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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 81.040.30

Q 35

GB/T 32661-2016

Spherical Silica Powder

球形二氧化硅微粉

Issued on: April 25, 2016

Implemented on: March 1, 2017

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration of the People's Republic of China.

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Spherical Silica Powder

1 Scope

This Standard specifies the terms and definitions, classification and labeling, requirements, test methods, inspection rules, marking, packaging, transportation and storage of spherical silica powder.

This Standard is applicable to spherical silica powder used in electronic packaging materials fillers, copper-clad laminate fillers, etc. in the electronics and electrical appliance industries. Spherical silica powder used for other purposes may refer to this Standard.

2 Normative References

The following documents are essential to the application of this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 191 Packaging – Pictorial Marking for Handling of Goods

GB/T 6284 Chemical products for industrial use - General method for determination of water content - The loss of mass on drying method

GB/T 6678 General principles for sampling chemical products

GB/T 6908-2008 Analysis of water used in boil and cooling system-Determination of electrical conductivity

GB/T 9724 Chemical reagent—General rule for the determination of pH

GB/T 11446.7 Test Method for Trace Anion in Electronic Grade Water by Ion Chromatography

GB/T 14640-2008 Water used in industrial circulating cooling system and boiler - Determination of potassium and sodium

GB/T 16418-2008 Particle system - Vocabulary

GB/T 19077.1 Particle size analysis - Laser diffraction methods - Part 1: General principles

GB/T 23774 Inorganic chemicals for industrial use - General method for the determination of whiteness

JC/T 753-2001 Methods for chemical analysis of class making sands

SJ 3228.2 General rules for methods of analysis for high purity arenaceous quartz

SJ 3228.3 Determination of loss on ignition in high purity arenaceous quartz

SJ/T 10675-2002 Silicon dioxide micro-powder for electronic and electrical equipment industry

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Median diameter

The particle size corresponding to when the cumulative particle size distribution percentage reaches 50%. Written as D_{50} , in μm .

NOTE: Adapted from GB/T 16418-2008, definition 2.2.1.9.

3.2 Degree of sphericity

The degree of particles close to a sphere.

NOTE 1: The sphericity of the sphere is 1.

NOTE 2: Adapted from GB/T 16418-2008, definition 2.2.2.16.

3.3 Rate of spheroidization

The ratio of the number of spherical particles to the total number of counted particles.

4 Classification and Labeling

4.1 Classification

4.1.1 According to use, it is divided into: electronic grade spherical silica powder (QYG-H), ordinary spherical silica powder (QYG-R).

4.1.2 According to the median particle diameter D_{50} range, it is divided into five specifications: 030, 020, 010, 005 and 002.

4.2 Labeling

Labeled by application and median particle size specifications.

Sodium hexametaphosphate, analytically pure, mass fraction 0.5%.

6.5.3 Device

6.5.3.1 Microscope: with CCD module, pixels no less than 1 million, magnification no less than 100 times.

6.5.3.2 Computer: equipped with image processing software.

6.5.4 Determination

6.5.4.1 Using sodium hexametaphosphate with a mass fraction of 0.5% as the dispersion medium, take an appropriate amount of sample and disperse it evenly on the glass slide.

6.5.4.2 Use a microscope to observe the sample and adjust the focus to the clearest field of view.

6.5.4.3 Select an area with well-dispersed particles in the field of view and record the image of this area on the computer.

6.5.4.4 Use image processing software to analyze the recorded images and determine the projected area and peripheral length of each particle.

6.5.5 Calculation

The degree of sphericity of a single particle is calculated according to Formula (1):

$$\Phi_a = 4\pi A / B^2 \quad \dots\dots\dots (1)$$

Where:

Φ_a - The measured degree of sphericity of a single particle;

A - The area of particle projection, in μm^2 ;

B - Peripheral length of particle projection, in μm .

The measurement result of degree of sphericity is the arithmetic mean of the degree of sphericity of 50 randomly selected complete particles in the image. Incomplete particles at the edge of the image shall not be measured.

6.6 Rate of spheroidization

6.6.1 Principle

The ratio of the number of non-spherical particles to the total number of particles in the statistical field of view is calculated. The basis for determining non-spherical particles is particles with a degree of sphericity less than 0.85 or particles with acute-angled surfaces on

It shall be carried out according to the provisions of SJ 3228.3.

6.10 Extraction liquid

6.10.1 Preparation of extraction liquid

It shall be carried out according to the provisions of 6.11 in SJ/T 10675-2002.

6.10.2 Conductivity of extraction liquid

It shall be carried out according to the provisions of GB 6908.

6.10.3 pH value of extraction liquid

It shall be carried out according to the provisions of GB 9724.

6.10.4 Na⁺ content in the extraction liquid

It shall be carried out according to the provisions of Clause 5 of GB/T 14640-2008.

6.10.5 K⁺ content in the extraction liquid

It shall be carried out according to the provisions of Clause 4 of GB/T 14640-2008.

6.10.6 Cl⁻ content in the extraction liquid

It shall be carried out in accordance with the provisions of GB/T 11446.7.

6.11 Screening allowance

6.11.1 Principle

Weigh a certain amount of sample; use a standard sieve to screen out particles with a particle size larger than the required size; weigh the mass of the particles; and calculate their ratio to the mass of the weighed sample.

6.11.2 Materials and instruments

Materials and instruments are as follows:

- a) Standard screen;
- b) Analytical balance;
- c) Ultrasonic dispersion instrument;
- d) 1000mL beaker;
- e) Washing bottles.

h) Microscope, the optical magnification is no less than 60 times.

6.12.3 Determination

6.12.3.1 Add 100g of sample into a beaker containing 700mL of water; stir evenly with a glass rod; and disperse ultrasonically for 3 min.

6.12.3.2 Use distilled water to clean the casing of the magnetic rod; insert the casing vertically into the liquid in the beaker; then insert the magnetic rod into the casing; and stir clockwise along the periphery of the container for 1 min.

6.12.3.3 Take out the magnetic rod and outer sleeve casing together; move them to another beaker; pull out the magnetic rod; clean the surface of the outer sleeve casing with a washing bottle; and collect the washing liquid.

6.12.3.4 Filter all the washing liquid through a double-layer screen, where the upper screen has a pore size of 100 μ m and the lower screen has a pore size of 20 μ m.

6.12.3.5 Use a washing bottle to wash the magnetic foreign matter on each screen into a small beaker; let it stand for 2 min; pour off the supernatant; and transfer the magnetic foreign matter in the beaker to dust-free filter paper to absorb the water.

6.12.3.6 Use a 60 $\times$ microscope to observe the number of magnetic foreign objects on the dust-free filter paper.

6.12.3.7 Repeat the above operation 3 times.

6.12.4 Calculation

Take the average number of magnetic foreign matters on each screen for 3 times, in pieces.

6.13 Crystalline SiO₂ content

It shall be carried out according to the provisions of 6.10 in SJ/T 10675-2002.

6.14 SiO₂ content

It shall be carried out according to the provisions of Clause 5 of JC/T 753-2001.

6.15 Fe₂O₃ content

It shall be carried out according to the provisions of Clause 7 of JC/T 753-2001.

6.16 Al₂O₃ content

It shall be carried out according to the provisions of Clause 6 of JC/T 753-2001.

6.17 CaO content

It shall be carried out according to the provisions of Clause 9 of JC/T 753-2001.

7 Inspection Rules

7.1 Inspection classification

Inspection is divided into exit-factory inspection and type inspection.

7.2 Batching

For spherical silica powder produced with the same raw materials, the same equipment, and the same process parameters, each ton constitutes an inspection batch, and any amount less than one ton is counted as a batch.

7.3 Exit-factory inspection

7.3.1 Inspection items

The exit-factory inspection items include items other than moisture content, loss on ignition, and crystalline SiO₂ content specified in Clause 5.

7.3.2 Judgment rules

If all the inspection items specified in the provisions are qualified, the batch of products is qualified. If any one of the inspection items is unqualified, double the sampling amount to inspect the item, the total sampling amount is no less than 2kg, and the mass after the quartering method is no less than 250g. If the re-inspection result is qualified, the batch of products is qualified; if the re-inspection result is unqualified, it is deemed unqualified. If there are two or more unqualified items in the inspection items, the batch of products shall be unqualified.

7.4 Type inspection

7.4.1 General

Type inspection shall be carried out when any of the following situations occurs:

- a) Trial prototype identification of new products or old products transferred to other factory for production;
- b) After formal production, if there are major changes in raw materials and processes, which may affect product performance;
- c) When the exit-factory inspection results are significantly different from the last type inspection;

d) During normal production, conduct once a year.

7.4.2 Inspection items

Type inspection items are all items specified in Clause 5.

7.4.3 Judgment rules

If all the inspection items specified in the provisions are qualified, the batch of products is qualified. If any one of the inspection items is unqualified, double the sampling amount to inspect the item, the total sampling amount is no less than 2kg, and the mass after the quartering method is no less than 250g. If the re-inspection result is qualified, the batch of products is qualified; if the re-inspection result is unqualified, it is deemed unqualified. If there are two or more unqualified items in the inspection items, the batch of products shall be unqualified.

8 Marking, Packaging, Transportation and Storage

8.1 Marking

The packaging bag is printed with the following information, such as product name, trademark, manufacturer, detailed address, product specifications and models, production batch number, and net content.

Each bag of product shall be accompanied by a certificate of conformity. The certificate shall indicate the following information, such as product name, manufacturer, trademark, inspection results, batch number or date of delivery, inspector number and inspection department seal.

The outer packaging shall have a graphic mark that complies with GB/T 191.

8.2 Packaging

Spherical silica powder is generally packed in bags or barrels. Packaging must be strong, clean, airtight, and dry.

8.3 Transportation

During the transportation process, the transportation tools must be kept clean and cannot be mixed with other goods. Pay attention to preventing rain, moisture, broken packages, and pollution.

8.4 Storage

It shall be stored in a ventilated, dry and clean room; and pay attention to prevent rain, moisture, package breakage and pollution.

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