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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 38061-2019

Cerium dioxide nanomaterial

纳米二氧化铈

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Standardization Administration of the PRC

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed by the Chinese Academy of Sciences.

This standard is under the administration of the Subcommittee on Nanomaterials of the National Technical Committee on Nanotechnology of Standardization Administration of China (SAC/TC 279/SC 1).

The drafting organisations of this standard are the National Center for Nanoscience and Technology, Inner Mongolia Xinyu Rare Earth Functional Materials Co., Ltd., Shenzhen Xianggen Biotechnology Co., Ltd., Xuancheng Jingrui New Material Co., Ltd., the Metallurgical Industry Information Standards Research Institute and the Institute of Process Engineering of the Chinese Academy of Sciences.

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1 Scope

China's national product standard for nano cerium dioxide powder, the near-spherical grade with the formula CeO2 and a relative molecular mass of 172.11. Nano ceria is the most heavily produced of the rare earth nanomaterials, and it earns that position by doing two unrelated jobs extremely well. It is the abrasive used to polish glass and silicon: the chemical-mechanical polishing of wafers, hard disk substrates and display glass is done

with ceria slurries because ceria attacks silica chemically as well as mechanically, and nothing else leaves that surface. And it takes up and gives back oxygen reversibly as it switches between two oxidation states, which is why it is the oxygen storage component of automotive three-way catalysts. Both jobs depend on properties that a chemical assay alone cannot capture, and that is what makes this standard necessary: two powders can both be 99.9 percent CeO2 and behave completely differently in a slurry, because what matters is the size of the crystallites, the surface area they present and how badly the particles have clumped together. The standard therefore specifies not only the purity limits but the mean particle size by electron microscopy, the mean crystallite size from the X-ray diffraction line width, the BET specific surface area and an agglomeration index which is simply the ratio of the two size measurements. It also gives the test methods, the inspection rules, the marking and quality certificate, and the packaging, transport and storage.

This standard specifies the requirements, the test methods, the inspection rules, the marking and quality certificate, and the packaging, transport and storage of cerium dioxide nanomaterial.

This standard applies to near-spherical cerium dioxide nano powder materials.

Molecular formula: CeO2.

Relative molecular mass: 172.11 (according to the 2015 international relative atomic masses).

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 191-2008 Packaging - Pictorial marking for handling of goods

GB/T 6678 General principles for sampling chemical products

GB/T 6682-2008 Water for analytical laboratory use - Specification and test methods

GB/T 8170-2008 Rules of rounding off for numerical values and expression and judgement of limiting values

GB/T 12690.2 Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides - Part 2: Determination of loss on ignition in rare earth oxides - Gravimetric method

GB/T 14635-2008 Chemical analysis methods for rare earth metals and their compounds - Determination of total rare earth content

GB/T 18115.2-2006 Chemical analysis methods for rare earth impurities in rare earth

metals and their oxides - Determination of the individual rare earth impurities in cerium

GB/T 19587 Determination of the specific surface area of solids by gas adsorption using the BET method

HG/T 3696.1, HG/T 3696.2 and HG/T 3696.3 Inorganic chemicals - Preparation of standard volumetric solutions, reagents and products for chemical analysis, Parts 1, 2 and 3

3 Requirements

3.1 Appearance: a milky white or pale yellow powder, free from visible foreign matter.

3.2 Cerium dioxide nanomaterial shall comply with the requirements of Table 1.

Table 1 - Performance indicators. Mass fraction of total rare earths (REO): not less than 99.00 percent, tested according to 4.2.1. Mass fraction of rare earth impurities: not more than 0.10 percent, tested according to 4.2.2. Mass fraction of the non-rare-earth impurity Fe2O3: not more than 0.006 percent, tested according to 4.2.3. Mass fraction of the non-rare-earth impurity SiO2: not more than 0.01 percent, tested according to 4.2.3. Mass fraction of the non-rare-earth impurity CaO: not more than 0.035 percent, tested according to 4.2.3. Mass fraction of the non-rare-earth impurity Cl: not more than 0.035 percent, tested according to 4.2.3. Loss on ignition (mass fraction): not more than 1.00 percent, tested according to 4.2.3. Mass fraction of cerium dioxide (CeO2/REO): not less than 99.90 percent, tested according to 4.2.4. Mean particle size by electron microscopy: not more than 100 nm, tested according to 4.3.1. Mean crystallite size d by the XRD line broadening method: not more than 50 nm, tested according to 4.3.2. Specific surface area (BET): not less than 50 m2/g, tested according to 4.4. Agglomeration index T: not more than 200, tested according to 4.5.

4 Test methods

WARNING: some of the reagents used in these test methods are toxic or corrosive and the operator shall be careful. Any splash on the skin shall be washed off at once with water, and in serious cases medical treatment shall be sought immediately. When flammable substances are used, heating with an open flame is strictly forbidden.

4.1 General provisions. Unless otherwise specified, the reagents and the water used in this standard are analytical grade reagents and grade three water as specified in GB/T 6682-2008. Unless otherwise specified, the standard volumetric solutions, the reagents and the products used in the tests are prepared in accordance with HG/T 3696.1, HG/T 3696.2 and HG/T 3696.3.

4.2 Determination of the cerium dioxide and impurity contents. 4.2.1 The total rare earths (REO) are analysed by method 1 of GB/T 14635-2008. 4.2.2 The rare earth impurities are analysed by the method specified in GB/T 18115.2-2006. 4.2.3 The non-rare-earth

impurities and the loss on ignition are analysed by the method specified in GB/T 12690.2. 4.2.4 The cerium dioxide content is calculated as CeO2/REO = [1 - (rare earth impurities / total rare earths REO)] x 100 percent.

4.3.1 Determination of the mean particle size by electron microscopy. A sample of the cerium dioxide is taken and dispersed with an ultrasonic vibrator in a mixed liquid of water and ethanol (volume ratio 1:1) used as the solvent; one or two drops are placed on a prepared thin film, which is put on the specimen stage of the electron microscope. At a magnification of about 100 000 times, an area in which the particles are distinct, uniform and concentrated is selected and photographed. On the photograph the long axis d1 and the short axis ds of not less than 100 individual particles are measured against the nanometre scale bar (a computer program may be used for the statistical processing) and the arithmetic mean is taken. The mean particle size is calculated as the sum of (d1 + ds) divided by 2n, where n is the number of particles measured and the sizes are in nanometres.

4.3.2 Determination of the mean crystallite size by the X-ray diffraction (XRD) line broadening method. The gain of the diffractometer is adjusted so that the height of the diffraction peak of the crystal plane being measured reaches its maximum within the recording range. A polycrystalline silicon standard sample is measured and the half-peak width of its 28.4 degree peak is taken as the reference half-peak width. The half-peak width of the 28.6 degree diffraction peak of the cerium dioxide sample is then measured, and the mean crystallite size d is calculated by the Scherrer formula as 0.89 times the X-ray wavelength divided by the product of the half-peak width in radians and the cosine of the diffraction angle. The X-ray wavelength is 0.154 nm; the measured half-peak width shall be corrected for the doublet and for the instrument factor, and both the half-peak width and the diffraction angle shall be converted to radians in the calculation. The result d is the mean thickness of the crystallite perpendicular to the crystal plane, in nanometres.

4.4 Determination of the specific surface area. About 0.6 g of the cerium dioxide sample is weighed to the nearest 0.001 g and the specific surface area (BET) is determined by the method specified in GB/T 19587.

4.5 Determination of the agglomeration index. About 0.05 g of the cerium dioxide sample is weighed and placed in a 50 mL beaker; two or three drops of sodium hexametaphosphate dispersant are added and the sample is wetted and dispersed by stirring with a glass rod; 10 mL to 20 mL of water are then added and the sample is dispersed ultrasonically for 10 min at a power of 250 W. The volume mean particle size D is measured with a laser particle size analyser (measuring range 0.01 micrometres to 3 500 micrometres) according to its sampling requirements, and the mean crystallite size d is taken from 4.3.2. The agglomeration index T is calculated as T = D/d, where D is the mean diameter in nanometres of the agglomerated particles obtained by the laser particle size analyser after dispersion and d is the mean crystallite size in nanometres obtained in 4.3.2.