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JIS

JAPANESE INDUSTRIAL STANDARD

Titanium dioxide for chemical fiber

JIS K 1409-1994

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

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JAPANESE INDUSTRIAL STANDARD
J I S

Titanium dioxide for chemical fiber
TiO_2 *FW*: 79.88

K 1409-1994

1. Scope This Japanese Industrial Standard specifies titanium dioxide for chemical fiber.

Remarks 1. The standards cited in this Standard are listed in Attached Table 1.

2. The units and numerical values given in { } in this Standard are based on the traditional units, and appended for informative reference.

2. Classification Classification shall be as follows.

(1) Class 1 Water-dispersion type

(2) Class 2 Water-aggregation type

3. Quality The quality, when it is tested in accordance with 5., shall conform to the requirements in Table 1.

Table 1. Quality

Item		Classification	
		Class 1	Class 2
Titanium dioxide (TiO_2)	%	98.0 min.	97.5 min.
pH value		6.0 to 8.0	6.0 to 8.0
Iron (Fe)	%	0.005 max.	0.005 max.
Calcium (Ca)	%	0.01 max.	0.2 max.
Water content	%	0.5 max.	0.5 max.
Ignition loss	%	0.3 max.	0.3 max.
Insoluble by sulfuric acid	%	0.3 max.	0.3 max.
Residue on sieve	%	0.03 max.	0.03 max.
Number of coarse grains (number of grains with major diameter of 5 µm or longer)		10 max.	10 max.
Whiteness	%	96 min.	96 min.
Tinting strength		Value of tinting strength by evaluation sample±50	Value of tinting strength by evaluation sample±50
Degree of in-water dispersion	%	90 min.	—

4. Sampling method

(1) Preparation of lot Lot shall be the material produced under the same condition, and consist of titanium dioxide for chemical fiber with definite amount which is regarded as with the same quality.

(2) Operation Choose at random 2 containers(1) from the paper-made container or the like at every lot, take out the specified quantity more than twice of the required for test from possible center part of each container, and then mix them sufficiently to prepare sample.

Note(1) The number of containers to be chosen may follow the agreement between the parties concerned.

(3) Reduction Reduction shall be carried out on the mixed sample in accordance with a conically quartering method or the like to get twice quantity of the required for test.

(4) Storing Store the sample in a sample container capable of preventing light and moisture after tight stoppering, and record the name of sample, date of sampling, lot number, and other needed items.

5. Test method

5.1 General matters The general matters, applicable commonly to the tests, shall follow JIS K 0050.

5.2 Rounding-off of numerical values Follow JIS Z 8401.

5.3 Titanium dioxide (TiO_2)

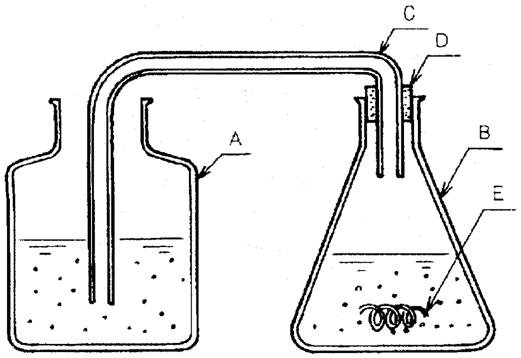
5.3.1 Summary Add sulfuric acid and ammonium sulfate into sample, heat them to dissolve it, allow it to cool, add water and hydrochloric acid, add metal aluminum to reduce titanium, allow it to cool, add potassium thiocyanate solution as indicator, and titrate it with ammonium iron (III) sulfate solution to determine titanium dioxide.

5.3.2 Apparatus Apparatus shall be as follows.

(1) Thermostatic dryer Capable of controlling at 105±2°C.

(2) Reducing apparatus Its example is shown in Fig. 1.

Fig. 1. Example of reducing apparatus



A : Wide-mouthed bottle [Sodium hydrogencarbonate solution (saturated)]
B : Erlenmeyer flask 500 ml
C : U-tube
D : Rubber stopper
E : Metal aluminum

5.3.3 Reagents Reagents shall be as follows.

(1) Sulfuric acid Specified in JIS K 8951.

(2) Ammonium sulfate Specified in JIS K 8960.

(3) Hydrochloric acid Guaranteed reagent specified in JIS K 8180.

(4) Metal aluminum Prepare aluminum wire or rod type with No. 1200 of alloy number specified in JIS H 4040, cut it to make one piece about 3 g, and wash with hydrochloric acid (1+5) prepared by using hydrochloric acid specified in (3), prior to employing.

(5) Sodium hydrogencarbonate solution (saturated) Solution prepared using sodium hydrogencarbonate specified in JIS K 8622.

Informative reference: The solubility of sodium hydrogencarbonate is 9.6 g per 100 g water at 20°C.

(6) Potassium thiocyanate solution (saturated) Solution prepared using potassium thiocyanate specified in JIS K 9001.

Informative reference: The solubility of potassium thiocyanate is 217 g per 100 g water at 20°C.

(7) 0.1 mol/*l* ammonium iron (III) sulfate solution The preparation and standardization of 0.1 mol/*l* ammonium iron (III) sulfate solution shall be as follows.

Preparation Dissolve 49 g of ammonium iron (III) sulfate 12-water, specified in JIS K 8982, in about 300 ml water containing 10 ml hydrochloric acid (1+1) which has been prepared using hydrochloric acid specified in (3), transfer it into a 1000 ml measuring flask, and add water up to the marked line.

Standardization Take 25 ml of prepared ammonium iron (III) sulfate solution into a 250 ml iodine flask or a 250 ml Erlenmeyer flask with a ground stopper using a transfer pipet, add 35 ml of water and 3 ml of hydrochloric acid (1+1), add 3 g of potassium iodide specified in JIS K 8913, immediately stopper it, shake it gently, and allow it to stand in dark place for 10 min. Titrate isolated iodine with 0.1 mol/*l* sodium thiosulfate solution, make its color faint yellow, add either 10 ml of sodium acetate solution (250 g/*l*) which has been prepared using sodium acetate (anhydrous) specified in JIS K 8372 or 10 ml of ammonium acetate solution (250 g/*l*) which has been prepared using ammonium acetate specified in JIS K 8359, add 3 ml of starch solution as indicator, and titrate on it to find an end point when blue color by iodine starch disappears. Separately, carry out a blank test under the same condition, and calculate the factor of 0.1 mol/*l* ammonium iron (III) sulfate solution according to the following formula.

$$f = \frac{(a - a')}{25} \times f'$$

where, f : factor of 0.1 mol/*l* ammonium iron (III) sulfate solution

a : quantity of 0.1 mol/*l* sodium thiosulfate solution needed for titration (ml)

a' : quantity of 0.1 mol/*l* sodium thiosulfate solution needed for blank test (ml)

25 : quantity of 0.1 mol/*l* ammonium iron (III) sulfate solution (ml)

f' : factor of 0.1 mol/*l* sodium thiosulfate solution

Remarks 1. 0.1 mol/*l* sodium thiosulfate solution The preparation and standardization of 0.1 mol/*l* sodium thiosulfate solution shall be as follows.

Preparation Dissolve 26 g of sodium thiosulfate pentahydrate specified in JIS K 8637 and 0.2 g of sodium carbonate specified in JIS K 8625 in 1000 ml of carbon dioxide-free water specified in 3.6(3) of JIS K 8001.

Add 10 ml of 3-methyl-1-butanol specified in JIS K 8051 thereto, stopper it, shake sufficiently, and allow it to stand for 2 days.

Standardization Take 25 ml of 1/60 mol/*l* potassium iodate solution into a 250 ml iodine flask or a 250 ml Erlenmeyer flask with a ground stopper using a transfer pipet, add 20 ml of water, add 3 g of potassium iodide specified in JIS K 8913 and 5 ml of hydrochloric acid (2+1) which has been prepared using hydrochloric acid of (3), immediately stopper it, shake gently, and allow it to stand in dark place for 5 min. Titrate isolated iodine with 0.1 mol/*l* sodium thiosulfate solution, make its color faint yellow, add 3 ml of starch solution as indicator, and find an end point when blue color by iodine starch disappears.

Separately, carry out a blank test under the same condition, and calculate the factor of 0.1 mol/*l* sodium thiosulfate solution according to the following formula.

$$f = \frac{25}{b - b'}$$

where, f : factor of 0.1 mol/*l* sodium thiosulfate solution

25 : quantity of 1/60 mol/*l* potassium iodate solution (ml)

b : quantity of 0.1 mol/*l* sodium thiosulfate solution needed for titration (ml)

b' : quantity of 0.1 mol/*l* sodium thiosulfate solution needed for blank test (ml)

2. 1/60 mol/*l* potassium iodate solution Heat potassium iodate specified in JIS K 8005 at 130°C for about 2 h, allow it to cool in a desiccator with silica gel for drying agent, weigh its 0.8917 g (in case of 100% purity), dissolve it in water, transfer it into a 250 ml measuring flask, and add water up to the marked line.

3. Starch solution Add 1 g of starch (soluble) specified in JIS K 8658 in about 10 ml of water, pour it into about 200 ml of hot water while agitating it, boil for about 1 min, allow it to cool and if necessary, filter it. Prepare this solution when it is used.

5.3.4 Operation Operation shall be as follows.

(1) Dry sample for about 2 h in a thermostatic dryer kept at 105±2°C, weigh its 0.2 g to the nearest 0.1 mg, and transfer it into a 500 ml Erlenmeyer flask.

(2) Add a little amount of water, shake it to make it milky condition, add 30 ml of sulfuric acid and 12 g of ammonium sulfate, and heat at first gently, finally strong to dissolve it.

(3) After cooling, add 120 ml of water and 40 ml of hydrochloric acid, and shake sufficiently to dissolve it.

(4) Add 3 g of metal aluminum, immediately insert a U-tube with a rubber stopper, insert its other end into a wide-mouthed bottle in which sodium hydrogencarbonate solution (saturated) has been put, and make hydrogen gas evolve.

(5) After the solution becomes transparent violet owing to completely dissolving of metal aluminum, allow it to stand for a while, and cool it to get 50°C or lower using running water(2).

Note(2) At this time, sodium hydrogencarbonate solution flows backward in the Erlenmeyer flask, and this flowing will be stopped owing to the generation of carbon dioxide.

(6) Disconnect the U-tube with a rubber stopper, add 3 ml of potassium thiocyanate solution (saturated) as indicator, immediately titrate it with 0.1 mol/*l* ammonium iron (III) sulfate solution, and make it an end point when faint brown color of the liquid lasts for about 30 s.

5.3.5 Calculation Titanium dioxide shall be calculated as follows.

$$C = \frac{0.007988 \times d \times f}{S} \times 100$$

where, C : titanium dioxide (%)

0.007988 : amount of titanium dioxide equivalent to 1 ml of 0.1 mol/*l* ammonium iron (III) sulfate solution (g)

d : quantity of 0.1 mol/*l* ammonium iron (III) sulfate solution needed for titation (ml)

f : factor of 0.1 mol/*l* ammonium iron (III) sulfate solution

S : mass of sample (g)

5.4 pH value

5.4.1 Summary Add water in sample and boil it, allow it to cool, and measure pH value of supernatant or suspension using a pH meter.

5.4.2 Apparatus Apparatus shall be as follows.

(1) pH meter Type I or Type II of pH meter specified in 4.2 of JIS Z 8802.

(2) Glass electrode for ordinary temperature Specified in 3. of JIS Z 8805.

5.4.3 Reagent Reagent shall be as follows.

(1) Carbon dioxide-free water Specified in 3.6(3) of JIS K 8001.

5.4.4 Operation Operation shall be as follows.

(1) Weigh 5.0 g of sample to the nearest 0.1 g, and put it into a 300 ml Erlenmeyer flask (grade 1 of hard glass).

(2) Add 100 ml of carbon dioxide-free water, and boil it for 5 min.

(3) Complement its evaporating loss with carbon dioxide-free water and allow it to cool after stoppering.

(4) According to 7. of JIS Z 8802, measure pH value of supernatant or suspension using a pH meter.

5.5 Iron (Fe)

5.5.1 Summary Decompose sample with hydrochloric acid and hydrofluoric acid, oxidize it by adding nitric acid, color it adding boric acid, tartaric acid, ammonium acetate, hydroxylammonium chloride, and 1,10-phenanthroline, and measure its absorbance, to determine iron.

5.5.2 Apparatus and implements Apparatus and implements shall be as follows.

(1) Absorptiometric analyzer Specified in JIS K 0115.

(2) Platinum dish No. 50 specified in JIS H 6202.

(3) Hot plate Suitable one.

(4) Polyethylene watch glass Suitable one.

5.5.3 Reagents Reagents shall be as follows.

(1) Hydrochloric acid Guaranteed reagent specified in JIS K 8180.

(2) Hydrofluoric acid Specified in JIS K 8819.

(3) Nitric acid (1+1) Prepared using nitric acid specified in JIS K 8541.

(4) Boric acid Specified in JIS K 8863.

(5) Tartaric acid solution (200 g/*l*) Prepared using L(+)-tartaric acid specified in JIS K 8532.

(6) Ammonium acetate solution (500 g/*l*) Prepared using ammonium acetate specified in JIS K 8359.

(7) Hydroxylammonium chloride solution (100 g/*l*) Prepared using hydroxylammonium chloride specified in JIS K 8201.

(8) 1,10-phenanthroline solution (2 g/*l*) Prepared using 1,10-phenanthroline monohydrate (*o*-phenanthroline) specified in JIS K 8789.

(9) Iron reference solution (5 µg Fe/ml) Weigh 0.500 g of iron (99.5% purity or more) in a 200 ml beaker, add 8 ml of sulfuric acid (1+1) prepared using sulfuric acid of 5.3.3(1) and about 10 ml of water, heat them to dissolve it, add 1 ml of nitric acid (1+1) to oxidize iron, and boil it to expel nitrogen oxide. After cooling, transfer it into a 1000 ml measuring flask, and add water up to the marked line. When it is used, take 5 ml of this solution using a transfer pipet into a 500 ml of measuring flask, and add water up to the marked line, followed by shaking. Otherwise, employ the iron standard solution (Fe 5) specified in JIS K 0016.

5.5.4 Operation Operation shall be as follows.

(1) Weigh about 1 g of sample to the nearest 1 mg into a platinum dish, add 20 ml of hydrochloric acid and 5 ml of hydrofluoric acid, cover the dish with a polyethylene watch glass, and heat it on a hot plate to dissolve the sample.

(2) Add 3 ml of nitric acid (1+1), and heat it to expel nitrogen oxide. Then add 3 g of boric acid, stir sufficiently to dissolve it, and allow it to cool.

(3) Transfer it into a 100 ml measuring flask, add water up to the marked line, shake it sufficiently, and take 20 ml using a transfer pipet into a 100 ml measuring flask.

(4) Add orderly 15 ml of tartaric acid solution (200 g/*l*), 25 ml of ammonium acetate solution (500 g/*l*), and 5 ml of hydroxylammonium chloride solution (100 g/*l*), and add water to make about 80 ml, followed by sufficiently shaking(3). Add 10 ml of 1,10-phenanthroline solution (2 g/*l*), add water up to the marked line, and color it by shaking.

Note(3) The solution which has been treated according to (1) to (4) gives pH value of 4.4±0.2. When changing the quantity of solution fractioned at (3), it is needed to adjust its pH value.

(5) After about 20 min. standing, take a part of this solution into a 10 mm absorption cell of an absorptiometric analyzer, and measure its absorbance in the vicinity of 510 nm wavelength according to 6. of JIS K 0115 while setting blank test solution which has been treated as shown in (1) to (4) as a contrast solution.

(6) Employ titanium dioxide containing iron as little as possible (0.001% or less as Fe) for making working curve, carry out the procedures from (1) to (3), take stepwise from 0 ml to 6 ml of iron reference solution (5 μg Fe/ml) into some 100 ml measuring flasks, then carry out the procedures (4) and (5) respectively, and plot the working curve showing the relation between the amount of iron and absorbance.

5.5.5 Calculation Iron shall be calculated according to the following formula.

$$E = \frac{G}{S \times H} \times 100$$

where, E : iron (%)

G : amount of iron found on the working curve (g)

S : mass of sample (g)

H : aliquot ratio of sample solution

5.6 Calcium (Ca)

5.6.1 Summary Add hydrochloric acid and water into sample, heat to make it boil, extract calcium, filtrate it, and find the amount of calcium contained in solution by atomic absorption spectrometry.

5.6.2 Apparatus Apparatus shall be as follows.

(1) Thermostatic dryer Follow 5.3.2(1).

(2) Atomic absorption spectrometer Specified in JIS K 0121.

5.6.3 Reagents Reagents shall be as follows.

(1) Hydrochloric acid (1+1) Prepared using guaranteed reagent specified in JIS K 8180.

(2) Calcium reference solution (0.05 mgCa/ml) Weigh 2.497 g of calcium carbonate specified in JIS K 8617, dissolve it in 30 ml of hydrochloric acid (1+1), transfer it into a 1000 ml measuring flask, and add water up to the marked line. When it is to be used, take 25 ml of this solution using a transfer pipet into a 500 ml measuring flask, add water up to the marked line, and shake it to prepare reference solution.

5.6.4 Operation Operation shall be as follows.

(1) Dry sample in a thermostatic dryer adjusted at 105±2°C for about 2 h, and allow it to cool in a desiccator.

(2) Weigh its 1 g to the nearest 1 mg, and put it into a 300 ml beaker.

(3) Add 20 ml of hydrochloric acid (1+1) and 100 ml of water, cover it with a watch glass, and boil gently it on a sand bath for about 1 h to extract calcium.

(4) After cooling it, filtrate it through filter paper 5 grade C, transfer the filtrate into a 250 ml measuring flask, and add water up to the marked line.

(5) Take stepwise from 0 ml to 50 ml of calcium reference solution (0.05 mgCa/ml) into some 250 ml measuring flasks, respectively add 20 ml of hydrochloric acid (1+1), add water up to the marked line, shake it, and prepare the solution for making a working curve.

(6) Employ an atomic absorption spectrometer, measure absorbance of the solution obtained at (4) and (5) with 422.7 nm wavelength under the condition of acetylene-dinitrogen monooxide according to 6. of JIS K 0121.

(7) Plot the working curve showing relation between amount of calcium in solution at (5) and absorbance.

5.6.5 Calculation Calcium shall be calculated according to the following formula.

$$J = \frac{K}{S} \times 100$$

where, J : calcium (%)

K : amount of calcium found on the working curve (g)

S : mass of sample (g)

5.7 Water content

5.7.1 Summary Dry sample in a dryer, and weigh its loss to find water content.

5.7.2 Apparatus and implement Apparatus and implement shall be as follows.

(1) Flat-type weighing bottle 50 mm × 30 mm

(2) Thermostatic dryer Follow 5.3.2(1).

5.7.3 Operation Operation shall be as follows.

(1) Place uniformly about 2 g of sample on the bottom of a flat-type weighing bottle previously dried and weighed to the nearest 0.1 mg, to make its thickness 5 mm or less, stopper it, and weigh its mass to the nearest 0.1 mg.

(2) Remove the stopper, dry the weighing bottle and stopper in a thermostatic dryer adjusted at 105±2°C for about 2 h.

(3) Allow it to cool in a desiccator, and weigh its mass to the nearest 0.1 mg, to obtain its loss.

5.7.4 Calculation Water content shall be calculated according to the following formula.

$$L = \frac{M}{S} \times 100$$

where, L : water content (%)

M : loss (g)

S : mass of sample (g)

5.8 Ignition loss

5.8.1 Summary Heat strongly sample which has been dried, weigh its loss, and find the ignition loss.

5.8.2 Apparatus Apparatus shall be as follows.

(1) Thermostatic dryer Follow 5.3.2(1).

(2) Electric furnace Capable of heating at 900°C to 950°C.

(3) Crucible B type 15 ml specified in JIS R 1301.

5.8.3 Operation Operation shall be as follows.

(1) Dry sample in a thermostatic dryer adjusted at 105±2°C for about 2 h, and allow it to cool in a desiccator.

(2) Heat strongly a crusible alone in an electric furnace kept at 900°C to 950°C for about 2 h, and after standing it in a desiccator for cooling, weigh its mass to the nearest 0.1 mg.

(3) Weigh 2 g of sample, which has been dried and cooled according to (1), to the nearest 0.1 mg into the crucible, and heat it strongly in an electric furnace kept at 900°C to 950°C for about 2 h.

(4) After allowing it to cool in a desiccator, weigh its mass to the nearest 0.1 mg, and obtain its loss.

5.8.4 Calculation Ignition loss shall be calculated according to the following formula.

$$N = \frac{O}{S} \times 100$$

where, N : ignition loss (%)

O : loss of sample (g)

S : mass of sample (g)

5.9 Insoluble by sulfuric acid

5.9.1 Summary Add sulfuric acid and ammonium sulfate in sample, heat them to dissolve the sample, filtrate it to get residue, heat it strongly, and obtain the insoluble component by sulfuric acid.

5.9.2 Apparatus Apparatus shall be as follows.

(1) Thermostatic dryer Follow 5.3.2(1).

(2) Electric furnace Follow 5.8.2(2).

(3) Crucible Follow 5.8.2(3).

5.9.3 Reagents Reagents shall be as follows.

(1) Sulfuric acid Specified in JIS K 8951.

(2) Ammonium sulfate Specified in JIS K 8960.

(3) Sulfuric acid (1+20) Prepared using sulfuric acid specified in (1).

5.9.4 Operation Operation shall be as follows.

(1) Dry sample in a thermostatic dryer adjusted at 105±2°C for about 2 h, and allow it to cool in a desiccator.

(2) Weigh its about 3 g to the nearest 0.1 mg, and put it in a 500 ml beaker.

(3) Add about 30 g of ammonium sulfate and 75 ml of sulfuric acid, cover it with a watch glass, heat it to dissolve while swirling the beaker, and allow it to cool.

(4) While observing not to raise its temperature exceeding 50°C, add about 300 ml water and shake it, filtrate it through filter paper 5 grade C, and wash it with sulfuric acid (1+20).

(5) Place the residue together with the filter paper in a crucible, and heat gradually the filter paper to make it ash.

(6) Transfer the ash into a 250 ml conical beaker with a feather brush, add about 10 g of ammonium sulfate and 25 ml of sulfuric acid, heat it for dissolving, and then allow it to cool.

(7) Add 75 ml of water, filtrate it, wash it with sulfuric acid (1+20) to remove salts, and then wash with water to get rid of acid.

(8) Heat strongly a crucible in an electric furnace at 900°C to 950°C for about 2 h, allow it to cool in a desiccator, and weigh its mass to the nearest 0.1 mg.

(9) Put the residue together with filter paper in this crucible, heat at low temperature at first in an electric furnace to make ash, then raise its temperature gradually up to 900°C to 950°C, and heat strongly for about 30 min.

(10) After cooling it in a desiccator, weigh its mass to the nearest 0.1 mg, and find the mass of residue by means of subtracting the mass of crucible.

(11) Carry out the procedures from (3) to (9) to get blank test value.

5.9.5 Calculation The insoluble component by sulfuric acid shall be calculated according to the following formula.

$$P = \frac{Q - Q'}{S} \times 100$$

where, P : insoluble by sulfuric acid (%)

Q : mass of residue (g)

Q' : mass of residue given by blank test (g)

S : mass of sample (g)

5.10 Residue on sieve

5.10.1 Summary Moisten sample with ethanol, add water in it and agitate, and tilt the beaker on a sieve. Repeat this procedure several times, put the sieve in an evaporating dish, add water in it, and sweep slightly the net with a brush. Repeat this procedure, dry the sieve, and find residue on sieve.

5.10.2 Apparatus and implements Apparatus and implements shall be as follows.

(1) Brush Flat brush with soft hair, measuring about 25 mm in hair length and 15 mm in width.

(2) Net sieve Nominal size 45 μm specified in JIS Z 8801, with frame size of 75 mm in inside diameter at upper part of sieve surface (hereafter, referred to as "sieve").

(3) Evaporating dish Flat bottom type with 120 mm in diameter, specified in JIS R 1302.

(4) Thermostatic dryer Follow 5.3.2(1).

5.10.3 Reagents Reagents shall be as follows.

(1) Ethanol (95) Specified in JIS K 8102.

(2) Diethyl ether Specified in JIS K 8103.

5.10.4 Operation Operation shall be as follows.

(1) Dry the sieve in a thermostatic dryer adjusted at 105±2°C for about 30 min, allow it to cool in a desiccator, and weigh its mass.

(2) Dry it for another about 15 min, allow it to cool in a desiccator, and weigh its mass.

(3) Repeat this procedure, and record the mass given when the difference between 2 weighings reaches 1 mg or less.

(4) Weigh about 50 g of sample to the nearest 0.1 g into a 1000 ml beaker.

(5) Add ethanol (95) enough to moisten the sample, and press it slightly with a glass rod to crush lumps.

(6) Add 50 ml of water to mix sufficiently, and tilt the beaker to pour out the suspension on the sieve which has been put on a beaker. Moisten both surfaces of the net of the sieve with ethanol (95) in advance.

(7) Again pour about 50 ml of water on the remaining sample, and tilt similarly the beaker on the sieve. Repeat this procedure several times until whole sample is transferred on the sieve.

(8) While pouring a little quantity of water, pass nearly whole quantity of the sample through the sieve by means of vibrating it.

(9) Place the sieve in an evaporating dish, and put water to make its level 15 mm above the sieve net.

(10) Sweep slightly on the sieve with a brush. Make the sweeping rate once per second, lift it up from the evaporating dish every 20 trials to make the water flow down from the sieve mesh, and change water in the evaporating dish every 40 trials.

(11) Repeat this procedure until the sample cannot be recognized visually in the water kept in the evaporating dish, wash solid attached on the brush with water down onto the sieve, wash the sieve with ethanol (95) sufficiently, and finally wash it with diethyl ether.

(12) Dry the sieve in a thermostatic dryer adjusted at 105±2°C for about 30 min, allow it to cool in a desiccator, and weigh its mass.

(13) Again, dry it for about 15 min, allow it to cool in a desiccator, and weigh its mass.

(14) Repeat this procedure, record the mass of the sieve given when the difference between 2 weighings reaches 1 mg or less, and subtract the mass of the sieve which has been recorded at (3), to find the mass of residue.

5.10.5 Calculation The residue on a sieve shall be calculated according to the following formula.

$$R = \frac{T}{S} \times 100$$

where, R : residue on a sieve (%)

T : mass of residue (g)

S : mass of sample (g)

5.11 Number of coarse grains

5.11.1 Summary Add water in sample to stir them, add polyvinyl alcohol solution to make sample pasty, and find the number of coarse grains under a microscope.

5.11.2 Apparatus and implement Apparatus and implement shall be as follows.

(1) Microscope Biological microscope capable of adjusting magnifications from 200 to 400.

(2) Mortar Made of glass or agate, with 90 mm outside diameter.

5.11.3 Reagent Reagent shall be as follows.

Polyvinyl alcohol solution Add 89 ml of water in 11.0 g of polyvinyl alcohol on the market, with from 1500 to 1800 molecular weight, heat it up to 60°C to 80°C, and dissolve the alcohol. Employ the solution whose falling-ball viscosity is from 15 s to 20 s at 20°C.

The measurement of falling-ball viscosity shall be as follows.

Fill polyvinyl alcohol solution in a viscosity measuring tube(4) up to a definite height over an upper marked line, stand it vertically in a thermostat at 20±1°C, allow it to stand for about 1 h, drop the steel ball measuring 3.17500 mm in nominal diameter specified in JIS B 1501 from the center inlet of the viscosity measuring tube, and measure seconds by which the ball passes between upper and lower marked lines using a stopwatch.

Note(4) Glass tube measuring 20 mm in inside diameter and 300 mm in length, with 2 marked lines, that is, one is at about 50 mm above from its bottom and the other is at accurately 200 mm upper than it.

5.11.4 Operation Operation shall be as follows.

(1) Weigh about 1 g of sample to the nearest 0.1 mg into a mortar.

(2) Add 1.5 ml of water, and knead with a pestle for 5 min at the rate of 120 times per minute.

(3) Add 10 g of polyvinyl alcohol solution, weigh it to the nearest 10 mg, and knead it again to make it pasty.

(4) Into another mortar, put about 0.1 g of the pasty using a glass rod, weigh its mass to the nearest 0.1 mg, add 19.9 g of polyvinyl alcohol solution, weigh its mass to the nearest 10 mg, and knead it with a pestle until it gives uninformness.

(5) Place its 2.5 mg on a deck glass for a microscope with a glass rod, weigh it to the nearest 0.1 mg, put a cover glass on it to make sample's diameter about 10 mm, and perpare the sample for a microscopic examination.

(6) Count the number of coarse grains which has 5 µm or longer in major diameter under the microscope, and obtain the average of 3 test samples for a microscopic examination.

5.12 Whiteness

5.12.1 Summary Press sample into a cell, make its surface flat, and measure its whiteness using a photoelectric colorimeter.

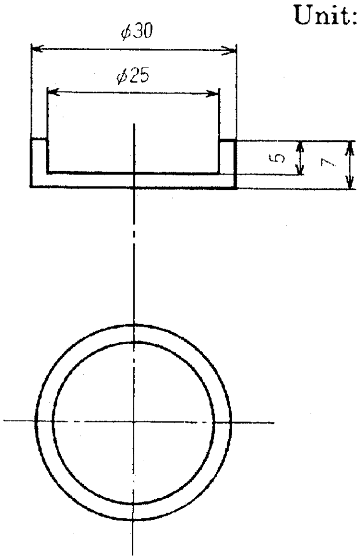
5.12.2 Apparatus and implement Apparatus and implement shall be as follows.

(1) Photoelectric colorimeter Specified in 5.2 of JIS Z 8722.

(2) Cell Its example is shown in Fig. 2.

Fig. 2. Example of cell

Unit: mm



5.12.3 Operation Operation shall be as follows.

(1) Take about 10 g of sample on a cell, place a glass plate on it, press it strongly with a finger tip to make sample surface the same level as that of the cell, and then remove the glass plate while sliding it along the upper sample surface on the cell. Make thickness of the sample 5 mm or thicker.

(2) Employ a photoelectric colorimeter, measure Y value on sample surface according to 5. of JIS Z 8722, and adopt this value as whiteness of the sample.

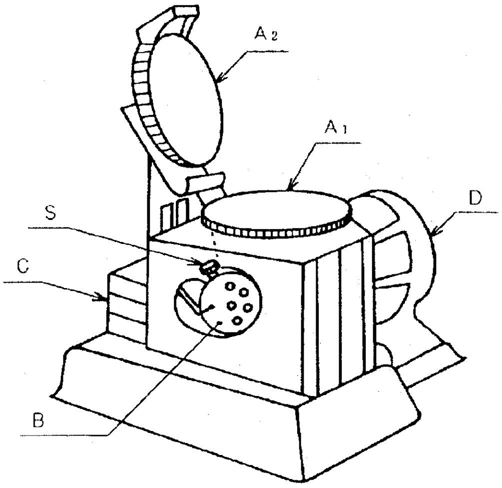
5.13 Tinting strength

5.13.1 Summary Add auxiliary pigment and boiled linseed oil in sample, and knead them until it gives uniform color. Treat similarly evaluation sample. And roll sample and evaluation sample flat on a colorless transparent glass plate side by side, and compare their tinting strength by naked eyes observing from surface or back surface of the glass plate, under scattered daylight.

5.13.2 Apparatus and implements Apparatus and implements shall be as follows.

(1) Hoover-type muller Consisting of glass plates measuring 180 mm to 200 mm in diameter and capable of setting about 100 revolutions min^{-1} {rpm} and of pre-settable 25 or its multiple of number of revolutions. Fig. 3 shows its example.

Fig. 3. Example of Hoover-type muller



A_1 : Plate for kneading (180 mm to 200 mm in diameter)

A_2 : Plate for kneading (180 mm to 200 mm in diameter)

B : Adjusting device of number of revolutions

C : Dead weight for pressure adjustment

D : Motor

S : Switch

(2) Spatula Knife type or straight-edge type, made of steel.

(3) Glass plate Colorless transparent, measuring about 2 mm in thickness.

5.13.3 Material Material shall be as follows.

(1) Auxiliary pigment Carbon black, consisting of grains with about 75 nm diameter, specified in JIS K 5107.

(2) Boiled linseed oil Boiled linseed oil specified in JIS K 5421.

(3) Evaluation sample This is used for evaluation of quality, and shall be titanium dioxide either of the lot which has been specified by manufacturer for the purpose of quality control or of the lot which has been agreed between parties concerned.

5.13.4 Operation Operation shall be as follows.

(1) Weigh 2 g of sample and 0.02 g of auxiliary pigment to the nearest 0.1 mg.

(2) Put them center of the lower kneading plate of Hoover-type muller, add 1.0 ml of boiled linseed oil, knead slightly them with a spatula.

(3) Then, put an upper kneading plate, and knead it 4 times by 50 revolutions per once, using 3 dead weights (totally about 68 kg mass) for pressure adjustment.

(4) Separately, carry out the procedures from (1) to (3) on evaluation sample.

(5) Apply these both on a glass plate side by side.

(6) Compare them observing from surface or back surface of the glass plate by naked eyes, under scattered daylight(5).

Note(5) Scattered daylight means the light from northern window avoiding direct sunshine, obtained during from 3 h later than sunrise to 3 h earlier than sunset. The standard light C, however, specified in 3.1 of JIS Z 8720, may be used as a scattered light.

(7) When both colors do not show coincidence, they shall be mated by adjusting the amount of auxiliary pigment to be added in sample.

5.13.5 Calculation Tinting strength shall be calculated according to the following formula.

$$U = \frac{V}{0.020} \times W$$

where, U : tinting strength

V : mass of auxiliary pigment added into sample (g)

0.020 : mass of auxiliary pigment taken at first (g)

W : value of tinting strength of evaluation sample

5.14 Degree of in-water dispersion

5.14.1 Summary Disperse sample in water, put it into a cylinder for dispersion measurement, allow it to stand for specified duration, and obtain the degree of dispersion making use of the concentration difference before and after standing.

5.14.2 Apparatus and implements Apparatus and implements shall be as follows.

(1) Vibrator With 100 mm of amplitude and 90 min^{-1} {rpm} of number of vibrations.

(2) Thermostatic dryer Capable of adjusting at 140±10°C.

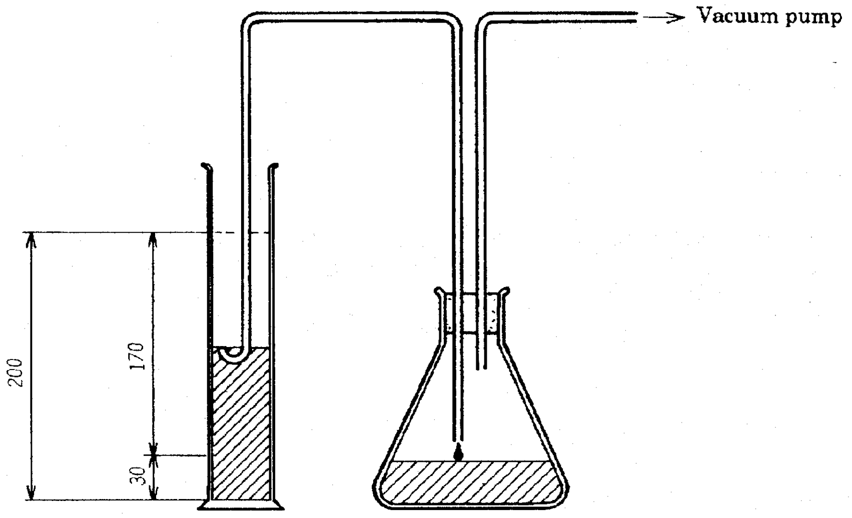
(3) Thermostatic water tank Capable of adjusting at 20±1°C.

(4) Cylinder for dispersion measurement Measuring 37±2 mm in inside diameter and 250 mm in height, and having 2 marked lines at both 30 mm above and 200 mm above from its bottom.

(5) Device for sucking dispersion liquid Fig. 4 shows its example.

Fig. 4. Example of device for sucking dispersion liquid

Unit: mm



5.14.3 Operation Operation shall be as follows.

(1) Weigh 15 g of sample into a weighing bottle to the nearest 0.1 g, transfer it into a 500 ml Erlenmeyer flask in which 285 g of water has previously been put, stir it for 20 min with a vibrator, and disperse sample in water.

(2) Take 20 ml of this dispersion liquid into a 20 ml beaker whose mass has previously been measured to the nearest 0.1 mg, and immediately weigh its mass to the nearest 0.1 mg, to let it be Y_1.

(3) Place this in a thermostatic dryer kept at 140±10°C, dry it for about 6 h to evaporate water, transfer it into a desiccator to cool it down to room temperature, and weigh its mass to the nearest 0.1 mg, to let it be Y_2.

(4) Pour remnant dispersion liquid into a cylinder for dispersion measurement to get 200 mm high liquid column, and allow it to stand in a thermostatic water tank for about 5 h.

(5) Employ a device for sucking dispersion liquid, and suck the dispersion liquid which has stood for about 5 h into a 500 ml Erlenmeyer flask equipped with a vacuum pump. Carry out the sucking under the condition that sucking inlet is nearly contacting to liquid surface, and complete sucking when liquid level reaches 30 mm high from the bottom.

(6) Shake the liquid sucked in, take its 20 ml into a 20 ml beaker whose mass has previously been measured to the nearest 0.1 mg, and immediately weigh its mass to the nearest 0.1 mg, to let it be Y_3.

(7) Place this in a thermostatic dryer kept at 140±10°C, dry it for about 6 h to evaporate water, transfer it into a desiccator to cool it down to room temperature, and weigh its mass to the nearest 0.1 mg, to let it be Y_4.

5.14.4 Calculation The degree of in-water dispersion shall be calculated according to the following formula.

$$X = \frac{Y_4}{Y_3} \times \frac{Y_1}{Y_2} \times 100$$

where, X : degree of in-water dispersion (%)

Y_1 : mass of dispersion liquid before standing (g)

Y_2 : mass of dispersion liquid before standing, after drying (g)

Y_3 : mass of dispersion liquid after standing (g)

Y_4 : mass of dispersion liquid after standing, after drying (g)

6. Inspection The inspection, when being tested according to 5., shall conform to the requirements in Table 1.

7. Marking The following items shall be marked on the container of titanium dioxide for chemical fiber.

(1) Title of Standard
(2) Classification
(3) Net mass
(4) Manufacture number or lot number
(5) Name of manufacturer or its abbreviation

Attached Table 1. Cited standards

JIS B 1501	Steel balls for ball bearings
JIS H 4040	Aluminium and aluminium alloy rods, bars and wires
JIS H 6202	Platinum dishes for chemical analysis
JIS K 0016	Iron standard solution
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 5107	Carbon black (pigment)
JIS K 5421	Boiled oil and boiled linseed oil
JIS K 8001	General rule for test methods of reagents
JIS K 8005	Reference materials for volumetric analysis
JIS K 8051	3-Methyl-1-butanol
JIS K 8102	Ethanol (95)
JIS K 8103	Diethyl ether
JIS K 8180	Hydrochloric acid
JIS K 8201	Hydroxylammonium chloride
JIS K 8359	Ammonium acetate
JIS K 8372	Sodium acetate
JIS K 8532	L(+)-Tartaric acid
JIS K 8541	Nitric acid
JIS K 8617	Calcium carbonate
JIS K 8622	Sodium hydrogen carbonate
JIS K 8625	Sodium carbonate
JIS K 8637	Sodium thiosulfate pentahydrate
JIS K 8658	Starch
JIS K 8789	1,10-Phenanthroline monohydrate
JIS K 8819	Hydrofluoric acid
JIS K 8863	Boric acid
JIS K 8913	Potassium iodide
JIS K 8951	Sulfuric acid
JIS K 8960	Ammonium sulfate

Attached Table 1. Cited standards (continued)

JIS K 8982	Ammonium iron (III) sulfate 12-water
JIS K 9001	Potassium thiocyanate
JIS R 1301	Porcelain crucibles for chemical analysis
JIS R 1302	Porcelain basins for chemical analysis
JIS Z 8401	Rules for rounding off of numerical values
JIS Z 8720	Standard illuminants and sources for colorimetry
JIS Z 8722	Methods of colour measurement - Reflecting or transmitting objects
JIS Z 8801	Test sieves
JIS Z 8802	Methods for determination of pH of aqueous solutions
JIS Z 8805	Glass electrodes for measurement of pH

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