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Anhydrous neodymium chloride

无水氯化钕

*(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 English translation, the Chinese original shall be considered authoritative.

This standard is drafted in accordance with the rules given in the GB/T 1.1-2020 *Directive for standardization—Part* 1: *Structure and drafting of standards*.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. The issuing body of this document shall not be held responsible for identifying any or all such patent rights.

This standard was proposed and prepared by SAC/TC 229 National Technical Committee on Rare Earth of Standardization Administration of China.

Anhydrous neodymium chloride

1 Scope

This document specifies the classification, technical requirements, testing methods, inspection rules, marking, packaging, transport, storage and accompanying documents of Anhydrous neodymium chloride.

This document is applicable to anhydrous neodymium chloride prepared by chemical method, which is used as the raw materials for catalytic materials and other rare earth functional materials.

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

GB/T 12690.7-2021 *Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides—Part* 7; *Determination of silicon content*

GB/T 12690.8-2021 *Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides—Part* 8; *Determination of sodium content*

GB/T 12690.15-2018 *Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides—Part* 15; *Determination of calcium content*

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

GB/T 16484.13 *Chemical analysis method of rare earth chlorides and light rare earth carbonates—Part* 13: *Determination of ammonium chloride content*

GB/T 16484.16 *Chemical analysis method of rare earth chlorides and light rare earth carbonates—Part* 16: *Determination of water insoluble substance content in rare earth chloride—Gravimetry*

GB/T 16484.20 *Chemical analysis method of rare earth chlorides and light rare earth carbonates—Part* 20: *Determination of nickel oxide, manganese oxide, lead oxide, aluminum oxide, zinc oxide, and thorium oxide content—Inductively coupled plasma mass spectrometry*

GB/T 16484.21 *Chemical analysis method of rare earth chlorides and light rare earth carbonates—Part* 21: *Determination of iron oxide content—1,10-phenanthroline spectrophotometry*

GB/T 18115.4 *Chemical analysis methods of rare earth impurities in rare earth metals and their oxides—Part* 4: *Determination of lanthanum, cerium, praseodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium and yttrium contents in neodymium metal and oxide*

GB 39176 *Rare earth products packaging, marking, transport and storage*

3 Terms and definition

There are not any terms and definitions that need to be defined in this document.

4 Classification

4.1 Product classification

Products is divided into three grades according to chemical composition, which are NdCl3(Anhydrous)-4N, NdCl3(Anhydrous)-3N and NdCl3(Anhydrous)-2N5.

4.2 Product grade

The grade of anhydrous neodymium chloride consists two parts. The first part represents neodymium chloride with symbols of elements -- "NdCl3". "(Anhydrous)" means that the product without any crystal water to distinguish from neodymium chloride hydrate. The second part represents the grade of product which is stated by the relative purity of neodymium oxide accounting for total rare earth content of products. When the relative purity (mass fraction) of rare earth in the product is equal to or greater than 99%, it is represented by the quantity of number "9" in the mass percentage and a capital number "N" ("N" is the first letter of "nine"). For example, 99% relative purity is represented by 2N and 99.5% relative purity is represented by 2N5; The first part and second part are separated by a symbol "-".

The concrete representation of label is as follows:

NdCl3 (Anhydrous)- ××

Represent the grade of the product

The chemical formula of neodymium chloride, "Anhydrous" means without water

5 Technical requirements

5.1 chemical composition

The chemical composition of product shall meet the requirements in Table 1. Other special requirements on chemical composition can be negotiated by the supplier and buyer.

Table 1 Product label and corresponding chemical composition

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Grade | | | | NdCl3 (Anhydrous)-4N | NdCl3 (Anhydrous)-3N | NdCl3 (Anhydrous)-2N5 |
| Chemical composi-tion  (mass fraction)  % | Nd2O3/REO, no less than | | | 99.99 | 99.90 | 99.50 |
| REO，no less than | | | 65.80 | 65.80 | 65.80 |
| Impurity content，  max | Rare earth impurity  /REO | La2O3 | 0.001 | 0.005 | 0.02 |
| CeO2 | 0.001 | 0.01 | 0.05 |
| Pr6O11 | 0.003 | 0.05 | 0.30 |
| Sm2O3 | 0.003 | 0.02 | 0.03 |
| Eu2O3 | The combination 0.002 | The combination 0.015 | The combination 0.10 |
| Gd2O3 |
| Tb4O7 |
| Dy2O3 |
| Ho2O3 |
| Er2O3 |
| Tm2O3 |
| Yb2O3 |
| Lu2O3 |
| Y2O3 |
| Non rare earth impurity | Fe2O3 | 0.003 | 0.005 | 0.01 |
| SiO2 | 0.005 | 0.01 | 0.02 |
| CaO | 0.002 | 0.01 | 0.02 |
| NiO | 0.002 | - | - |
| Al2O3 | 0.005 | - | - |
| MnO2 | 0.002 | - | - |
| Na2O | 0.002 | - | - |
| NH4Cl | 1.0 | 1.0 | 1.0 |
| Water insoluble substance(mass fraction)/%，max | | | | 0.10 | | |
| Moisture(mass fraction)/%，max | | | | 0.8 | | |

5.2 Appearance

5.2.1 The product is the powder or granular form, which the color is greyish-purple.

5.2.2 The product shall be clean, without visible inclusions.

6 Testing method

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

6.1.2 The analysis of rare earth impurities shall be carried out in accordance with the rules of GB/T 18115.4. The sample shall be weighed immediately after opened. The mass shall be weighed accurately which is calculated based on the total rare earth content(REO), and dissolved to constant volume according to the method requirements.

6.1.3 The analysis of iron oxide (Fe2O3) shall be carried out in accordance with the rules given in GB/T 16484.21.

6.1.4 The analysis of sodium oxide (Na2O) shall be carried out in accordance with the rules given in GB/T 12690.8-2021 Method 1.

6.1.5 The analysis of silicon dioxide (SiO2) shall be carried out in accordance with the rules given in GB/T 12690.7-2021 Method 1(acid soluble silicon).

6.1.6 The analysis of calcium oxide (CaO) shall be carried out in accordance with the rules given in GB/T 12690.15-2018 Method 1.

6.1.7 The analysis of nickel oxide (NiO), aluminum oxide (Al2O3) and manganese oxide (MnO2) shall be carried out in accordance with the rules given in GB/T 16484.20.

6.1.8 The analysis of ammonium chloride (NH4Cl) shall be carried out in accordance with the rules given in GB/T 16484.13.

6.1.9 The analysis of water insoluble substances shall be carried out in accordance with the rules given in GB/T 16484.16.

6.1.10 The analysis of moisture shall be carried out in accordance with the rules given in Annex A.

6.2 Appearance

Appearance of products shall be inspected visually in nature light.

6.3 Numerical rounding

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

7 Inspection

7.1 Inspection and acceptance

7.1.1 The products shall be inspected by supplier or third testing organizations, and supplier must guarantee the quality to meet this document and the order form.

7.1.2 The buyer shall inspect the quality of the product received in accordance with the rules given in this document. If the testing results don’t meet this document, notification of the discrepancies shall be informed to the supplier within one month from the date of receipt. Both parties shall resolve it through negotiation. In case of arbitration, it can be entrusted to an organization approved by both parties, and sampling should be taken by the both parties on the buyer side.

7.2 Batching

Products should be inspected in batch, and each batch of products should be in the same grade.

7.3 Inspection items

Chemical composition and appearance quality shall be inspected for each batch of products.

7.4 Sampling and sample preparation

7.4.1 Quantity of sampling for chemical composition and appearance quality testing shall meet the requirements in Table 2.

Table 2 Quantity of sampling for chemical composition and appearance quality testing

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Number of packages(buckets) | 1-10 | 11-49 | 50-100 | ＞100 |
| Number of sampling | 100% of the number of packages(buckets) | 10 | 20% of the  number of packages(buckets) | Square root of the double number of packages(buckets) rounded up to an integer |

7.4.2 Sampling method for chemical composition testing

Product sampling shall be carried out in a dry inert gas protected glove box. Three points should be taken at center as well as equal distances around the center of each piece(bag) using a non-metallic sampler. The sampling amount for each piece(bag) shall not be less than 10g. After mixed uniform, samples shall be quickly shrunk to the required quantity by quartering method and placed in vacuum-packed bags. Then these bags shall be sealed in vacuum immediately and stored in the desiccator.

7.5 Judgement of inspection results

7.5.1 In case of any unqualified inspection results of chemical composition, double amount of samples shall be taken from the same batch of products for retest. If the results remain unqualified, this batch of products should be judged as unqualified.

7.5.2 If the results of appearance inspections are not in conformity with this document, this batch of products should be judged unqualified directly.

8 Marking, packaging, transport, storage and accompanying document

8.1 Packaging

The minimum product packaging adopts foil laminated film bags lined with plastic bags, and it is required to seal in vacuum before put into monolayer plastic bags. then it is further packaged into iron buckets. The inner wall of the iron buckets need to be lined with shock-absorbing materials which are also used to separate and fix the vacuum packaged products. The net weight of minimum product packaging is 5kg. Each bucket has a net weight of 25 kg or 50 kg. If the buyer has special requirements for packaging, it can be negotiated by the supplier and the buyer.

8.2 Marking, transport and storage

Marking, transport and storage of products shall be carried out in accordance with the rules given in GB/T 39176.

8.3 Accompanying documents

Each batch of the products shall be attached with accompanying documents including the quality certificate. The quality certificate shall be carried out in accordance with the rules given in GB 39176.Besides that, it also should contain:

a) Product certification;

b) Inspection report in the process of quality control and finished product inspection report;

c) Instruction book;

d) Others.

Annex A

(annex normative)

Determination of moisture in anhydrous neodymium chloride

A.1 Method summary

The following operations are all in glove box. First, a certain amount of sample is weighed under the inert atmosphere and placed in a moisture bottle with a lid. The sample is heated in the Coulomb Karl Fischer moisture meter. Moisture of the sample will enter the meter with inert gas. The power of electrolysed iodine in Karl Fisher reagent is equivalent to the power of electrolysed water. Moreover, the power of electrolysed iodine is proportional to its mass consumed according to Faraday's law of electrolysis. Thus, moisture content of the sample can be calculated by measuring the power consumed.

A.2 Reagents and materials

A.2.1 A moisture bottle with a lid: The moisture content shall be less than 20 ppm under sealed conditions.

A.2.2 High-purity argon gas: volume fraction ≥ 99.99%.

A.2.3 Karl Fisher electrolyte:

Preparation of Karl Fisher reagent：

Solution A: Dissolve 50g of iodine in 80mL of pyridine and shake until all iodine is dissolved. Then add 260mL of anhydrous methanol into it and orange crystals will appear.

Solution B: Inject dried sulfur dioxide into 40mL of pyridine to make into 120mL of orange solution.

Drip solution B into solution A slowly in an ice bath. At the same time, the crystals of solution A slowly dissolves to obtain a dark brown Karl Fischer reagent. After the solution cools to room temperature, cork the bottle and store it in the desiccator for 24 hours before use.

Attention: When preparing, diluting, and using Karl Fischer reagent, it should be done in a well ventilated area. Karl Fischer reagent is strongly corrosive and highly toxic, avoid contact with skin and inhalation into the body.

Preparation of Karl Fischer electrolyte:

Preparation of the anode electrolyte: Chloroform, methanol and Karl Fischer reagent are mixed in a 3:3:1 ratio and the mixture is stored in a stoppered brown bottle.

Attention: If the solution is light yellow or shows excessive moisture content, high concentrations of Karl Fischer reagent can be added to consume the moisture. But the dosage of Karl Fischer reagent shall not exceed 20mL. Otherwise it is necessary to choose a new solvent with less moisture or a high concentrations Karl Fischer reagent to prepare the electrolyte.

The cathode electrolyte is the same as the anode electrolyte.

A.3 Apparatuses

A.3.1 Karl Fischer moisture meter: The sensitivity is 0.01ugH2O; It shall meet the requirement of JJG 1154-2018;

A.3.2 Circulating glove box: Water content is less than 0.0001%; Oxygen content is less than 0.0003%.

A.3.3 Analytical balance: Minimum scale value is 0.1 mg.

A.3.4 Cassette furnace: temperature ≥ 260 ℃.

A.4 samples

The sealed samples shall be weighed immediately after opened in the glove box.

A.5 Testing procedures

A.5.1 Test portion

Samples are weighed according to Table A.1. The results are accurate to 0.0001g. Then, put them into a moisture bottle with a lid. Pay attention to the distance between the needle and the surface of the sample to avoid clogging the needle.

Table A.1 Sample weighting

| Moisture content  (mass fraction)/% | 0.0001-0.20 | 0.20-1.0 |
| --- | --- | --- |
| Sample weighting/g | 1.0 | 0.5 |

A.5.2 Parallel test

Two-parallel test of the products shall be carried out.

A.5.3 Blank test

Open the Coulomb Karl Fischer moisture meter and place the blank water bottles with lids on the Cassette furnace in sequence. Then carry out the blank test according to the selected measurement conditions, and repeat the test until the numerical value stabilizes.

A.5.4 Calibration of the apparatus

The accuracy of Karl Fischer reagent can be tested by injecting a known moisture content reagent regularly, And detection intervals may be carried out at the initial use of new reagents or after every ten measurements.

A.5.5 Determination

A.5.5.1 Put an appropriate amount of electrolyte into the electrolytic cell, according to the requirements of the Karl Fischer moisture meter, Then turn on the apparatus, and open the magnetic stirring device subsequently. At the same time, adjust the stirring speed to be constant.

A.5.5.2 Place the sample moisture bottles on the Cassette furnace, and heat it up to 260 ℃ to measure the sample. Record the results after the numerical value stabilizes.

A.6 Test data processing

The testing results can be got through reading the apparatus display directly and retain two significant digits.