

JIS

JAPANESE INDUSTRIAL STANDARD

Methods for Chemical Analysis of Dolomite

JIS M 8851—1983

Translated and Published

by

Japanese Standards Association

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Methods for Chemical Analysis of Dolomite

M 8851-1983

(Reaffirmed: 1990)

1. Scope

This Japanese Industrial Standard specifies the methods for chemical analysis of dolomite.

2. General Matters

The general matters common to methods for chemical analysis are in accordance with JIS K 0050, JIS K 0115 and JIS K 0121.

3. Analysis Items

The analysis items ⁽¹⁾ specified in this Standard are as follows:

- Ignition loss (Ig. loss)
- Silicon dioxide (SiO_2)
- Iron (III) oxide (Fe_2O_3)
- Aluminium oxide (Al_2O_3)
- Calcium oxide (CaO)
- Magnesium oxide (MgO)
- Phosphorus pentoxide (P_2O_5)
- Total sulfur (Total S)

Note ⁽¹⁾ Silicon dioxide (except the gravimetric method), iron (III) oxide, aluminium oxide, calcium oxide, magnesium oxide and phosphorus pentoxide are determined under respective element states, but, for convenience, these are marked by the form of oxides.

4. Sampling and Handling of Sample

4.1 The analysis sample shall be taken with cares so as to express the average composition by mixing thoroughly with stirring and, confirmed with that foreign matters are not mixed in.

4.2 Relating to the analysis sample, pulverize the material so as its 100 % passes through a 149- μm sieve of JIS Z 8801, spread its approximately 10 g thinly in a flat weighing bottle (50 mm in diameter), after drying in a air bath at 105 to 110°C for not less than 1 h, preserve in a desiccator, and then weigh out.

4.3 Relating to weighing-out of analysis sample, use a chemical balance, and read out the specified amount to the nearest place of 0.1 mg.

Applicable Standards: See page 29.

5. Arrangement of Analyzed Values

5.1 The analyzed results shall be expressed by percentage and, rounded off as follows according to JIS Z 8401.

- (1) The ignition loss, calcium oxide, and magnesium oxide are rounded off to one place of decimal or less.
- (2) Silicon dioxide, iron (III) oxide, aluminium oxide, and total sulfur by gravimetric method are rounded off to two places of decimals or less.
- (3) Phosphorus pentaoxide and total sulfur by thermal decomposition-potassium iodate titrimetric method are rounded off to three places of decimals or less.

5.2 At analysis, the blank test shall be carried out through all operations and the contents, corrected.

5.3 The analysis shall, as a rule, be repeated twice or more on the same sample, when the difference is larger than the numerical value shown in tolerance (%) of Table 1, again carry out the analysis twice or more, and obtain the average value within the tolerance.

Table 1. Tolerances (²)

Unit: %

Ignition loss	Silicon dioxide				Iron (III) oxide	
	Perchloric acid dehydration gravimetric method		Molybdenum blue absorptiometry		1,10-phenanthroline absorptiometry	
	Division	Tolerance	Division	Tolerance	Division	Tolerance
0.2	0.10 or over to and excl. 1.00	0.03	Under 0.40	0.02	Under 0.10	0.01
					0.10 or over to and excl. 0.50	0.03
	1.00 or over	0.05	0.40 or over to and excl. 1.00	0.04	0.50 or over to and excl. 1.50	0.05

Iron (III) oxide		Aluminium oxide			
Atomic absorption spectrophotometry		EDTA-bismuth back-titrimetric method		Atomic absorption spectrophotometry	
Division	Tolerance	Division	Tolerance	Division	Tolerance
Under 0.10	0.01	Under 0.50	0.02	Under 0.50	0.02
0.10 or over to and excl. 0.50	0.03				
0.50 or over to and excl. 1.00	0.05	0.50 or over	0.04	0.50 or over to and excl. 1.00	0.04

Table 1 (Cont'd)

Unit: %

Calcium oxide	Magnesium oxide	Phosphorus pentaoxide			
		Molybdenum blue absorptiometry		Phosphorus vanado molybdate absorptiometry	
		Division	Tolerance	Division	Tolerance
0.3	0.2	Under 0.050	0.002	0.030 or over to and excl. 0.100	0.004
		0.050 or over to and excl. 0.100	0.006		
		0.100 or over to and excl. 1.000	0.008	0.100 or over	0.007

Total sulfur

Thermal decomposition-potassium iodate titrimetric method		Barium sulfate gravimetric method	
Division	Tolerance	Division	Tolerance
Under 0.050	0.003	0.01 or over	0.01
0.050 or over to and excl. 0.100	0.005		

Note (2) In the case where the two analyzed values extend over two sections, the tolerance of corresponding division of average value of two analyzed values is applied.

6. Ignition Loss Determination Method

6.1 Division of Methods The determination method of ignition loss shall be in accordance with gravimetric method.

6.2 Gravimetric Method

6.2.1 Summary The loss when the sample is ignited at $1050 \pm 50^\circ\text{C}$ until becoming constant is measured.

6.2.2 Weighing-Out Amount of Sample Weigh out the sample of 1.0 g.

6.2.3 Operation Carry out the determination operation according to the following procedures:

- (1) Weigh out the sample into a platinum crucible (for example No. 30) (3), apply the cover with opening a slight clearance, heat at first at a low temperature, gradually raise the temperature and at last ignite at $1050 \pm 50^\circ\text{C}$ in an electric furnace for 1 h. Apply cover tightly, leave cool in a desiccator and thereafter weigh the mass.

- (2) Repeat the ignition each for 15 min, and obtain the loss when having become constant amount ⁽⁴⁾.

6.2.4 Calculation Calculate the ignition loss according to the following formula.

$$\text{Ignition loss (\%)} = \frac{w_1 - w_2}{W} \times 100$$

where, w_1 : mass of platinum crucible containing sample before igniting (g)

w_2 : mass of platinum crucible containing sample after igniting (g)

W : weighed-out amount of sample (g)

Notes ⁽³⁾ A porcelain crucible may be used.

⁽⁴⁾ Usually, the constant amount can be obtained by 1 h igniting.

7. Silicon Dioxide Determination Method

7.1 Division of Method The determination method of silicon dioxide shall be in accordance with either one of the following.

- (1) Perchloric Acid Dehydration Gravimetric Method This method applies to the sample not less than 0.10 % in silicon dioxide content.
- (2) Molybdate Blue Absorptiometry The method applies to the sample less than 1.00 % in silicon dioxide content.

7.2 Perchloric Acid Dehydration Gravimetric Method

7.2.1 Summary Decompose the sample with hydrochloric acid and perchloric acid to generate white fume by heating, and filter to divide the silicic acid as insoluble matter. Ignite the precipitate to weigh the mass, add hydrofluoric acid, heat to evaporate the silicon dioxide, thereafter again ignite to weigh the mass, and obtain the difference of masses before and after.

7.2.2 Reagents Reagents shall be as follows.

- (1) Hydrochloric Acid (1 + 1, 1 + 100)
- (2) Perchloric Acid (60 %)
- (3) Hydrofluoric Acid

7.2.3 Weighing-Out Amount of Sample Weigh out 1.00 g of sample.

7.2.4 Operation The determination operation shall be carried out according to the following procedures.

- (1) Weigh out ⁽⁵⁾ the sample into a beaker (200 ml), add a small amount of water to make slurry state, cover with a watch glass and add hydrochloric acid (1 + 1) 5 ml little by little from the edge of beaker. When having ended the vigorous reaction, wash the watch glass with a small amount of water to remove.
- (2) Add perchloric acid 5 ml, heat on a sand bath, when the dense white fume of perchloric acid has started to generate again cover with the watch glass and heat successively for 10 min.
- (3) After cooling for a while, add hydrochloric acid (1 + 1) 5 ml and warm water approximately 50 ml to mix by shaking, when the insoluble matters have precipitated approximately filter immediately with a filter paper (Class 5 B), wash with hot hydrochloric acid (1 + 100) several times, and further wash ⁽⁶⁾ sufficiently with hot water. Receive the filtrate and washings into a beaker (300 ml) and preserve.
- (4) Put the insoluble matters together with the filter paper into a platinum crucible (for example No. 30) to dry, gradually heat to incinerate so as the flame not to be generated, thereafter ignite at $1050 \pm 50^\circ\text{C}$ for 1 h, after cooling in a desiccator weigh the mass, and repeat the ignition until becoming constant amount ⁽⁴⁾.
- (5) Wet the insoluble matters with two or three drops ⁽⁷⁾ of hydrochloric acid, add hydrochloric acid 5 ml, and heat on a sand bath to evaporate to dryness. Ignite at $1050 \pm 50^\circ\text{C}$ for 10 min, and after leaving cool in a desiccator, weigh the mass.

Further, preserve the platinum crucible adhered with residual as it is.

7.2.5 Calculation Calculate the content of silicon dioxide in the sample according to the following formula.

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

where, w_1 : mass weighed in 7.2.4 (4) (g)

w_2 : mass weighed in 7.2.4 (5) (g)

W : weighed-out amount of sample (g)

Notes ⁽⁵⁾ In the case of sample containing a large amount of organic substances, preliminarily heat the weighed out sample in a porcelain crucible to 500 to 600°C , and after decomposing the organic substances, transfer into a beaker.

⁽⁶⁾ If the washing is insufficient, there is a fear for explosion scattering at the time of incineration of filter paper. Specially, in case where insoluble matters are large amount, cares are required.

- (7) In case where insoluble matters are a large amount, increase suitably.

Remark: In the case where the silicon dioxide is an extremely small amount and determination of this is not required specially, the operation of 7.2.4 (5) may be abbreviated and, marked as acid insoluble residue. In this case, a porcelain crucible may be used for ignition of insoluble matters.

7.3 Molybdate Blue Absorptiometry

7.3.1 Summary Fuse the sample with sodium carbonate and boric acid, and dissolve in hydrochloric acid to make a constant volume. Separately take a specific amount of this solution, add ammonium molybdate to make silicon molybdate, allow oxalic acid and sulfuric acid to coexist, add L-ascorbic acid to color the molybdate blue and measure the absorbance.

7.3.2 Reagents Reagents shall be as follows. Provided that (4) to (7) shall be preserved in a plastics bottle.

- (1) Hydrochloric Acid (1 + 9)
- (2) Sulfuric Acid (1 + 3)
- (3) Mixed Fusing Agent [Sodium Carbonate (Anhydride) 2, Boric Acid 1]
- (4) Ammonium Molybdate Solution Dissolve ammonium molybdate (tetrahydrate) 25 g in warm water to make 500 ml. If required, filter. However, when the molybdic acid is deposited during storing, newly prepare.
- (5) Oxalic Acid Solution Dissolve oxalic acid (dihydrate) 25 g in water to dilute to 500 ml.
- (6) L-Ascorbic Acid Solution (5 w/v %) Preserve in a cool-and-dark place. However, in case of having colored during storing, newly preserve it.
- (7) Standard Silicon Dioxide Solution (1.0 mg SiO₂/ml) Take silicon dioxide (anhydrous silicic acid, precipitation made) into a porcelain crucible, after igniting at 1050 ± 50°C for approximately 1 h, leave as it is in a desiccator, weigh out its 0.250 g into a platinum crucible (for example No. 30), after mixing with sodium carbonate (anhydride) 2.0 g, cover it to heat at first at a low temperature, gradually raise the temperature and at last ignite at 1000°C or higher to fuse. After leaving cool, put the platinum crucible into a plastic beaker (200 ml) containing warm water approximately 100 ml, and oscilate at times to dissolve the melt. Wash the platinum crucible and cover with water to take out, after cooling, transfer the solution into a measuring flask of 250 ml, and dilute with water up to the marked line. Immediately transfer to a plastic bottle.

- (8) Reagent Blank Solution Weigh out mixed fusing agent 3.0 g, put into a platinum crucible, cover it, at first heat at a low temperature, gradually raise the temperature to fuse, and successively heat for 2 to 3 min. Hereinafter, operate similarly to 7.3.4 (2).

7.3.3 Weighing-Out Amount of Sample Weigh out 0.25 g of sample.

7.3.4 Operation The determination operation shall be carried out according to the following procedures.

- (1) Weigh out the sample into a platinum crucible (for example No. 30) ⁽⁸⁾, after mixing with mixed fusing agent 3.0 g, cover it, at first heat at a low temperature, gradually raise the temperature to fuse and successively heat for 2 to 3 min.
- (2) After leaving cool, put the platinum crucible into the beaker (300 ml) containing hot hydrochloric acid (1 + 9) 150 ml, and heat on a water bath to dissolve the melt. Wash the platinum crucible and cover with water to take out, after cooling the solution until ordinary temperature, transfer into a measuring flask of 250 ml and dilute with water up to the marked line.
- (3) Separately take this solution 25 ml correctly into a measuring flask of 100 ml, add ammonium molybdate solution 10 ml, mix by shaking and leave as it is for 5 min.
- (4) Next, while mixing by shaking, quickly add continuously oxalic acid 10 ml, sulfuric acid (1 + 3) 5 ml, and ascorbic acid solution 2 ml, and after leaving as it is for 1 min, dilute with water up to the marked line.
- (5) After leaving as it is for 5 min, take a part of this solution into the absorption cell of photometer and measure the absorbance near wavelength 650 nm.

7.3.5 Calculation Obtain the silicon dioxide amount from the working curve prepared according to 7.3.6, and calculate the silicon dioxide content in the sample according to the following formula.

$$\text{Silicon dioxide (\%)} = \frac{a \times 10^{-3}}{W} \times \frac{250}{25} \times 100$$

where, a : silicon dioxide detected amount obtained from 7.3.4 (5) (mg)

W : weighed out amount of sample (g)

7.3.6 Preparation of Working Curve Dilute the standard silicon dioxide solution with water correctly to 20 times, take stepwise its 0 to 5.0 ml (as silicon dioxide 0 to 0.25 mg) correctly into several measuring flasks of 100 ml containing each 25 ml of reagent blank solution preliminarily, add ammonium molybdate solution 10 ml to mix by shaking, and leave as it is for 5 min. Hereinafter, operate according to the procedures of 7.3.4 (4) and (5), prepare the relation curve between the obtained absorbance and the silicon dioxide content amount and take it as the working curve.

Note ⁽⁸⁾ In the case where reducing substances such as organic substance, sulfide, etc. are contained in the sample, heat at 500 to 600°C to decompose.

8. Iron (III) Oxide

8.1 Division of Methods The determination method of iron (III) oxide shall be in accordance with either one of the following.

- (1) 1, 10-Phenanthroline Absorptiometry This method applies to sample less than 1.50 % in iron (III) oxide content.
- (2) Atomic Absorption Spectrophotometry This method applies to sample less than 1.00 % in iron (III) oxide content.

8.2 1, 10-Phenanthroline Absorptiometry

8.2.1 Summary Dissolve the residue in a platinum crucible preserved according to 7.2.4 in hydrochloric acid by fusing with mixed fusing agent, and add to the filtrate and washings preserved in 7.2.4 to make the specified volume. Separately take a part of this, reduce the iron by ascorbic acid, add 1, 10-phenanthroline, adjust pH with ammonium acetic acid to color, and measure the absorbance.

8.2.2 Reagents Reagents shall be as follows.

- (1) Hydrochloric Acid (1 + 1)
- (2) Mixed Fusing Agent [Sodium Carbonate (Anhydride) 3, Boric Acid 1]
- (3) Ammonium Acetate Solution (20 w/v %)
- (4) L-Ascorbic Acid Solution (5 w/v %) Use the solution same as that of 7.3.2 (5).
- (5) 1, 10-Phenanthroline Solution Dissolve 1, 10-phenanthroline hydrochlorate (mono hydrate) 1 g in water, dilute to 1 l and preserve in a cool-and-dark place. When colorated during preservation, prepare newly.
- (6) Standard Iron (III) Oxide Solution-I (0.10 mg Fe₂O₃ ml) Weigh out ammonium sulfate iron (III) (12 hydrate) 0.604 g, add water approximately 100 ml and sulfuric acid (1 + 1) 10 ml to dissolve, thereafter transfer to a measuring flask of 1000 ml, and dilute with water up to the marked line.

8.2.3 Operation The determination operation shall be carried out according to the following procedures.

- (1) Add mixed fusing agent 2 g to the platinum crucible preserved in 7.2.4 (5), cover it, heat at first at a low temperature. and gradually raise the temperature to fuse.
- (2) After leaving cool, put the platinum crucible into a beaker (100 ml), heat by adding hot water, after dissolving the melt, cover with a watch glass and add hydrochloric acid (1 + 1) 5 to 7 ml little by little. Wash the platinum crucible and cover with water, take out and boil to expel carbon dioxide. After cooling, add the filtrate and washings in the beaker (300 ml) preserved according to 7.2.4 (3).