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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 12690.19-2018

Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides - Part 19: Determination of arsenic and mercury contents

稀土金属及其氧化物中非稀土杂质化学分析方法 第19部分：砷、汞量的测定

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Foreword

GB/T 12690 "Chemical Analysis Methods for Non-rare Earth Impurities in Rare Earth Metals and Their Oxides" is divided into 19 parts.

--- Part 1. Determination of carbon and sulfur content - High frequency - infrared absorption method;

--- Part 2. Determination of the amount of ignition in rare earth oxides;

---Part 3. Determination of moisture content in rare earth oxides;

--- Part 4. Determination of oxygen and nitrogen - pulse - infrared absorption method and pulse - heat conduction method;

---Part 5. Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium, iron;

---Part 6. Determination of iron content - Potassium thiocyanate, 1,10- phenanthroline spectrophotometric method;

--- Part 7. Determination of silicon content - Molybdenum blue spectrophotometric method;

---Part 8. Determination of sodium content by flame atomic absorption spectrometry;

---Part 9. Determination of chlorine content - silver nitrate turbidimetric method;

---Part 10. Determination of phosphorus content - Molybdenum blue spectrophotometric method;

---Part 11. Determination of magnesium content by flame atomic absorption spectrometry;

--- Part 12. Determination of enthalpy;

--- Part 13. Determination of the amount of molybdenum and tungsten;

1 Scope

GB/T 12690.19-2018 is the Chinese national standard on chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides - part 19: determination of

arsenic and mercury contents, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 17 September 2018 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 June 2019. Classification: ICS 77.120.99, CCS H14. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This part of GB/T 12690 specifies the method for determining the amount of arsenic and mercury in rare earth metals and their oxides. This section applies to the determination of the amount of arsenic and mercury in rare earth metals and their oxides. A total of 2 methods are included. Method

1 Inductively Coupled Plasma Mass spectrometry, method 2 atomic fluorescence method. Method 1 measurement range. 0.00005%~0.010%, method 2 measurement range. arsenic 0.00005%~ 0.010%; mercury 0.000005%~0.0010%. When method 1 and method 2 overlap in the measurement range, method 2 is used as arbitration. method.

2 Method 1. Inductively coupled plasma mass spectrometry

2.1 Principle of the method The sample is dissolved in nitric acid, and the argon plasma is used as the ionization source in the dilute nitric acid medium, and the standard mode and the reaction gas mode are used for direct measurement. Determine the base interference to correct the matrix interference.

2.2 Reagents

2.2.1 Hydrogen peroxide (30%), excellent grade.

2.2.2 Nitric acid (1 1).

2.2.3 Nitric acid (1 3).

2.2.4 Nitric acid (2 98).

2.2.5 Sodium hydroxide, excellent grade.

2.2.6 sodium hydroxide solution (100g/L).

2.2.7 Arsenic standard storage solution. accurately weigh 0.1320g of arsenic trioxide dried to constant weight in a sulfuric acid dryer, warmly dissolved in hydrogen and oxygen Sodium solution (2.2.6), transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix. 1 mL of this solution contained 100 μg of arsenic.

2.2.8 Mercury standard storage solution. accurately weigh 0.1350g of mercury chloride, dissolve it in water, transfer it to a 1000mL volumetric flask and dilute it to the mark with water. Mix well. 1 mL of this solution contained 100 μg of mercury. 2.2.9 standard storage solution. accurately weigh 0.2320g lanthanum nitrate, dissolve it with 10mL nitric acid solution (2.2.4), and transfer it into 1000mL capacity. Dilute to the mark with water and mix well. 1 mL of this solution contained 100 μg of hydrazine. 2.2.10 Standard storage solution. accurately weigh 0.1267g of cerium chloride, dissolve it in a small amount of water, and transfer it to a 1000mL volumetric flask. Release to the scale and mix. 1 mL of this solution contained 100 μg of hydrazine.

2.2.11 Mixed standard solution. separately remove 10.00mL of standard storage solution of arsenic and mercury (2.2.7~2.2.8) in a 100mL volumetric flask. Add 10 mL of nitric acid (2.2.2), dilute to the mark with water, and mix. 1 mL of this solution contained 10 μg of arsenic and mercury, respectively.

2.2.12 Mixed internal standard solution. Transfer 10.00mL of each standard solution of and (2.2.9~2.2.10) to 100mL volumetric flask Add 10 mL of nitric acid (2.2.2), dilute to the mark with water, and mix. 1mL of this solution contains 10μg of strontium and strontium, respectively, and then dilute the solution. Released to 1 mL of 1 μg of hydrazine and hydrazine standard solution.

2.2.13 Argon [phi(Ar) >= 99.999%].