

ERRATA

Page 1, the last line except one

Error: ...the size expressed with resistance

Correct: ...the sphere equivalent value

Page 16, Sub-clause 5.1.2 (3)

Error: ...the testing method of electric resistance

Correct: ...the electro-sensing zone method

Page 17

Replace the Table 22 to the following table:

Table 22. Specific surface area of white fused alumina

Class	Specific surface area m^2/g
No.1	4.2 to 4.5
No.2	1.4 to 1.7
No.3	0.65 to 0.9
No.4	0.35 to 0.45
No.5	0.10 to 0.20
No.6	0.05 to 0.10

Remarks: This erratum is for correcting the first edition of this Standard.

Japanese Standards Association

JIS

JAPANESE INDUSTRIAL STANDARD

Test powders and test particles

JIS Z 8901—1995

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JAPANESE INDUSTRIAL STANDARD

J I S

Test powders and test particles

Z 8901-1995

1. Scope This Japanese Industrial Standard specifies both: the test powders⁽¹⁾ which can be used for the dust separation test of a dust separator or an air filter, for the function test of various testers, and for wear test; and the test particles⁽²⁾ which can be used for the calibration of light-scattering automatic particle counter, the test of particle collecting rate for super high performance air filter, and the calibration of an automatic counter of particulate materials floating in air.

Notes (1) Test powders Powders are classified into test powders 1 and test powers 2, and powders 1 is for the separation test of a dust separator or air filter, for the function test of various testers, has a constant characteristics, and has typical characteristics of general powder.

Test powders 2 is for the verification of apparatus relating to powder and for the operating function test and the wear test of various testers against powders, has a constant characteristics, and has typical characteristics of general powder.

(2) Test particles Test particles are classified into test particles 1 and test particles 2, and test particles 1 is polystyrene group particles. Particles 1 mainly consists of styrene, and is monodispersed system which has the same sized solid particles and synthesized by emulsion polymerization. Usually, they are on the market as the type of concentrated suspension and are diluted with ultra pure water to make suitable concentration. Otherwise, they are sprayed or mixed with dry air to evaporate water, and prepared for fine particles for test.

Test particles 2 is fine particles for test which has been generated from dioctyl phthalate and stearic acid using an aerosol generator.

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

2. Definitions The definition of terms in this Standard shall be as follows except those defined in JIS Z 8103 and JIS Z 8122.

(1) particle size It is expressed by any of, the sieve opening of a test sieve measured by screening method, Stokes' equivalent diameter tested by sedimentation method, the circular equivalent diameter tested by a microscope method, or the sphere equivalent diameter tested by light scattering method, otherwise the size expressed with resistance tested by electric resistance testing method. It is also called grain size.

- (2) particle size distribution The distribution corresponding to particle size of particles consisting particle group.

Remarks: Generally, every type of powders, including the test powders, doesn't consist of the same sized particles, and there exist various sized particles, from the minimum diameter to the maximum diameter, with various mixing rate.

- (3) oversize In the particle size distribution of powders, percentage of particle group, having larger size than some particle sizes, to whole powders.
- (4) median diameter In the particle size distribution of powders, such particle sizes that the number or mass of powders which are larger than some particle sizes occupies 50 % of whole powders.
- (5) particle density The value obtained by division of the mass of whole powders by the real volume of the powders.
- (6) apparent density The value obtained by division of the mass of powders by the volume occupied by them.
- (7) mean particle size Averaged diameter of particle sizes which were photographed by optical microscope method or transmission electron microscope method.
- (8) CV value The value obtained by division of standard deviation by mean particle size, which expresses relatively dispersion.

$$CV \text{ value} = \text{standard deviation} / \text{mean particle size} \times 100 (\%)$$

Remarks: It is also called coefficient of variation.

3. Classification Test powders 1 shall be classified into 15 classes as shown in Table 1, and test powders 2 into 3 classes (13 classes by grading) as shown in Table 2; and test particles 1 into 12 classes as shown in Table 3, and test particles 2 into 2 classes as shown in Table 4.

Table 1. Classes of test powers 1

Classification	Material used	Range of median diameter (on mass basis) μm
Class 1	Quartz sand	185 to 200
Class 2	Quartz sand	27 to 31
Class 3	Quartz sand	6.6 to 8.6
Class 4	Talc	7.2 to 9.2
Class 5	Fly ash	13 to 17
Class 6	Ordinary Portland cement	24 to 28
Class 7	KANTO (Japanese) loam	27 to 31
Class 8	KANTO (Japanese) loam	6.6 to 8.6
Class 9	Talc	4.0 to 5.0

Table 1. (continued)

Classification	Material used	Range of median diameter (on mass basis) μm
Class 10	Fly ash	4.8 to 5.7
Class 11	KANTO (Japanese) loam	1.6 to 2.3
Class 12	Carbon black	-
Class 13	-	-
Class 14	-	-
Class 15	Mixture of test powders Class 8, Class 12, and cotton linter	-
Class 16	Calcium carbonate heavy	3.6 to 4.6
Class 17	Calcium carbonate heavy	1.9 to 2.4

Table 2. Classes of test powders 2

Classification	Material used	Range of median diameter (on mass basis) μm
GBL 30	Glass beads	29 to 31
GBL 40		40 to 42
GBL 60		58 to 60
GBL 100		99 to 101
GBM 20	Glass beads	21 to 23
GBM 30		29 to 31
GBM 40		40 to 42
No. 1	White fused alumina	1.6 to 2.5
No. 2		3.5 to 4.5
No. 3		7.4 to 8.6
No. 4		13.0 to 15.0
No. 5		28.0 to 32.0
No. 6		54.0 to 60.0

Table 3. Classes of test particles 1

Classifi- cation	Nominal diameter μm
No. 1	0.05
No. 2	0.08
No. 3	0.1
No. 4	0.2
No. 5	0.3
No. 6	0.5
No. 7	1
No. 8	2
No. 9	5
No. 10	10
No. 11	15
No. 12	25

Table 4. Classes of test particles 2

Classifi- cation	Material used	Range of median diameter (on pieces basis) μm
Class 1	Diocetyl phthalate	0.21 to 0.32
Class 2	Stearic acid	0.25 to 0.35

4. Test powders 1

4.1 Quality The quality of test powders 1 shall conform to the following items.

4.1.1 Test powders Class 1, Class 2 and Class 3 The quality of test powders Class 1, Class 2, and Class 3 (quartz sand) shall conform to the following items.

(1) Chemical composition The chemical composition shall meet the requirements shown in Table 5 when measured according to the methods in 4.4.2 (1).

Table 5. Chemical composition of Class 1, Class 2 and Class 3

Composition	Mass percentage %
SiO ₂	97 min.
Fe ₂ O ₃	} 3 max. in total
Al ₂ O ₃	
TiO ₂	
MgO	
Ignition loss	

- (2) Particle density Particle density shall be the value of 2.6 g/cm³ to 2.7 g/cm³ when measured according to the method in 4.4.3.

4.1.2 Test powders Class 4 and Class 9 The quality of test powders Class 4 and Class 9 (talc) shall conform to the following items.

- (1) Chemical composition The chemical composition shall meet the requirements shown in Table 6 when measured according to the methods in 4.4.2 (1).

Table 6. Chemical composition of Class 4 and Class 9

Composition	Mass percentage %
SiO ₂	60 to 63
Fe ₂ O ₃	0 to 3
Al ₂ O ₃	0 to 3
CaO	0 to 2
MgO	30 to 34
Ignition loss	3 to 7

- (2) Particle density Particle density shall be the value of 2.7 g/cm³ to 2.9 g/cm³ when measured according to the method in 4.4.3.

4.1.3 Test powders Class 5 and Class 10 The quality of test powders Class 5 and Class 10 (fly ash) shall conform to that of fly ash specified in JIS A 6201.

4.1.4 Test powders Class 6 The quality of test powders Class 6 (ordinary Portland cement) shall conform to that of Portland cement specified in JIS R 5210.

4.1.5 Test powders Class 7, Class 8, and Class 11 The quality of test powders Class 7, Class 8, and Class 11 (KANTO loam) shall conform to the following items.

- (1) Chemical composition The chemical composition shall meet the requirements shown in Table 7 when measured according to the methods in 4.4.2 (1).

Table 7. Chemical composition of Class 7, Class 8 and Class 11

Composition	Mass percentage %
SiO ₂	34 to 40
Fe ₂ O ₃	17 to 23
Al ₂ O ₃	26 to 32
CaO	0 to 3
MgO	3 to 7
TiO ₂	0 to 4
Ignition loss	0 to 4

- (2) Particle density Particle density shall be the value of 2.9 g/m³ to 3.1 g/cm³ when measured according to the method in 4.4.3.

4.1.6 Test powders Class 12 The quality of test powders Class 12 (carbon black) shall conform to the following items.

- (1) Physicochemical characteristics Physicochemical characteristics shall meet the requirements shown in Table 8 when measured according to the methods in JIS K 6221. Provided that granulated carbon black shall be excluded.

Table 8. Physicochemical characteristics of Class 12

Physicochemical characteristics	Mass percentage %
Absorbed amount of DBP	25 to 34
Adsorbed amount of iodine	22 to 30

4.1.7 Test powders Class 15 The quality of test powders Class 15 (mixed material of test powders Class 8, test powders Class 12, and cotton linter) shall meet the requirements shown in Table 9 when the mixture of test powders Class 8, test powders Class 12, and cotton linter (1.5 μm in diameter, and 1 mm or less in length) is measured according to the method in 4.4.5.

Table 9. Quality of Class 15

Employed powder	Mass percentage %
Test powders Class 8	72
Test powders Class 12	23
Cotton linter	5

4.1.8 Test powders Class 16 and Class 17 The quality of test powders Class 16 and Class 17 (calcium carbonate heavy) shall conform to the following items.

- (1) Chemical composition The chemical composition shall meet the requirements shown in Table 10 when measured according to the methods in 4.4.2 (3).

Table 10. Chemical composition of Class 16 and Class 17

Composition	Mass percentage %
CaO	54 to 56
MgO	0 to 3
SiO ₂	0 to 4
Al ₂ O ₃	0 to 3
Fe ₂ O ₃	0 to 1
Ignition loss	42 to 45

- (2) Particle density Particle density shall be the value of 2.7 g/cm³ to 2.8 g/cm³ when measured according to the method in 4.4.3.

4.2 Particle size distribution

4.2.1 Test powders Class 1, Class 2 and Class 3 The particle size distribution of each Class shall meet the requirements in Table 11 when test powders Class 1 is measured according to the method of 4.4.4 (1), when test powders Class 2 or Class 3, whose particle size is 75 µm or more, is measured according to the method of 4.4.4 (1), and those, whose particle size is less than 75 µm, is measured according to the method of 4.4.4 (2).

Table 11. Particle size distribution of Class 1, Class 2 and Class 3

Particle size µm	Oversize (on mass basis) %		
	Class 1	Class 2	Class 3
5	—	88 ± 5	61 ± 5
10	—	76 ± 3	43 ± 3
20	—	62 ± 3	27 ± 3
30	—	50 ± 3	15 ± 3
40	—	39 ± 3	9 ± 3
45	100	—	—
75	90 ± 3	20 max.	3 max.
106	80 ± 3	—	—
150	65 ± 3	—	—
212	45 ± 3	—	—
300	0	—	—

4.2.2 Test powders Class 4 and Class 9 The particle size distribution of test powders Class 4 and Class 9 shall meet the requirements in Table 12 when measured according to the method in 4.4.4 (2).

Table 12. Particle size distribution of Class 4 and Class 9

Particle size μm	Oversize (on mass basis) %	
	Class 4	Class 9
2	—	79 ± 5
4	—	55 ± 5
5	69 ± 5	—
8	—	23 ± 5
10	40 ± 5	—
16	—	6 ± 3
20	12 ± 5	—
30	—	—
40	3 ± 3	—
75	0	—

4.2.3 Test powders Class 5 and Class 10 The particle size distribution of test powders Class 5 and Class 10 shall meet the requirements in Table 13 when the powders with particle size of $106 \mu\text{m}$ or more are measured according to the method in 4.4.4 (1) and the powders with particle size less than $106 \mu\text{m}$ are measured according to the method in 4.4.4 (2).

Table 13. Particle size distribution of Class 5 and Class 10

Particle size μm	Oversize (on mass basis) %	
	Class 5	Class 10
2	—	82 ± 5
4	—	60 ± 5
5	84 ± 5	—
8	—	22 ± 3
10	60 ± 5	—
16	—	3 ± 3
20	32 ± 3	—
30	15 ± 3	—
40	8 ± 3	—
106	0	—

4.2.4 Test powders Class 6 The particle size distribution of test powders Class 6 shall meet the requirements in Table 14 when the powders with particle size of $75 \mu\text{m}$ or more are measured according to the method in 4.4.4 (1) and the powders with particle size less than $75 \mu\text{m}$ are measured according to the method in 4.4.4 (2).

Table 14. Particle size distribution of Class 6

Particle size μm	Oversize (on mass basis) %
5	88 ± 5
10	77 ± 3
20	60 ± 3
30	45 ± 3
40	33 ± 3
75	0

4.2.5 Test powders Class 7, Class 8 and Class 11 The particle size distribution of test powders Class 7, Class 8, and Class 11 shall meet the requirements in Table 15 when the powders with particle size of $75 \mu\text{m}$ or more are measured according to the method in 4.4.4 (1) and the powders with particle size less than $75 \mu\text{m}$ are measured according to the method in 4.4.4 (2).

Table 15. Particle size distribution of Class 7, Class 8 and Class 11

Particle size μm	Oversize (on mass basis) %		
	Class 7	Class 8	Class 11
1	—	—	65 ± 5
2	—	—	50 ± 5
4	—	—	22 ± 3
5	88 ± 5	61 ± 5	—
6	—	—	8 ± 3
8	—	—	3 ± 3
10	76 ± 3	43 ± 3	—
20	62 ± 3	27 ± 3	—
30	50 ± 3	15 ± 3	—
40	39 ± 3	9 ± 3	—
75	20 max.	3 max.	—

4.2.6 Test powders Class 12 The particle size of test powders Class 12 shall fall in the range from $0.03 \mu\text{m}$ to $0.20 \mu\text{m}$ when measured according to the method in 4.4.4 (3).

The particle size distribution is not prescribed particularly.

4.2.7 Test powders Class 16 and Class 17 The particle size distribution of test powders Class 16 and Class 17 shall meet the requirements in Table 16 when measured according to the method in 4.4.4 (2).

Table 16. Particle size distribution of Class 16 and Class 17

Particle size μm	Oversize (on mass basis) %	
	Class 16	Class 17
1	—	80 ± 5
2	76 ± 5	54 ± 5
4	—	18 ± 5
5	40 ± 5	—
8	—	4 ± 3
10	10 ± 5	—
16	—	0
20	0	—

4.3 Preparation method The preparation method of test powders shall be as follows.

- (1) Test powders Class 1, Class 2 and Class 3 Prepare the powders by grinding quartz sand in a ball mill, or by suitably compounding of quartz sand.
- (2) Test powders Class 4, Class 5, Class 9, Class 10 and Class 12 Prepare the powders by suitably compounding of talc, fly ash, or carbon black respectively.
- (3) Test powders Class 7, Class 8 and Class 11 Sinter KANTO loam at 800°C for 2 h, grind it in ball mill, and prepare the powders, otherwise after grinding the KANTO loam which has been sintered, suitably classify it, mix it, and prepare the powders.
- (4) Test powders Class 15 Mix KANTO loam, carbon black, and cotton linter in a powder mixer, and prepare the powders.
- (5) Test powders Class 16 and Class 17 Grind calcium carbonate heavy, classify it, suitably compound it, and prepare the powders.

4.4 Measuring method

4.4.1 Sampling The sampling method of sample for measurement shall follow the conically quartering method specified in JIS M 8100.

4.4.2 Chemical composition The measuring method of chemical composition shall be as follows.

- (1) The measurement of test powders Class 1, Class 2, Class 3 Class 4, Class 7, Class 8, Class 9 and Class 11, shall follow the prescription in JIS R 2212.
- (2) The measurement of test powders Class 5 and Class 10, shall follow the prescription in JIS A 6201.
- (3) The measurement of test powders Class 16 and Class 17, shall follow the prescription in JIS M 8850.

4.4.3 Particle density The measuring method of particle density shall be as follows.

(1) Instruments for use Instruments for use shall be as follows.

- (a) Pycnometer About 50 cm³ capacity.
- (b) Balance With reciprocal sensibility of 1/1000 g.
- (c) Thermostatic water bath Capable of keeping the sample in a pycnometer at specified temperature.

Remarks: For instance, capable of keeping at 20.0 ± 1°C.

(d) Vacuum degassing apparatus

(2) Sample Take about 10 g of sample, and dry it sufficiently.

(3) Procedures for measuring The procedures for measuring shall be as follows.

- (a) Mass of a pycnometer Weigh it which has been cleaned and dried.
- (b) Mass of the pycnometer with distilled water Fill distilled water in the pycnometer, make its temperature at θ °C by keeping it in a thermostatic water bath, and weigh swiftly its whole mass with no water drop on its outside.
- (c) Mass of the pycnometer with sample Place sample in the well dried pycnometer, which has been used in above-procedure, and weigh its whole mass.
- (d) Mass of the pycnometer with both distilled water and sample Place distilled water in the pycnometer by about half volume, and remove the gas and bubbles contained in the sample with often shaking by vacuum degassing method for about 20 min. After that, fill it with distilled water, make its temperature at θ °C by keeping it in a thermostatic water bath, and weigh swiftly its whole mass after wiping the water drops on its outside surface.

(4) Calculation Calculate the particle density ρ_p of powders to water at θ °C temperature, according to the following formula.

$$\rho_p = \frac{\rho (M_d - M_t)}{M + (M_d - M_t) - M_w}$$

where, ρ_p : particle density of powders (g/cm³)

ρ : density of water at θ °C temperature (g/cm³)

M : mass of pycnometer with distilled water (g)

M_d : mass of pycnometer with sample (g)

M_t : mass of pycnometer (g)

M_w : mass of pycnometer with both distilled water and sample (g)

12
Z 8901-1995

4.4.4 Particle size distribution Particle size distribution shall follow any one of the following three methods.

- (1) Screening method Screening method shall follow the specification in JIS Z 8815.
- (2) Sedimentation method Sedimentation method shall follow the specification in JIS Z 8821 or JIS Z 8822.

The marking method of measurement results shall follow the marking method of measurement results specified in JIS Z 8820.

Remarks: When adopting sedimentation method, other methods which give the equivalent particle size distribution to Andreasen pipette method or sedimentation balance method may be used.

- (3) Microscope method Microscope method shall be as follows.
 - (3.1) Instruments for use Instruments for use shall be as follows.
 - (a) Transmission electron microscope Having a resolution powerful enough to measure the minimum particle size.
 - (b) Tools for sample preparation of electron microscope One set
 - (3.2) Sample Use a little of sample which has been dried.
 - (3.3) Procedures for measuring The procedures for measuring shall be as follows.
 - (a) Disperse carbon black on a sheet mesh, on which carbon has been deposited by vacuum evaporation, owing to any one of a sprinkling method⁽³⁾, a paste method⁽⁴⁾, or a film-on-water method⁽⁵⁾, and prepare sample for an electron microscope.
- Notes (3) Sprinkling method Place carbon black of sample on the sustaining film, on which carbon has been deposited by vacuum evaporation, using absorbent cotton or a soft writing brush, loosen it lightly, and carry out either to directly observe as it is or to observe it after making again carbon deposition by vacuum evaporation.
- (4) Paste method Add a little of linseed oil into carbon black of sample, knead well them with a spatula to make paste, apply its modicum on a sustaining film on which carbon has been deposited by vacuum evaporation, immerse it in solvent specified in JIS K 8034 or JIS K 8271, and dissolve media (linseed oil) only, followed by washing.

(5) Film-on-water method Disperse sufficiently a hydrophobic carbon black in the dispersion medium specified in JIS K 8103, drip a little of it on water surface to let medium evaporate after it spreads on water, scoop up the particles, which are dispersed on water surface, with a mesh covered with sustaining film, and thus well dispersed sample can be completed.

(b) Select the resolution of electron microscope by which the particle with the minimum particle size can be observed, and prepare photographs by electron microscope.

(c) On the photographs, measure diameters of the maximum and minimum particles, and obtain the range of particle size.

4.4.5 Method of measuring mixing rate of Class 15 The method of measuring mixing rate of test powders Class 15 shall be as follows.

(1) Instruments for use Instruments for use shall be as follows.

(a) Electric furnace Capable of heating up to 1000°C.

(b) Crucible With about 30 cm³ capacity.

(c) Balance With reciprocal sensibility of 1/1000 g.

(2) Sample Use a little of sample which has been dried.

(3) Procedures for measuring The procedures for measuring shall be as follows.

(a) Put the sample of about 10 g in a crucible, place it in a desiccator, and weigh repeatedly it until it gets constant weight. Make this mass "A".

(b) Heat it in an electric furnace for about 1 h at 400°C, allow it to cool in a desiccator, weigh its mass, and make it "B". Subtract "B" from "A", and the mass of cotton linter can be obtained.

(c) Successively, heat the sample at (b) in an electric furnace at about 750°C for about 1 h, allow it to cool in a desiccator, and weigh its mass. Make this mass "C". Subtract "C" from "B", and the mass of carbon black can be obtained.

4.5 Cautions for handling When test powders 1 is used for various type of tests, unless otherwise specified, dry it sufficiently, disperse it as completely as possible, and submit it under no deteriorated condition.

4.6 Marking The containers of test powders 1 shall be legibly and indelibly marked with the following items.

(1) Class and material employed

Example: Test powders 1 Class 8 KANTO loam

14
Z 8901-1995

(2) Net mass

(3) Chemical composition

Example: Test powders 1 Class 8

Composition	Mass percentage %
SiO ₂	34 to 40
Fe ₂ O ₃	17 to 23
Al ₂ O ₃	26 to 32
CaO	0 to 3
MgO	3 to 7
TiO ₂	0 to 4
Ignition loss	0 to 4

(4) Particle density

Example: 2.9 g/cm³ to 3.1 g/cm³

(5) Particle size distribution or range of particle size

Example: Test powders 1 Class 8

Particle size μm	Oversize (on mass basis) %
5	61 $\pm$ 5
10	43 $\pm$ 3
20	27 $\pm$ 3
30	15 $\pm$ 3
40	9 $\pm$ 3
75	3 max.

(6) Name of manufacturer or its abbreviation

5. Test powders 2

5.1 Quality

5.1.1 Glass beads The quality of glass beads shall conform to the following items.

Remarks: The materials of glass beads shall be two kinds, that is, soda lime-silicate glass (GBL) and barium-titanate glass (GBM)⁽⁶⁾.

Note ⁽⁶⁾ Soda lime-silicate glass (GBL) and barium-titanate glass (GBM) have respectively different chemical composition and particle density.

- (1) Chemical composition The chemical composition of soda lime-silicate glass shall meet the requirements in Table 17 when measured according to the method specified in JIS R 3101. The chemical composition of balium-titanate glass shall meet the requirements in Table 18 when measured according to the method specified in JIS K 0121 or JIS R 3105.

Table 17. Chemical composition of soda lime-silicate glass

Composition	Mass percentage %
SiO ₂	70 to 73
Na ₂ O	12 to 15
CaO	7 to 12
MgO	1 to 5

Table 18. Chemical composition of balium-titanate glass

Composition	Mass percentage %
TiO ₂	33 to 38
BaO	41 to 48
SiO ₂	10 to 14
CaO	4 to 8

- (2) Particle density Particle density shall be from 2.1 g/cm³ to 2.5 g/cm³ for soda lime-silicate glass and from 4.0 g/cm³ to 4.2 g/cm³ for balium-titanate glass when measured according to measuring method for particle density specified in JIS R 3301.
- (3) Particle size distribution Particle size distribution shall meet the values stated in Table 19 and Table 20 when measured according to the testing method of electric resistance specified in JIS R 6002.

Table 19. Particle size distribution of soda lime-silicate glass

Unit: μm

Class	Particle size in case of 90 % oversize (mass basis)	Particle size in case of 50 % oversize (mass basis)	Particle size in case of 10 % oversize (mass basis)
GBL 30	26 min.	30 ± 1.0	34 max.
GBL 40	37 min.	41 ± 1.0	45 max.
GBL 60	55 min.	59 ± 1.0	63 max.
GBL 100	95 min.	100 ± 1.0	105 max.

16
Z 8901-1995

Table 20. Particle size distribution of barium-titanate glass
Unit: μm

Class	Particle size in case of 90 % oversize (mass basis)	Particle size in case of 50 % oversize (mass basis)	Particle size in case of 10 % oversize (mass basis)
GBM 20	18 min.	22 ± 1.0	26 max.
GBM 30	26 min.	30 ± 1.0	34 max.
GBM 40	37 min.	41 ± 1.0	45 max.

- (4) Refractive index Refractive index shall be from 1.51 to 1.53 for soda lime-silicate glass and from 1.92 to 1.94 for barium-titanate glass when measured according to the testing method of refractive index specified in JIS R 3301.
- (5) Appearance and shape Appearance and shape shall be judged by the total number of particles having such as ellipse, acute angle, opaque, alien matter, fusion of particles and the like, whose total shall be 5 % or less on number basis when measured according to the method on appearance and shape specified in JIS R 3301.

5.1.2 White fused alumina The quality of white fused alumina shall conform to the following items.

Remarks: White fused alumina is composed of corundum crystal which has been prepared by fusing alumina in an electric furnace, cooling to solidify, grinding it, and wet classification to arrange particle size.

- (1) Chemical composition Chemical composition shall be 99 % or more by the content of Al_2O_3 when measured according to JIS R 6123.
- (2) Particle density Particle density shall be from 3.9 g/cm^3 to 4.0 g/cm^3 when measured according to JIS R 6125.
- (3) Particle size distribution Particle size distribution shall meet the values stated in Table 21 when measured according to the testing method of electric resistance specified in JIS R 6002.

Table 21. Particle size distribution of white fused alumina
Unit: μm

Class	Particle size in case of 94 % oversize (mass basis)	Particle size in case of 50 % oversize (mass basis)	Particle size in case of 3 % oversize (mass basis)
No. 1	0.8 min.	2 ± 0.45	5 max.
No. 2	2.0 min.	4 ± 0.5	11 max.
No. 3	4.5 min.	8 ± 0.6	20 max.
No. 4	9.0 min.	14 ± 1	31 max.
No. 5	20 min.	30 ± 2	58 max.
No. 6	40 min.	57 ± 3	103 max.

- (4) Specific surface area Specific surface area shall meet the value in Table 22 when measured according to JIS Z 8830.

Table 22. Specific surface area of white fused alumina

Class	Specific surface area m^2/g
No. 1	4.3 to 4.5
No. 2	1.5 to 1.7
No. 3	0.7 to 0.9
No. 4	0.3 to 0.5
No. 5	0.1 to 0.3
No. 6	0.05 to 0.15

5.2 Cautions for handling When test powders 2 is used for various type of tests, unless otherwise specified, dry it sufficiently, disperse it as completely as possible, and submit it under no deteriorated condition.

5.3 Marking The containers of test powders 2 shall be legibly and indelibly marked with the following items.

- (1) Class and material employed
- (2) Net mass
- (3) Chemical composition
- (4) Particle density
- (5) Particle size distribution
- (6) Refractive index
- (7) Specific surface area
- (8) Name of manufacturer or its abbreviation

Example 1. Test powders 2 Case of GBM20 glass beads

- (1) Class and material employed GBM20 glass beads
- (2) Net mass 100 g

18
Z 8901-1995

(3) Chemical composition

Composition	Mass percentage %
TiO ₂	33 to 38
BaO	41 to 48
SiO ₂	10 to 14
CaO	4 to 8

(4) Particle density 4.0 g/cm³ to 4.2 g/cm³

(5) Particle size distribution

Oversize (on mass basis)		
Particle size of 90 %	Particle size of 50 %	Particle size of 10 %
18 min.	22 ± 1.0	26 max.

(6) Refractive index 1.92 to 1.94

(7) Specific surface area —

(8) Name of manufacturer or its abbreviation

Example 2. Test powders 2 Case of No. 1 white fused alumina

(1) Class and material employed No. 1 white fused alumina

(2) Net mass 500 g

(3) Chemical composition Al₂O₃ 99 % or more

(4) Particle density 3.9 g/cm³ to 4.0 g/cm³

(5) Particle size distribution

Oversize (on mass basis)		
Particle size of 94 %	Particle size of 50 %	Particle size of 3 %
0.8 min.	2 ± 0.45	5 max.

(6) Refractive index —

(7) Specific surface area 4.3 m²/g to 4.5 m²/g

(8) Name of manufacturer or its abbreviation

6. Test particles 1

6.1 Quality The quality of test particles 1 (polystyrene group particles) shall conform to the following items.

- (1) Specific gravity Specific gravity shall be from 1.04 to 1.07 when measured according to 6.3.1.
- (2) Refractive index Refractive index shall be from 1.54 to 1.65⁽⁷⁾ when measured according to 6.3.2.

Note (7) Refractive index shall be measured with sodium D line at 20°C.

- (3) Shape Shape shall be spherical.

6.2 Mean particle size Mean particle size shall meet the values in Table 23 when measured according to the method in Annex.

Other measuring method than the method in Annex may be used if it can give the equivalent result as that by Annex.

Table 23. Mean particle size of test particles 1

Class	Nominal diameter μm	Range of mean particle size μm	The maximum CV value %
No. 1	0.05	0.045 to 0.055	10.0
No. 2	0.08	0.076 to 0.084	3.0
No. 3	0.1	0.095 to 0.105	3.0
No. 4	0.2	0.190 to 0.210	3.0
No. 5	0.3	0.285 to 0.315	3.0
No. 6	0.5	0.475 to 0.525	3.0
No. 7	1	0.950 to 1.050	3.0
No. 8	2	1.900 to 2.100	3.0
No. 9	5	4.750 to 5.250	3.0
No.10	10	9.50 to 10.50	3.0
No.11	15	14.25 to 15.75	3.0
No.12	25	23.75 to 26.25	3.0

20
Z 8901-1995

6.3 Measuring method

6.3.1 Specific gravity The measuring method of specific gravity shall be as follows.

- (1) Apparatus and implements Apparatus and implements shall be as follows.
 - (a) Standard hydrometer I type hydrometer specified in JIS K 2249.
 - (b) Sample bottle Glass bottle with about 10 ml capacity.
 - (c) Stirring rod Made of the material free from the material giving damage to the properties of sample and capable of stirring strong enough to make the dispersion of the sample in a container uniform.
- (2) Reagents Reagents shall be as follows.
 - (a) NaCl Prepare water solution with specific gravity of 1.0 to 1.2.
 - (b) Water Ultra pure water⁽⁸⁾

Note ⁽⁸⁾ Refined water generated by passing through any one of thin membran filter, ultrafiltration membrane, or reverse osmosis membrane, and whose resistivity is at least 16 MΩ·cm at 25°C.

- (3) Preparation of sample Generally, prepare sedimentation from the liquid containing particles owing to standing⁽⁹⁾.

Note ⁽⁹⁾ When particles make no sedimentation, use centrifugal separation.

- (4) Preparation of reagent The preparation of reagent shall be as follows.

- (a) Prepare NaCl water solutions with various specific gravities.
- (b) Adjust the specific gravity of each solution using standard hydrometer.

Remarks: Prepare about 10 solutions which cover target range.

- (5) Procedures for measuring The procedures for measuring shall be as follows.

- (a) Place 10 ml of standard solution of specific gravity in a sample bottle⁽¹⁰⁾.

Note ⁽¹⁰⁾ Carry out measurement at from 20°C to 22°C temperature.

- (b) Put a little of particle sedimentation into (a).

- (c) Mix them satisfactorily with a stirring rod, allow it to stand for about 16 h, and observe it on the next day.
- (d) Observe the solutions with 0.002 interval of specific gravity each other, read specific gravity value when particles stay in the solution, and make the value the specific gravity of the particles.

When particles are floating or submerged instead staying midway, the middle value between them shall be made the specific gravity of the particles.

Example: When 1.054 solution makes particle go down and 1.056 makes float, adopt the value 1.055.

6.3.2 Refractive index

6.3.2.1 The refractive index of particles, which is soluble in solvent, shall be measured as follows.

- (1) Apparatus and implements Apparatus and implements shall be as follows.
 - (a) Refractometer Abbe's refractometer.
 - (b) Sample bottle Glass bottle with about 10 ml capacity.
 - (c) Thermostatic water bath Capable of keeping the sample in a sample bottle at $20.0 \pm 1^\circ\text{C}$.
- (2) Reagents Reagents shall be as follows.
 - (a) Toluene Guaranteed reagent
 - (b) Water Ultra pure water⁽⁸⁾
- (3) Procedures for measuring The procedures for measuring shall be as follows.

(3.1) Preparation of sample solution

- (a) Wash sufficiently polystyrene group particles with ultra pure water⁽⁸⁾, separate centrifugally them once, dry them by freezing, and pulverize them.
- (b) Dissolve a decided amount of powder with each particle size into solvent (toluene), and prepare a series of the solutions of polystyrene group particles whose concentrations are different⁽¹¹⁾.

Note ⁽¹¹⁾ Prepare about 10 solutions which cover target range.

(3.2) Preparation of refractometer

- (a) Connect Abbe's refractometer to a thermostatic water bath at 20°C .

- (b) Measure the refractive index of toluene, and confirm the refractive index of toluene is just 1.49.

- (3.3) Measurement of refractive index Drip the solutions of each polystyrene group particles in a refractometer one by one, and read swiftly the value. Plot the obtained data on a section paper, read the value obtained by extrapolation up to 100 % concentration using the method of least squares, and make the value the refractive index of the polystyrene group particles.

6.3.2.2 The refractive index of particles, which is insoluble in solvent, shall be measured as follows.

- (1) Apparatus and implements Apparatus and implements shall be as follows.
- (a) Refractometer Abbe's refractometer
 - (b) Microscope Transmission optical microscope
 - (c) Implements Slide glass and cover glass
- (2) Reagents Reagents shall be as follows.
- (a) Potassium mercurous iodide (HgKI_2)⁽¹²⁾ Guaranteed reagent
 Note ⁽¹²⁾ Be careful when handling because of its poisonous property.
 - (b) Water Ultra pure water⁽⁸⁾
- (3) Preparation of test solutions for refractive index Dissolve HgKI_2 in ultra pure water, and prepare the test solutions for refractive index, which have 0.0005 or 0.001 interval each other, while measuring the refractive index of each solution.
- (4) Preparation of polystyrene group particles Wash enough polystyrene group particles with ultra pure water, and dry them.
- (5) Measurement of refractive index The measuring method of refractive index shall be as follows.
- (a) Connect Abbe's refractometer to the thermostatic water bath kept at 20°C.
 - (b) Disperse polystyrene group particles on a slide glass.
 - (c) Drop about 5 μl test solution for refractive index on (b), and place a cover glass on it.
 - (d) Observe it through a microscope, and find the refractive index by which polystyrene group particles are the most hardly observed, to make it the refractive index of the polystyrene group particles.

6.4 Cautions for handling When test particles 1 is to be used for calibration test of a light-scattering automatic particle counter, the following shall be followed.

- (1) Case of the concentrated suspension by polystyrene group particles
Prior to use, stir each container according to the following procedures, and make the particles in the container dispersed.

Remarks: Test particles 1 has usually put in ultra pure water⁽⁸⁾ kept in a plastic container with about 10 ml capacity, and it makes a suspension of polystyrene group particles. Therefore standing it for long period may let the polystyrene group particles sink bottom of the container. Especially be careful the particles with 0.5 μm or more particle size.

- (a) Shake the container several times with hands.
(b) Stir the container two or three times using a touch mixer⁽¹³⁾ for each 10 s.

Note ⁽¹³⁾ It is a mixer by whose vibration inside of a plastic container can be stirred by contacting the container on the touch surface of the mixer.

- (c) Immerse the container in an ultrasonic cleaning device⁽¹⁴⁾ for about 10 min.

Note ⁽¹⁴⁾ Suitable one is of 50 W to 1 kW of capacity and 20 kHz to 50 kHz of frequency.

- (d) To limit the contamination from outside to the least, carry out opening and closing of the container in clean surroundings.
(e) After taking out necessary amount of suspension of polystyrene group particles from the container, immediately close it with a cover not to let it dry.
(f) When the suspension of polystyrene group particles is diluted or mixed with dispersion medium, employ it in the pH range from 6 to 9. Provided that never use the dispersion medium dissolvable polystyrene group particles.

Remarks: Some type of organic solvents may cause dissolving or swelling, leading to the deformation or degeneration of particles.

- (2) Case of powdered polystyrene group particles In case of powdered polystyrene group particles, carry out as follows.

- (a) Take out powdered polystyrene group particles from a container, and immediately close it with a cover.
(b) Avoid direct sunshine.

6.5 Cautions when storing The cautions when storing shall be as follows.

- (1) In case of suspension of polystyrene group particles, never freeze it. After opening it, store it in a refrigerator.
- (2) Store with shielding against direct sunshine.

6.6 Marking The containers of test particles 1 shall be legibly and indelibly marked with the following items.

- (1) Class and nominal diameter

Example: Test particles 1 (polystyrene group particles)
No. 1 0.05 μm

- (2) Net mass

- (3) Specific gravity

Example: 1.04 to 1.07

- (4) Refractive index

Example: 1.54 to 1.65

- (5) Mean particle size

Example: Test particles 1 No. 1

Nominal diameter μm	Range of mean particle size μm	Maximum CV value %
0.05	0.045 to 0.055	10.0

- (6) Name of manufacturer or its abbreviation

7. Test particles 2

7.1 Quality The quality of test particles 2 shall conform to the following items.

- (1) Class 1 of test particles 2 The quality of Class 1 of test particles 2 shall be No. 1 of dioctyl phthalate specified in JIS K 6753.
- (2) Class 2 of test particles 2 The quality of Class 2 of test particles 2 shall be the guaranteed grade of stearic acid specified in JIS K 8585.

7.2 Particle size distribution The particle size distribution of test particles 2 shall be as follows.

- (1) Class 1 of test particles 2 The Class 1 of test particles 2 shall be the aerosol prepared with using a pressurized-spraying type (Ruskin nozzle type), a heating-condensation type, or generator similar to them, and its particle size distribution shall meet the requirements specified in Table 24 when measured according to 7.3.1.

- (2) Class 2 of test particles 2 The Class 2 of test particles 2 shall be the aerosol prepared with using a heating-condensation type or generator similar to it, and its particle size distribution shall meet the requirements specified in Table 24 when measured according to 7.3.1 or 7.3.2.

Table 24. Particle size of Class 1 and Class 2 of test particles 2

Item	Class 1	Class 2
Median diameter (on number basis) μm	0.21 to 0.32	0.25 to 0.35
Geometric standard deviation σ_g	1.43 to 1.83	1.4 max.

7.3 Measuring method

7.3.1 Light scattering method The measurement by light scattering method shall be as follows.

- (1) Instrument for use Out of the light scattering automatic particle counters specified in JIS B 9921, employ that capable of measuring the particles of 0.1 μm or 0.12 μm or more.
- (2) Procedures for measuring The procedures for measuring shall be as follows.
 - (a) Confirm that the generator for test particles is stationarily generating test particles.
 - (b) When the numerical concentration of test particles, generated from the generator, exceeds the detection limit of a measuring apparatus, lower its concentration by means of a diluting device.
 - (c) Inhale them into an introducing inlet of a light scattering automatic particle counter.
 - (d) Confirm that the output is in specified condition by the light source for calibration, decide the mode for measuring particle size, run the particle counter, and measure numerical concentration of every particle size.

7.3.2 Scanning electron microscope

- (1) Apparatus and implements Apparatus and implements shall be as follows.
 - (a) Scanning electron microscope Having resolution strong enough to be able to measure the minimum particle size of 0.2 μm .
 - (b) Ion spattering device Capable of making vapor deposition of conducting material as gold or the like on cover glass or sheet mesh on which particles have been placed.
 - (c) Replica of diffraction grating This is the transfer or reproduction of the unevenness on the surface owing to making carbon coating on diffraction grating.

- (d) Device for catching particles The device for catching particles shall be an impactor or thermal precipitator.
- (2) Procedures for measuring The procedures for measuring shall be as follows.
- (a) Confirm that the generator for test particles is stationarily generating test particles.
 - (b) With using an impactor or thermal precipitator, place stearic-acid particles on a cover glass or sheet mesh.
 - (c) With using an ion spattering device, carry out vapor deposition of gold or other conducting material on the stearic acid particles which have been placed on the cover glass or mesh sheet, and fix them on the sample base.
 - (d) Put the sample base of (c) in the sample room of a scanning electron microscope, and after specified procedures, carry out taking photographs.
 - (e) Choose about 200 particles, measure particle sizes each of which has the same direction, plot the data on a logarithmic probability paper, and obtain the median diameter on number basis and geometric standard deviation.

7.4 Cautions for handling When test particles 2 is offered to various tests or investigations, unless otherwise specified, it shall be dried sufficiently and be used under as dispersed condition as possible.

The melted liquid of test particles 2 Class 2, though at first showing faint yellow color, becomes brown color due to oxidation and mingling of alien matter in keeping with time for use. The limit for continuous use is one month or is that its color turns brown, and in such case it shall be displaced with new one.

Attached Table 1. Cited standards

JIS A 6201	Fly ash
JIS B 7506	Gauge blocks
JIS B 9921	Light scattering automatic particle counter
JIS K 0121	General rules for atomic absorption spectrochemical analysis
JIS K 2249	Crude petroleum and petroleum products - Determination of density and petroleum measurement tables based on a reference temperature (15°C)
JIS K 6221	Testing methods of carbon black for rubber industry
JIS K 6753	Di-2-ethylhexyl phthalate
JIS K 8034	Acetone
JIS K 8103	Diethyl ether
JIS K 8271	Xylene
JIS K 8585	Stearic acid
JIS M 8100	Particulate materials - General rules for methods of sampling
JIS M 8850	Methods for chemical analysis of limestone
JIS R 2212	Chemical analysis of refractory bricks and refractory mortars
JIS R 3101	Method for chemical analysis of Soda-Lime-Magnesia-Silica Glasses
JIS R 3105	Methods for chemical analysis of borosilicate glasses
JIS R 3301	Glass beads for traffic paint
JIS R 5210	Portland cement
JIS R 6002	Testing methods for abrasive grain size
JIS R 6123	Method for chemical analysis of aluminous abrasives
JIS R 6125	Testing method for specific gravity of artificial abrasives
JIS Z 8103	Glossary of terms used in instrumentation

Attached Table 1. (continued)

JIS Z 8122	Contamination control - Vocabulary
JIS Z 8815	Test sieving - General requirements
JIS Z 8820	General rules for the determination of particle size distribution by sedimentation in liquid
JIS Z 8821	Determination of particle size distribution by the sedimentation in liquid using the pipette apparatus
JIS Z 8822	Determination of particle size distribution by the weight of sedimentation in liquid
JIS Z 8830	Determination of the specific surface area of powders by gas adsorption methods

Annex Measuring method of mean particle size of test particles 1

1. Scope This Annex specifies the measuring method of mean particle size of test particles 1 by using either transmission electron microscope or transmission optical microscope.

Remarks 1. Transmission electron microscope can be used for measuring mean particle size with nominal diameter range from 0.05 μm to 5 μm of test particles 1, and transmission optical microscope for that with nominal diameter range from 10 μm to 25 μm .

2. The standard cited in this Annex is as follows.

JIS B 7506 Gauge blocks

2. Apparatus and implements

2.1 Method by transmission electron microscope The apparatus and implements used for transmission electron microscope method shall be as follows.

- (1) Transmission electron microscope Transmission electron microscope (hereafter referred to as "electron microscope") shall have the resolution strong enough to measure the minimum particle size 0.05 μm .
- (2) Ion spattering device Capable of hydrophilic treatment of the sheet mesh which has been covered with collodion film and reinforced with carbon.
- (3) Scale loupe Measurable 0.1 mm scale by 7 times magnifying.
- (4) Replica of diffraction grating This is the transfer or reproduction of the unevenness on the surface owing to making carbon coating on diffraction grating.

2.2 Method by transmission optical microscope The apparatus and implements used for transmission optical microscope method shall be as follows.

- (1) Transmission optical microscope Transmission optical microscope (hereafter referred to as "optical microscope") shall have the resolution strong enough to measure test particle with 10 μm or more nominal diameter, and can take photographs of particles.
- (2) Gauge block Total length 10 mm block with the first class quality specified in JIS B 7506.
- (3) Objector-type length measuring apparatus Measurable 0.01 mm scale by 10 times magnifying.
- (4) Objective micrometer With 100 lines/mm (scale of 0.01 mm).
- (5) Scale loupe Measurable 0.1 mm scale by 7 times magnifying.

3. Method by transmission electron microscope

3.1 Preparation of sample The preparation of sample shall be as follows.

- (1) Place collodion film on sheet mesh, and reinforce it with carbon.
- (2) Carry out hydrophilic treatment on the mesh prepared at (1) using an ion spattering device. In case of the mesh needing no hydrophilic treatment (for instance, made of plastic as polyvinylformal, or plastic covered supporting film), however, the hydrophilic treatment is not needed.
- (3) Take 20 μl of polystyrene group particles in a clean container, dilute it with 5 ml of ultra pure water, pick up the mesh at (2) with a pincette, let it touch softly on water surface, and put dilution liquid on the mesh.
- (4) Dry naturally the mesh at (3) for about 16 h, and use it as the sample to be tested. When employed the desiccator with silica gel, drying for about 3 h is enough.

3.2 Adjustment of electron microscope Electron microscope shall be adjusted as follows.

- (1) Confirm an ordinary alignment of axis has been finished.
- (2) Adjust the height of sample using an magnification for observation.
- (3) Adjust focusing and astigmatism by using sample particles.

3.3 Measuring reduction ratio of particle Because polystyrene group particles get reduction by radiation of electron beam, correction is needed, therefore, reduction ratio shall be measured. Its measurement shall be as follows.

- (1) Setting up of observing condition Observing conditions shall be set up as 80 kV to 200 kV of accelerating voltage and 10 pA/cm² of current density on a fluorescent screen⁽¹⁾.

Note ⁽¹⁾ These values differ according to the type of electron microscope.

- (2) Photographing Photographing shall be carried out as follows.

- (a) The particles for photograph shall be the same as those for measurement.
- (b) Place about 50 particles in one visual field, and take photograph after 30 s from the radiation of electron beam.
- (c) While electron beam is being radiated, without changing its visual field, take photograph after 60 s, then successively every 30 s.

Though adjusting of focusing may take 30 s or longer, finish it in short time as possible.

- (d) Continue these procedures for 5 min to 6 min, and take photographs at every radiation period.

(3) Calculation of reduction ratio Reduction ratio shall be calculated as follows.

- (a) On the film photographed at (2), read diameter of particles to the nearest 0.05 mm using a loupe with 0.1 mm scale, and measure mean particle size at every radiation period.
- (b) Plot the diagram showing both the measured value of particle size and "t" of time being radiated by electron beam, find the particle size, probably given when the time radiated by electron beam is zero, according to extrapolation regressed to polynomial expression by method of least squares, and calculate the reduction ratio at 30 s after radiation starts according to the following formula.

$$S_{30} = \frac{t_{30}}{t_0}$$

where, S_{30} : reduction ratio at 30 s after radiation starts

t_{30} : particle size at 30 s after radiation starts (μm)

t_0 : particle size after t_0 of radiation period (μm)

3.4 Observation and photographing of particles Make it a condition for photographing to wait 30 s after electron beam radiated, take photographs of 200 particles which were set as new target, and measure their particle sizes with a loupe.

Be careful not to observe again the visual field which was once observed.

3.5 Correction of magnifying power of electron microscope The magnifying power of electron microscope shall be corrected as follows.

- (1) Photograph the replica of diffraction grating with the magnifying power used when particles were observed.
- (2) After films were developed, read interval between diffraction gratings by 50 spots using a scale loupe.
- (3) Calculate the mean of the values obtained at (2), and calculate the corrected value of magnifying power making use of the ratio of the value to maker's published value.

$$C = \frac{l}{l'}$$

where, C : corrected value of magnifying power

l : mean of measured distances between diffraction gratings (μm)

l' : maker's published distance between diffraction gratings (μm)

3.6 Calculation of particle size after correction The particle size after correction shall be calculated as follows.

$$D_c = \frac{D}{C_a \times C \times S_{30}} \times 1000$$

where, D_c : particle size after correction (μm)

D : particle size read by scale loupe (mm)

C_a : marked magnifying power of electron microscope

C : corrected value of magnifying power of electron microscope

S_{30} : reduction ratio at 30 s after electron beam was radiated

Making use of particle size after correction, calculate mean particle size and CV value⁽²⁾.

Note ⁽²⁾ Instead, an automatic plotting machine, by which particle size can be automatically calculated after correction, may be serviceable.

4. Transmission optical microscope

4.1 Adjustment of optical microscope Optical microscope shall be adjusted as follows.

- (1) Adjust an optical axis, diaphragm of visual field, focusing, condition of stage and so on of an optical microscope, and get ready to obtain clear image of particles.
- (2) Attach a camera, and get ready for photographing.

4.2 Verification of objector-type length measuring apparatus Magnify 10 times a gauge block using an objector-type length measuring apparatus, measure whole length (10 mm) of the gauge block, carry out ten times this measuring, and calculate correction factor A according to the following formula.

$$A = \frac{S}{S'}$$

where, A : correction factor

S : mean of measured values of gauge block (mm)

S' : whole length of gauge block (mm)

4.3 Verification of objective micrometer Magnify 100 times an objective micrometer (hereafter referred to as "micrometer") using an objector-type length measuring apparatus, measure whole length (1 mm), repeat this measuring ten times, and calculate correction factor B according to the following formula.

$$B = \frac{r}{r'}$$

where, B : correction factor

r : mean of measured values of micrometer (mm)

r' : whole length of micrometer (1 mm)

4.4 Correction of magnifying power of optical microscope The magnifying power of an optical microscope shall be corrected as follows.

- (1) Put the micrometer, which has been verified, on a stage of a optical microscope, and take its photographs with the same magnifying power as that for taking photographs of test particles.
- (2) Count the number of scale lines of the micrometer which was centrally observed in about 10 mm visual field of scale loupe, and measure the distance covering that number of scale lines.

Example: When 3 scale widths of a micrometer are contained in above visual field, total distance covering these 3 widths shall be the distance of scale lines.

- (3) Repeat five times the procedures in (1) and (2).
- (4) Calculate the distance of one scale width according to the following formula.

$$E = \frac{e}{e'}$$

where, E : distance of one scale width (mm)

e : distance of scale lines (mm)

e' : number of scale lines

- (5) Calculate correction factor C according to the following formula.

$$C = \frac{A \times \bar{E}}{0.01 \times B \times O}$$

where, C : correction factor

$\bar{E}$: mean one scale width between two scale lines (mm)

O : magnifying factor for photographing

A : correction factor

B : correction factor

0.01 : length of one scale width (0.01 mm)

34.

Z 8901-1995

4.5 Preparation of sample Sample shall be prepared as follows.

- (1) In case of suspension of particles, wash several times it with ultra pure water.
- (2) Drip suspension of particles on a slide glass, and dry naturally with wind.

If number of particles is too many, dilute suitably it with ultra pure water, then drip it, and dry naturally with wind.

4.6 Photographing Carry out photographing as follows.

- (1) Put the slide glass, on which test particles have been placed, on the stage of a microscope, and observe them with magnifying power to make a particle size about 10 mm.
- (2) Search the place where particles are sparsely scattered, and take photographs of about total 300 particles.

4.7 Measurement of particle size Particle size shall be measured as follows.

- (1) Select out at random isolated particles out of particle group on the photograph, put them on the middle of scale loupe, read them to the nearest 0.05 mm, measure the values of 200 particles, and record them.
- (2) Correct the measured value at (1) according to the following formula, and calculate the particle size after correction.

$$D_c = \frac{D}{O} \times \frac{1}{C}$$

where, D_c : particle size after correction (μm)

O : magnifying power for photographing

D : measured value (μm)

C : correction factor

- (3) Making use of (1) and (2), calculate mean particle size and CV value.

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