

JIS

JAPANESE INDUSTRIAL STANDARD

**Method for Chemical Analysis
of Aluminous Abrasives**

JIS R 6123—1987

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

J I S

Method for Chemical Analysis of
Aluminous Abrasives

R 6123-1987

1. Scope

This Japanese Industrial Standard specifies the method for chemical analysis of aluminous abrasives, hereinafter referred to as the "abrasives".

Remark: The units and numerical values given in { } in this Standard are based on the traditional units and are currently the criteria in force.

2. Analytical Items

The analytical items for abrasives shall be divided according to the type as given in Table 1.

Table 1. Analytical Items

Type Analytical items	A	WA	PA	HA	AE	AZ
Ignition loss (Ig. loss)	○	○	○	○	○	○
Silicon dioxide (SiO ₂)	○	○	○	○	○	○
Ferric oxide (Fe ₂ O ₃)	○	○	○	○	○	○
Titanium oxide (TiO ₂)	○	—	△	△	○	○
Calcium oxide (CaO)	○	—	—	—	○	—
Magnesium oxide (MgO)	○	—	—	—	○	—
Zirconium oxide (ZrO ₂)	○	—	—	—	○	○
Sodium oxide (Na ₂ O)	—	○	○	○	—	○
Chromium oxide (Cr ₂ O ₃)	—	—	○	—	—	—
Aluminium oxide (Al ₂ O ₃)	○	○	○	○	○	○

Remark: The mark ○ means the item to be analyzed and the mark △ means the item to be analyzed only when titanium oxide is contained.

Applicable Standards, Corresponding International Standard and Reference Standard: See page 35.

3. General Items

The general items commonly applied to chemical analysis shall be in accordance with JIS K 0050, those to related absorptiometry, in accordance with JIS K 0115, and those related to atomic absorption analysis, in accordance with JIS K 0121.

The symbols for classes of the abrasives shall conform to JIS R 6111 (see Table 2).

Table 2

Division	Class	Symbol
Aluminous abrasives	Brown aluminous abrasive	A
	White aluminous abrasive	WA
	Pink aluminous abrasive	PA
	Mono-crystalline fused aluminous abrasive	HA
	Artificial emery abrasive	AE
	Aluminous zinconia abrasive	AZ

4. Sample

Take approximately 10 g of the abrasives from one testing unit according to JIS R 6003 put in a flat weighing bottle (60 mm x 30 mm). After drying for 1 h in an air oven of $105 \pm 5^\circ\text{C}$, dose the bottle tight and keep it in a desiccator to supply as a sample.

Sample shall, as a rule, be treated in the state of original grit, but, in case pulverization is necessary, the mortar with no likelihood of contaminating the sample shall be used (for instance, the mortar made of boron carbide). The sample, however, shall be pulverized to such a degree as passing through the sieve of 150 μm , and over-pulverization shall be avoided.

5. Rounding off Analysis Results

Analysis results shall be expressed in percentage, and be rounded off to the second decimal place in accordance with JIS Z 8401.

6. Determination Methods by Classification

The determination methods for abrasives shall be divided according to the class.

- (1) Determination methods of A and AE
- (2) Determination methods of WA, PA and HA
- (3) Determination method of AZ

7. Determination Methods of A and AE

7.1 Determination of Ignition Loss

7.1.1 Summary Heat strongly the sample at 1000°C, and determine the decrease in weight as the ignition loss.

7.1.2 Apparatus and Device Apparatuses and device to be used shall be as follows:

- (1) Platinum Crucible with Cap (hereinafter referred to as the "crucible") For instance, No. 30 specified in JIS H 6201.
- (2) Electric Furnace Shall be capable of keeping temperature at 1000°C.
- (3) Thermometer Shall be capable of measuring temperature up to 1000°C.

7.1.3 Quantity of Sample Approximately 1 g of sample shall be weighed out.

7.1.4 Operation Determination operation shall be carried out as follows:

- (1) Weigh approximately 1 g of the sample accurately to the nearest 0.1 mg into the crucible preliminarily set at a constant weight of 1000 ± 50°C.
- (2) Place the crucible in the electric furnace controlled at 1000 ± 50°C, and heat strongly for 1 h. Then cool sufficiently in a desiccator, and weigh its mass. Repeat this operation until constant weight is obtained.
- (3) Ignition loss shall be calculated from the following formula:

$$\text{Ignition loss (\%)} = \frac{m_2}{m_1} \times 100$$

where Ignition loss (%) : content of ignition loss (%)

m_1 : quantity of sample taken (g)

m_2 : quantity decreased by ignition (g)

7.2 Determination of Silicon Dioxide

7.2.1 Division of Determination Methods Either of the following two methods shall be adopted for silicon dioxide determination.

- (1) Gravimetric method
- (2) Molybdenum yellow absorptiometry

7.2.2 Gravimetric Method

7.2.2.1 Summary Add sodium carbonate anhydride and boric acid to the sample put in a crucible, and, after fusing them by heating, dissolve them in sulfuric acid. By heating it until white a fume generates, make it insoluble silicon dioxide, and evaporate to disperse it by adding hydrofluoric acid. From the weight decreased, determine the content percentage of silicon dioxide.

7.2.2.2 Reagents Reagents to be used shall be as follows:

- (1) Sodium Carbonate Anhydride
- (2) Boric Acid
- (3) Sulfuric Acid (1 + 1)
- (4) Hydrofluoric Acid

7.2.2.3 Device and Apparatus Device and apparatuses shall be as follows:

- (1) Crucible For instance, No. 30.
- (2) Electric Furnace Shall be capable of keeping temperature at 1000°C.
- (3) Thermometer Shall be capable of measuring temperature up to 1000°C.

7.2.2.4 Quantity of Sample Approximately 0.5 g of sample shall be weighed out.

7.2.2.5 Operation Determination operation shall be carried out as follows:

- (1) Put 3 g of sodium carbonate anhydride and 2 g of boric acid in a crucible, and heat them to dehydrate on a burner⁽¹⁾.

Note (1) As soon as the sodium carbonate anhydride and boric acid are fused, remove the crucible from the burner. If heated excessively long, the sample may become hard to fuse.

- (2) Into this crucible, weigh out accurately approximately 0.5 g of the sample to the nearest 0.1 mg, put on the cap and fuse them by heating.
- (3) After cooling, transfer the fused substance together with the crucible and cap into a 300 ml beaker containing 100 ml of water and 30 ml of sulfuric acid (1 + 1), and completely dissolve the fused substance in the crucible by heating.

- (4) Rinse the crucible and cap satisfactorily with water and draw them out from the beaker, heat to evaporate the solution until white a fume of sulfuric acid generates on a sand bath, and further heat for 5 to 10 min.
- (5) After cooling, add 100 ml of warm water and, after dissolving soluble salts completely by heating, filter the solution immediately through filter paper (Class 5 B). Wash the precipitate with warm water until no sulfuric ion reaction is recognized.
- (6) Gather the filtrate and washings in a 250 ml transfer flask, and dilute the solution pouring water up to the marked line, and store it to be used for the determination of ferric oxide and titanium oxide.
- (7) Place the precipitate together with the filter paper in the crucible, and, after drying it gradually to dehydrate sufficiently, incinerate it by heating at low temperature.
- (8) Next, heat it strongly in the electric furnace at 1000°C for 1 h and, after cooling sufficiently in a desiccator, weigh its mass. Repeat this operation until a constant weight is obtained.
- (9) After moistening with a few drops of sulfuric acid (1 + 1), add 2 to 3 ml of hydrofluoric acid, and heat it on a sand bath taking care not to let the contents scatter.
- (10) Even after no more white fume of sulfuric acid is recognized, further heat strongly approximately 1000°C for 15 min, leave to cool sufficiently in a desiccator, and weigh its mass. Repeat this operation until constant weight is obtained.
- (11) The content percentage of silicon dioxide shall be calculated from the following formula:

$$\text{SiO}_2 (\%) = \frac{m_2 - m_3}{m_1} \times 100$$

where $\text{SiO}_2 (\%)$: content percentage of silicon dioxide (%)

m_1 : quantity of sample taken (g)

m_2 : quantity weighed at operation (8) (g)

m_3 : quantity weighed at operation (10) (g)

7.2.3 Molybdenum Yellow Absorptiometry

7.2.3.1 Summary Put the sample in a crucible, and fuse by heating after adding sodium carbonate anhydride and boric acid. Dissolve them in sulfuric acid, and measure the absorbancy of molybdenum yellow produced by the addition of ammonium molybdate solution to determine the silicon dioxide.

7.2.3.2 Reagents Reagents to be used shall be as follows:

- (1) Sodium Carbonate Anhydride
- (2) Boric Acid
- (3) Sulfuric Acid (1 + 1) Its specific gravity shall be 1.54⁽²⁾

Note ⁽²⁾ Pay attention to the specific gravity of sulfuric acid because sulfuric acid left for a long time under unsealed condition does not make 1.54 of specific gravity when dilute to the ratio of (1 + 1).

- (4) Ammonium Molybdate Solution (10 w/v %)
- (5) Silicon Dioxide Reference Solution (0.1 mg SiO₂/ml) Weigh out 0.100 g of silicon dioxide (99.9 % min.) in a crucible and, after adding 2 g of sodium carbonate anhydride, fuse them by heating. After dissolving in a 300 ml plastic beaker with water, transfer in a 1 l transfer flask, dilute pouring water up to the marked line, and store in a plastic bottle.

7.2.3.3 Apparatus The following apparatus shall be used.

- (1) Crucible For instance, No. 30
- (2) Photometer Either a photoelectric photometer or a photoelectric spectrophotometer shall be used.

7.2.3.4 Quantity of Sample Approximately 0.5 g of sample shall be weighed out.

7.2.3.5 Operation Determination operation shall be carried out as follows:

- (1) Put 6.0 g of sodium carbonate anhydride and 4.0 g of boric acid into a crucible, and heat to dehydrate on a burner.
- (2) Into this crucible, weigh out approximately 0.5 g of the sample accurately to the nearest 0.1 mg, and after putting a cap fuse them by heating.
- (3) After cooling, transfer the fuse substance together with the crucible and cap into a 300 ml beaker containing 12 ml of sulfuric acid (1 + 1) and 100 to 150 ml of water, and dissolve the fused substance in the crucible completely by heating. Then, rinse both the crucible and cap satisfactorily with water and draw them out.
- (4) After cooling the solution in the beaker, transfer it in a 250 ml transfer flask, and dilute it pouring water up to the marked line. After full agitation, carry out the following operation on an aliquot of the solution, and store the remaining solution to be used for determination of ferric oxide and titanium oxide.

- (5) Take accurately 50 ml out of this solution into a 100 ml transfer flask⁽³⁾ and, after adding 3 ml of ammonium molybdate solution (10 w/v %), dilute it pouring water up to the marked line, followed by agitation.

Note ⁽³⁾ If operation follows this direction correctly, pH would become 1.0 to 1.3 when diluted to 100 ml.

- (6) In a duration of 5 to 15 min, place a part of the solution into a 10 mm absorption cell, and measure the absorbances with reference to blank test solution in the vicinity of 420 nm wavelength. Obtain silicon dioxide content by the use of the working curve prepared in advance.
- (7) The content percentage of silicon dioxide shall be calculated from the following formula:

$$\text{SiO}_2 (\%) = \frac{m_2}{m_1 \times \frac{50}{250}} \times 100$$

where $\text{SiO}_2 (\%)$: content percentage of silicon dioxide (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of silicon dioxide detected in aliquot (g)

7.2.3.6 Preparation of Working Curve Stepwise parcel out 0 to 25.0 ml of silicon dioxide reference solutions (0.1 mg SiO_2 /ml) (0 to 2.5 mg as SiO_2) into plural transfer flasks of each 100 ml, add 50 ml water and 0.5 ml sulfuric acid (1 + 1) to each of them, carry out the operation after addition of ammonium molybdate solution (10 w/v %) as described in 7.2.3.5 (5) and plot a relation curve between the quantity of silicon dioxide and the absorbance to take as the working curve.

7.3 Determination of Ferric Oxide

7.3.1 Summary Add hydroxylammonium chloride solution to the solution stored in 7.2 to reduce ferric ions to ferrous ions and, after colouring the solution by adding 1,10-phenanthroline solution, determine ferric oxide content by measuring its absorbance.

7.3.2 Reagents Reagents to be used shall be as follows:

- (1) Sodium Acetate Solution (50 w/v %)
- (2) Hydroxylammonium Chloride Solution (0.5 w/v %)⁽⁴⁾

Note ⁽⁴⁾ It is permitted to mix hydroxylammonium chloride solution and 1,10-phenanthroline solution in advance and to use 10 ml of the solution.

- (3) 1,10-Phenanthroline Solution (0.2 w/v %)⁽⁴⁾

- (4) Iron Reference Solution (0.1 mg Fe₂O₃/ml) Weigh out 0.070 g of iron (99.5 % min.) in a 300 ml beaker, dissolve by adding 5 ml nitric acid (1 + 1) and 5 ml sulfuric acid (1 + 1) and heat until a white fume of sulfuric acid is generated. Dissolve them by adding 10 ml nitric acid (1 + 6) and water, transfer into a 1 l transfer flask and dilute to the marked line with water, one ml this solution contains 0.01 mg of ferric oxide.
- (5) Iron Reference Solution (0.01 mg Fe₂O₃/ml) 10 ml of iron reference solution (0.1 mg Fe₂O₃/ml) into a 100 ml transfer flask, add 5 ml of nitric acid (1 + 6) and dilute to the marked line with water.

7.3.3 Apparatus For the apparatus, either a photoelectric photometer or a photoelectric spectrophotometer shall be used.

7.3.4 Operation

7.3.4.1 In the Case of A The determination operation shall be carried out as follows:

- (1) Take correctly 50 ml from the solution stored in 7.2.2.5 (6), or 7.2.3.5 (6) into a 100 ml beaker.
- (2) Add sodium acetate solution (50 w/v %) so as to make pH of the solution 3.5⁽⁵⁾, and transfer it into a 100 ml transfer flask.

Note ⁽⁵⁾ To control pH, use pH meter or Bromphenol Blue (BPB) in pH test paper.
- (3) Add 5 ml of hydroxylammonium chloride solution (0.5 w/v %) to the solution, and mix by agitation.
- (4) Add 5 ml of 1,10-phenanthroline solution (0.2 w/v %) and, after diluting the mixture pouring water up to the marked line, agitate it to mix.
- (5) After 15 min, put a portion of the solution into a 10 mm absorption cell, and measure its absorbance with reference to blank test solution in the vicinity of 500 nm wavelength. Obtain ferric oxide content by the use of the working curve prepared in advance.
- (6) The content percentage of ferric oxide shall be calculated from the following formula:

$$Fe_2O_3 (\%) = \frac{m_2}{m_1 \times \frac{50}{250}} \times 100$$

where $Fe_2O_3 (\%)$: content percentage of ferric oxide (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of ferric oxide detected in aliquot (g)

7.3.4.2 In the Case of AE The determination operation shall be carried out as follows:

- (1) Take correctly 25 ml from the solution stored in 7.2.2.5 (6) into a 250 ml transfer flask, and dilute the solution pouring water up to the marked line. Transfer correctly 10 ml from the above solution into a 100 ml beaker, and add 40 ml of water to it.
- (2) Carry out the operations from 7.3.4.1 (2) to (5).
- (3) The content percentage of ferric oxide shall be calculated from the following formula:

$$Fe_2O_3 (\%) = \frac{m_2}{m_1 \times \frac{25}{250} \times \frac{10}{250}} \times 100$$

where $Fe_2O_3 (\%)$: content percentage of ferric oxide (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of ferric oxide detected in aliquot (g)

7.3.5 Preparation of Working Curve Stepwise parcel out 0 to 10.0 ml of iron reference solutions (0.01 ml Fe_2O_3 /ml) (0 to 0.1 mg as Fe_2O_3) into plural beakers of 100 ml, add 50 ml water to each of them, carry out the operation after the addition of sodium acetate solution as described in 7.3.4.1 (2) and plot a relation curve between the quantity of ferric oxide and the absorbance to take as the working curve.

7.4 Determination of Titanium Oxide

7.4.1 Summary Add hydrogen peroxide water to the solution stored in 7. to obtain colouring, and determine titanium oxide by measuring its absorbance.

7.4.2 Reagents Reagents to be used shall be as follows:

- (1) Hydrogen Peroxide Water (1 + 9)
- (2) Phosphoric Acid (1 + 9)
- (3) Sulfuric Acid (1 + 10)
- (4) Titanium Oxide Reference Solution (1 mg TiO_2 /ml) Weigh out 1.000 g titanium oxide (99.9 % min.) in a platinum dish, add 20 ml of sulfuric acid (1 + 1) and 30 ml of hydrofluoric acid, and fuse them by heating on a sand bath. After a white fume of sulfuric acid begins to be generated, further heat for 10 min, leave to cool, add 85 ml of sulfuric acid (1 + 1), transfer the solution into a 1 l transfer flask, and dilute it pouring water up to the marked line.

10
R 6123-1987

7.4.3 Apparatus As the apparatus, either photoelectric photometer or photoelectric spectrophotometer shall be used.

7.4.4 Operation Determination operation shall be as follows:

7.4.4.1 In the Case of A The determination operation shall be carried out as follows:

- (1) Take correctly 50 ml from the solution stored in 7.2.2.5 (6) or 7.2.3.5 (4) into a 100 ml transfer flask.
- (2) Add 5 ml of hydrogen peroxide water (1 + 9).
- (3) Add sulfuric acid (1 + 10) up to the marked line and agitate to mix it.
- (4) 10 min later, transfer a part of the solution into a 10 mm absorption cell, and measure the absorbance with reference to the blank test solution in the vicinity of 420 nm wavelength. Obtain titanium oxide content by the use of the working curve prepared in advance.
- (5) The content percentage of titanium oxide shall be calculated from the following formula:

$$TiO_2 (\%) = \frac{m_2}{m_1 \times \frac{50}{250}} \times 100$$

where $TiO_2 (\%)$: content percentage of titanium oxide (%)

m_1 : quantity of sample taken (g)

m_2 : quantity of titanium oxide detected in aliquot (g)

7.4.4.2 In the Case of AE The determination operation shall be carried out as follows:

- (1) Take correctly 50 ml from the solution stored in 7.2.2.5 (6) into a 100 ml transfer flask.
- (2) Add 10 ml of phosphoric acid (1 + 9) and then 5 ml of hydrogen peroxide water (1 + 9) to the solution.
- (3) Carry out the operations from 7.4.4.1 (3) to (4).
- (4) The content percentage of titanium oxide shall be calculated from the formula of 7.4.4.1 (5).

7.4.5 Preparation of Working Curve

7.4.5.1 In the Case of A Stepwise parcel out 0 to 5.0 ml titanium oxide reference solutions (1 mg TiO_2 /ml) (0 to 5 mg as TiO_2) into 100 ml transfer flasks, add 50 ml sulfuric acid (1 + 10) to each of them, carry out the operation after the addition of hydrogen peroxide water as described in 7.4.4.1 (2) and prepare the relation curve between the quantity of titanium oxide and the absorbance to take as the working curve.