric acid solution, add 2 ml of ascorbic acid solution, transfer to a 100 ml measuring flask, dilute up to the marked line with water, and allow to stand for 30 min.
- (10) Take a portion of this solution into the absorption cell of a photometer, and measure the absorbance near 650 nm in wavelength.

7.2.5 Calculation Obtain the amount of residual silicon dioxide from the working curve prepared in 7.2.6, and calculate the content of silicon dioxide in the sample according to the following formula.

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

where, w_1 : mass weighed in 7.2.4 (5) (g)
 w_2 : mass weighed in 7.2.4 (6) (g)
 a : detection amount of residual silicon dioxide of 7.2.4 (10) (g)
 W : weighed out amount of sample (g)

7.2.6 Preparation of Working Curve Deal out stepwise 0 to 6.0 ml of standard silicon dioxide solution (0 to 0.30 mg as silicon dioxide) into a plastic beaker (100 ml), add 1 ml of hydrochloric acid (1 + 4), hereafter operate in accordance with the procedures of 7.2.4 (8) to (10), and prepare the relation curve between the obtained absorbances and the amounts of silicon dioxide to be taken as the working curve.

7.3 Flocculation Gravimetry Absorptiometry Joint Method

7.3.1 Summary Fuse a sample with sodium carbonate and boric acid, dissolve in hydrochloric acid, and add polyethylene oxide. After flocculation, filter. Ignite the precipitate, weigh, and add hydrofluoric acid. After vaporizing silicon dioxide, ignite again, weigh, and obtain the amount of principal silicon dioxide from its loss. Separately take the filtrate, and obtain the amount of residual silicon dioxide by a molybdenum blue absorptiometry. Take the sum of both amounts as the total amount of silicon dioxide.

7.3.2 Reagents The reagents shall be as follows:

- (1) Boric acid
- (2) Polyethylene oxide solution Add little by little 0.1 g of polyethylene oxide into 200 ml of water while mixing by stirring to be dissolved, and reserve in a plastic bottle. It is preferable not to use the solution for which two weeks have elapsed after preparation.
- (3) For other reagents, use the same reagents as those given in 7.2.2 (2) to (11).

7.3.3 Weighing out Amount of Sample Weigh out 0.50 g of the sample.

7.3.4 Operation Carry out the determining operation in accordance with the following procedures:

- (1) Weigh out a sample onto a platinum dish (for instance, number 75), and add 1.5 g of sodium carbonate (anhydride) and 0.2 g of boric acid. After mixing, heat initially at a low temperature, gradually raise the temperature, and fuse by igniting at about 1000°C ⁽¹⁾.
- (2) Cover with a watch glass. After standing to cool, add 20 ml of hydrochloric acid (1 + 1). After dissolving by heating on a water bath, continue to heat it as it is for about 20 min.

- (3) Remove the watch glass without washing with water⁽²⁾, and add a suitable amount of powdery filter paper. After sufficiently mixing by stirring, add about 10 ml of polyethylene oxide solution, sufficiently mix by stirring, and allow to stand for about 5 min.
- (4) Filter by using filter paper (class 5 B). After washing several times with hot hydrochloric acid (1 + 50), wash sufficiently with hot water. Receive the filtrate and washings with a beaker (300 ml), and reserve it for determination of residual silicon dioxide.
- (5) Put the precipitate together with the filter paper into a platinum crucible (for instance, number 30), and add one drop of sulfuric acid (1 + 1). After incinerating the filter paper at a low temperature, ignite at $1100 \pm 50^\circ\text{C}$ for about 1 h. After standing to cool in a desiccator, weigh the mass, and repeat the ignition until to become constant mass.
- (6) Moisten impure silicon dioxide with water, add three drops of sulfuric acid (1 + 1) and about 10 ml of hydrofluoric acid, heat on a sand bath, and dry up by evaporation. Ignite at $1100 \pm 50^\circ\text{C}$ for about 5 min. After standing to cool in a desiccator, weigh the mass.
- (7) Hereinafter, operate in accordance with the procedures of 7.2.4 (7) to (10).

7.3.5 Calculation Obtain the amount of residual silicon dioxide from the working curve prepared in 7.2.6, and calculate the content of silicon dioxide in the sample according to the following formula.

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

where, w_1 : mass weighed in 7.3.4 (5) (g)
 w_2 : mass weighed in 7.3.4 (6) (g)
 a : detection amount of residual silicon dioxide of 7.3.4 (7) (g)
 W : weighed out amount of sample (g)

7.3.6 Preparation of Working Curve Operate in the same way as in 7.2.6.

Notes ⁽¹⁾ When fusion time is too long, the sample becomes difficult to be dissolved in hydrochloric acid.

⁽²⁾ Washing of the watch glass is performed in the case of the operation of (4).

8. Method for Determination of Aluminium Oxide

8.1 Classification of Methods The method for determination of aluminium oxide shall be in accordance with either one of the following methods.

- (1) Oxine gravimetric method
- (2) EDTA-zinc back titrimetric method

8.2 Oxine Gravimetric Method

8.2.1 Summary Decompose a sample with hydrofluoric acid and sulfuric acid. After drying up by evaporation, fuse with potassium pyrosulfate, and dissolve in hydrochloric acid to make constant volume. Separately take a portion thereof, reduce iron with ascorbic acid, mask with o-phenanthroline, add oxine, regulate pH with ammonia and ammonium acetate, and generate aluminium oxine chelate. After maturing the precipitate on a water bath, filter. After drying, weigh the mass.

8.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 1)