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Rare Earth Standard of the People's Republic of China

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Lanthanum-cerium sulfide

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(English Translation)

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Foreword

SAC/TC 229 is in charge of this English translation. In case of any doubt about the contents of the English translation, the Chinese original shall be considered authoritative.

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

This standard was proposed and prepared by SAC/TC 229.

Lanthanum-cerium sulfide

1 Scope

This standard specifies the requirements, testing methods, inspection, labelling, packaging, transportation, storage and quality certificate of lanthanum-cerium sulfide.

This standard is applicable to lanthanum-cerium sulfide produced by lanthanum-cerium oxide , lanthanum-cerium carbonate or other lanthanum-cerium compounds using chemical processes, to be used as pigment plastics, coatings, enamel, glass, rubber, printing ink etc.

2 Normative references

The following referenced 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 1717 *Determination of pH value of an aqueous suspension of pigments*

GB/T 1864 *General methods of test for pigments and extenders-Comparison of colour of pigments*

GB/T 5211.3 *Determination of volatile matter of pigments at 105℃*

GB/T 5211.15 *General methods of test for pigments and extenders-Part15: Determination of oil absorption value*

GB/T 5211.19 *Determination of relative tinting strength and colour on reduction of coloured pigments-Visual comparison method*

GB/T 5211.20 *Comparison of the colour, in full-shade systems, of white ,black and coloured pigments-Colorimetric method*

GB/T 8170 *Rules of rounding off for numerical values & expression and judgement of limiting values*

GB/T 12690.5 *Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides-Part5: Determination of cobalt, manganese, lead,nickel, copper,zinc,aluminum, chromium, magnesium, cadmium.vanadium and iron contents*

GB/T 12690.19 *Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides-Part19: Determination of arsenic and mercury contents*

GB/T 14635 *Rare earth metals and their compounds-Determination of total rare earth content*

GB/T 17803 *Designation system for rare earth products*

GB/T 20170.1 *Test methods for physical characters of rare earth metals and their compounds-Determination for particle size distribution of rare earth compounds*

HG/T 3852 *Determination of residue on sieve of pigments*

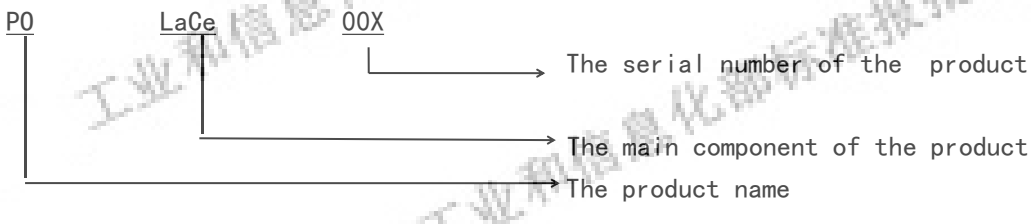
HG/T 3853 *Determination of resistance to heat of dry powder of pigments*

3 Requirements

3.1 Classification

3.1.1 Product designations are designed as $(LaCe)_2S_3$-65Ce and $(LaCe)_2S_3$-50Ce on the chemical composition. The rule for the product designation shall comply with the specification of GB/T 17803.

3.1.2 The corresponding designations to pigment are P0-LaCe-001 and P0-LaCe-002 . The example is shown as follow:



3.2 Chemical composition, physical and chemical properties

Chemical composition of the product shall comply with the requirements in Table 1. The physical and chemical properties of the product shall comply with the requirements in Table2. If the buyer has any special requirement, it shall be negotiated by the supplier and the buyer.

Table 1 Chemical composition

Designation			$(LaCe)_2S_3$-65Ce	$(LaCe)_2S_3$-50Ce
			P0-LaCe-001	P0-LaCe-002
Chemical composition (mass fraction)%	main component	REO	≥78	
		La_2O_3/REO	35±2	50±2
		CeO_2/REO	65±2	50±2
		S	≥18	
	impurity content	Cd	≤0.0001	
		Pb	≤0.0001	
		Hg	≤0.0001	
		Cr	≤0.001	

Table 2 Physical and chemical properties

Designation		$(LaCe)_2S_3$-65Ce	$(LaCe)_2S_3$-50Ce
		P0-LaCe-001	P0-LaCe-002
Physical and chemical properties	Color	Orange	Light orange
	Residue on sieve of 45μm/%	<0.3	
	Median particle size(D[V,50])/μm	2～3	
	Oil absorption / (g/100g)	35～50	
	pH of suspension	7～8	
	Volatile/%	<0.3	
	Heat resistance/℃	300±20	
	Relative coloring strength/%	95～120	
	Total color difference (△E)	≤3	

3.3 Appearance

3.3.1 The product shall be dry powder.

3.3.2 The product shall be clean, without visible inclusions.

4 Testing method

4.1 Chemical composition

4.1.1 Determination of total rare earth content (REO) shall be carried out in accordance with the rules given in GB/T 14635.

4.1.2 Determination of La_2O_3 and CeO_2 components shall be agreed by the supplier and the buyer.

4.1.3 Determination of sulfur content shall be carried out in accordance with the rules given in appendix A.

4.1.4 Determination of lead, chromium, cadmium shall be carried out in accordance with the rules given in GB/T 12690.5.

4.1.5 Determination of mercury shall be carried out in accordance with the rules given in GB/T 12690.19.

4.2 Physical and chemical properties

4.2.1 Measurement of the color shall be carried out using the visual method or instrument method. The visual method shall be carried out in accordance with GB/T 1864. The instrument method shall be carried out in accordance with GB/T 5211.20, GB/T 5211.20 is arbitration method. If the buyer has special requirements, it shall be negotiated by the supplier and buyer.

4.2.2 Measurement of residue on sieve shall be carried out in accordance with HG/T 3852.

4.2.3 Measurement of central particle size(D[V,50]) shall be carried out in accordance with GB/T20170.1.

4.2.4 Determination of oil absorption shall be carried out in accordance with GB/T 5211.15.

4.2.5 Determination of pH of water suspension shall be carried out in accordance with GB/T 1717.

4.2.6 Determination of volatile shall be carried out in accordance with GB/T 5211.3.

4.2.7 Determination of heat resistance shall be carried out in accordance with HG/T 3853.

4.2.8 Determination of relative coloring strength shall be carried out in accordance with GB/T 5211.19. If the buyer has special requirements, it shall be negotiated by the supplier and buyer.

4.2.9 Determination of total color difference (△E) shall be carried out in accordance with GB/T 5211.20.

4.3 Appearance

The appearance of the product should be inspected visually under natural scattering light.

4.4 Numerical rounding

Numerical rounding shall be carried out in accordance with the rules given in GB/T 8170.

5 Inspection rules

5.1 Inspection and acceptance

5.1.1 The supplier shall inspect the product, and guarantee the product quality to comply with the requirement of this standard, and provide the quality certificate.

5.1.2 The buyer shall inspect the quality of the received product in accordance with the rules given in this standard. If the testing results do not comply with this standard, notification of the discrepancies shall be raised to the supplier within two months from the date on receipt of products and solve the problem through negotiation. In case of arbitration, it could be entrusted to an organization approved by both parties, and sampling should be made by both parties on the buyer side.

5.2 Batching

Products shall be submitted in batches for inspection, and each batch of products shall be of the same designation.

5.3 Inspection items

Appearance quality, chemical composition, physical and chemical properties shall be inspected for each batch of products.

5.4 Sampling and sample preparation

The number of sampling for product shall comply with the requirements in Table 3. The sampling amount of each package (bag) shall be no more than 10g. After being mixed, the sample shall be divided to the required amount by quartering method and put into sample bag for sealing.

Table 3 Number of product sampling

Number of packages (bags)	1～5	6～49	50～100	＞100
Number of sampling(bags)	100% of the number of packages (bags)	5	10% of the number of packages (bags) rounded positively to an integer	Square root of the number of packages (bags) rounded positively to an integer

5.5 Judgement of inspection results

5.5.1 If the chemical composition, physical or chemicalproperties analysis result is not in conform with the rules of this standard, double amount of samples shall be taken from the same batch of products for retest. If the results remain unqualified, this batch of products shall be judged as unqualified.

5.5.2 If the result of appearance inspection is not in conform with the rules of this standard, the batch of products shall be judged as unqualified .

6 Labelling, packaging, transportation, storage and quality certificate

6.1 Labelling

Each packages shall include:

a) Name of the supplier;
b) Name and designation of the product;
c) Batch number;
d) Net weight and gross weight;
e) Date of ex-factory and moisture-proof sign or notes.

6.2 Packaging

The products shall be packed in double layered plastic bags with the net weight of 10 kg or 25 kg for each package. The outer packaging shall be the plastic drum, paper box or iron drum, If the buyer have special requirements, both supplier and buyer shall have further negotiation.

6.3 Transportation and storage

Products shall be stored in a ventilated, dry, light-proof , and clean place, and not be exposed in the open air . Products shall be loaded lightly, avoid collision, prevent from the rain, and keep away from acid or alkali items in transportation.

6.4 Quality certificate

Each batch of the product shall be attached with a quality certificate, indicating:

a) Name of the supplier;
b) Name and designation of product;
c) Batch number;
d) Inspection results, stamp of the supplier’s quality inspection department;
e) The standard code;
f) Date of ex-factory.

Appendix A
(Normative)
Chemical analysis for rare earth sulfide
Determination of sulfer content-
Indirect iodine method

A.1 Scope

The appendix specifies determination method of sulfur for lanthanum-cerium sulfide.
The appendix is applicable to the determination method of sulfur content(mass fraction) between 5.00 % and 40.00 % in lanthanum-cerium sulfide.

A.2 Principle

The test portion reacts with hydrochloric acid and comes out as hydrogen sulfide. Then the hydrogen sulfide reacts with the excess iodine standard solution, and the residual iodine content shall be titrated with the sodium thiosulfate standard solution, then indirectly calculate the sulfur content in the rare earth sulfide.

A.3 Reagents

A.3.1 Sodium hyposulfite;

A.3.2 Sodium carbonate;

A.3.3 Reference potassium dichromate;

A.3.4 Potassium iodide;

A.3.5 Iodin;

A.3.6 Sulfuric acid (1+4) ;

A.3.7 Hydrochloric acid (1+1);

A.3.8 Starch (10g/L), weigh 1g of starch and dissolve it in 10 mL water. Adding 90 mL of boiled water while stirring, continue to boiling for 5min, then cool it for later use.

A.3.9 Sodium thiosulfate standard solution

A.3.9.1 Preparation: weigh 26g of sodium thiosulfate (A.3.1) and dissolve it in 1000 mL of water. Add 0.2 g of sodium carbonate (A.3.2) into the solution then slowly boil it for 10 min and cool it for standby. Place the prepared solution in a brown bottle for two weeks, then calibrate it with a reference substance.
A.3.9.2 Calibration: weigh 0.15 g of reference potassium dichromate (A.3.3), pre-dry it at 140℃~150℃ to a constant weight(accurate to 0.0001 g), then dissolve it in an iodine flask with 25 mL of water. Mix the solution well after adding 2 g of potassium iodide (A.3.4) and 20 ml of sulfuric acid (A.3.6), then place it in the dark for 10 min. Titrate with the prepared

sodium thiosulfate standard solution (A.3.9.1) after adding 150 ml water. Add 3 ml starch solution (A.3.8) when near the titration ending point, then titrate the solution until the color turn to bright green from blue. Calibrate 3 times in parallel, and the consumed volume range of three sodium thiosulfate standard solutions (A.3.9.1) shall not exceed 0.10 mL, and calculate the mean value. Blank test shall be carried out in parallel.

A.3.9.3 The molarity concentration of sodium thiosulfate standard solution is calculated by formula (A.1):

$$c_{(Na_2S_2O_3)} = \frac{1000 \times m}{(V - V_0) \times 49.03} \quad \cdots\cdots (A.1)$$

Where:

$c_{(Na_2S_2O_3)}$—is the molarity concentration, in moles per liter (mol/L), of sodium thiosulfate standard solution.

m—is the mass, in gram (g), of potassium dichromate.

V—is the volume, in milliliters (mL), of sodium thiosulfate standard solution consumed.

V_0—is the volume, in milliliters (mL), of sodium thiosulfate standard solution consumed in blank test.

49.03—is the ratio, in grams per mole (g/mol), 1/6 of molar mass of potassium dichromate.

A.3.10 Iodine standard solution

A.3.10.1 Preparation: weigh 35 g of potassium iodide (A.3.4) and dissolve it in 100 mL of water, add 13 g of iodine (A.3.5) into the solution then dissolve, dilute to 1000 mL with water, mix well, place the prepared solution in a brown corked bottle.

A.3.10.2 Calibration: Take 10 mL of iodine standard solution (A.3.10) by a buret and put it into an iodine flask filled with 150 mL of water. Titrate with sodium thiosulfate standard solution (A.3.9), Add 3 mL of starch solution (A.3.8) when near to light yellow, then titrate the solution until the blue disappears. Calibrate 3 times in parallel, and the consumed volume range of three sodium thiosulfate standard solution (A.3.9) shall not exceed 0.10 mL, and calculate the mean value.

A.3.10.3 The molarity concentration of iodine standard solution is calculated by formula (A.2):

$$c_1 = \frac{c \times V}{V_1} \quad \cdots\cdots (A.2)$$

Where:

c_1—is the molarity concentration, in moles per liter (mol/L), of iodine standard solution.

c—is the actual concentration, in moles per liter (mol/L), of sodium thiosulfate standard solution.

V—is the volume, in milliliters (mL), of sodium thiosulfate standard solution consumed.

V_1—is the volume, in milliliters, (mL), of iodine standard solution.

A.4 Procedure

A.4.1 Test portion

Weigh 0.3g of the test sample to the nearest 0.0001 g.

A.4.2 Parallel tests

Take parallel test portions for determination and calculate the mean value.

A.4.3 Blank test

Carry out blank test with the test portion.

A.4.4 Determination

Weigh the test portion (A.4.2) into iodine flask (300 mL), add 35 mL iodine standard solution (A.3.10) accurately by a buret. Add 10 mL hydrochloric acid (A.3.7) slowly (shake the flask while dropping), shake vigorously for 2 min after covering the bottle plug. Titrate with sodium thiosulfate standard solution (A.3.9). Add 5 mL starch solution (A.3.8), When near to light yellow, then continue to titrate until the blue disappears.

A.4.5 Expression of results

The sulfur content, expressed as mass fraction, is calculated by formula(A.3):

$$w_s = \frac{(c_1V_1 - cV)\times 16.03}{1000\times M}\times 100\% \qquad \text{.....(A.3)}$$

Where:

w_s—is the mass fraction, in percent (%), of sulfur in the sample.

c_1—is the molarity concentration, in moles per liter(mol/L), of iodine standard solution.

V_1—is the volume, in milliliters(mL), of iodine standard solution added.

c—is the concentration, in moles per liter (mol/L), of sodium thiosulfate standard solution.

V—is the volume, in moles per liter (mL), of sodium thiosulfate standard solution consumed in titration.

16.03—is the ratio, in grams per liter(g/mol), 1/2 of molar mass of sulfur.

M—is the mass, in grams(g), of the sample.

A.4.6 Tolerance

The difference of the testing results among laboratories shall be no more than the tolerances listed in table A.1.

Table A.1 Tolerance

Mass fraction %	Tolerance %
＞5.00～20.00	0.40
＞20.00～40.00	0.50

A.5 Quality guarantee and control

Verify the validity of the method by standard sample, or controlling sample. Check it at least once a month with the standard sample or controlling sample. Try to find out the cause in case of abnormalities and correct the mistakes then verify it again.