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稀土金属及其氧化物中非稀土杂质

化学分析方法

第5部分：钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒、铁量的测定

**Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides**

**Part 5: Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron contents**

（送审稿）

**GB/T12960.5—201X**

代替Replacement of GB/T 12690.5-2003

中华人民共和国国家标准

NATIONAL STANDARD OF THE PEOPLE’SREPUBLIC OF CHINA

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前言Foreword

本标准按照GB/T 1.1-2009 给出的规则起草。

This national standard was drafted according to the specification from GB/T 1.1-2009

GB/T 12690《稀土金属及其氧化物中非稀土杂质化学分析方法》共分为18个部分：

GB/T 12690“Chemical analysis methods fornon-rare earth impurities inrare earth metals and their oxides”consists of the following 18 parts:

――第1部分：碳、硫量的测定高频-红外吸收法；

――Part 1: Determination of Carbon, SulfurHigh frequency infrared absorptionspectrometric method

――第2部分：稀土氧化物中灼减量的测定重量法；

――Part 2: Determination of loss on ignition in the rare earth-Oxide Gravimetric method

――第3部分：稀土氧化物中水分量的测定重量法；

――Part 3: Determination of moisture in the rare earth-Oxide Gravimetric method

――第4部分：氧、氮量的测定脉冲-红外吸收法和脉冲-热导法；

――Part 4: Determination ofoxygen, nitrogenPulse-infrared absorptionspectrometric method and Pulse-thermal conductivity method

――第5部分：钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒、铁量的测定

――Part 5: Determination of Cobalt, Manganese, Lead, Nickel, Copper,

――第6部分：铁量的测定硫氰酸钾、1，10-二氮杂菲分光光度法；

――Part 6: Determination of ironKalium thiocyanate, 1, 10-phenanthroline spectrophotometric method

――第7部分：硅量的测定钼蓝分光光度法；

――Part 7: Determination of silicon Molybdenum blue photometric method

――第8部分：钠量的测定火焰原子吸收光谱法；

――Part 8: Determination of sodium Flame Atomic Absorption Spectrometry

――第9部分：氯量的测定硝酸银比浊法；

――Part 9: Determination of chlorine Silver nitrate turbidimetric method

――第10部分：磷量的测定钼蓝分光光度法；

――Part 10: Determination of phosphorusMolybdenum blue photometric method

――第11部分：镁量的测定火焰原子吸收光谱法；

――Part 11: Determination of Magnesium Flame Atomic Absorption Spectrometry

――第12部分：钍量的测定；

――Part 12: Determination of thorium

――第13部分：钼、钨量的测定；

――Part 13: Determination of Molybdenum and tungsten

――第14部分：钛量的测定；

――Part 14: Determination of Titan

――第15部分：钙量的测定；

――Part 15: Determination ofcalcium

――第16部分：氟量的测定离子选择性电极法；

――Part 16: Determination of fluorine ion selective electrode meth

――第17部分：稀土金属中铌、钽量的测定；

――Part 17: Determination of Niobium and tantalum in rare earth metals

――第18部分：锆量的测定。

――Part 18: Determination of zirconium

本部分为第5部分。

This is part 5.

本部分代替GB/T 12690.5-2003《稀土金属及其氧化物中非稀土杂质化学分析方法铝、铬、锰、铁、钴、镍、铜、锌、铅的测定电感耦合等离子体发射光谱法；钴、锰、铅、镍、铜、锌、铝、铬的测定电感耦合等离子体质谱法》。本部分包含两个分析方法，方法1为电感耦合等离子体原子发射光谱法，方法2为电感耦合等离子体质谱法。当两个方法的分析范围出现重叠时，以方法2作为仲裁方法。

This part replace the GB/T 12690.5-2003, “Chemical analysis methods fornon-rare earth impurities inrareearth metals and their oxides- Part 5: Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron contents, inductively coupled plasma atomic emission spectrometric method; Cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium determination of inductively coupled plasma mass spectrometric method”

This partconsists oftwo analytical methods. The method 1 is inductively coupled plasma atomic emission spectrometric method. The method 2 is inductively coupled plasma mass spectrometric method.Method 2 is selected as the arbitration method in the overlapping content range of the two methods.

本部分与GB/T 12690.5-2003 相比，除编辑性修改外主要技术变化如下：

There are some significant technical changes in this documentover its previous edition. They are as follows:

——扩大了方法1的适用范围，新增了镁、镉、钒含量的测定（见表1）；

——对方法1中铝、铬、铁、钴、镍、铜、铅含量的测定范围进行了调整（见表1）；

——方法1中新增了Pr、Sm、Tb、Dy、Ho、Er、Tm、Yb、Lu 中的非稀土杂质分析谱线（见表5）；

——方法1中根据被测元素的含量范围调整了相应的进样浓度（见2.5.4）；

——方法2增加了镁、镉、钒的测定（见表2）；

——Supplement thedetermination of magnesium, cadmium and vanadiumin method 2. (Seen in Table 2)

——方法2铅、镍的测定范围下限由0.0002%改为0.0001%，锌、铝的测定范围下限由0.0005%改为0.0003%，铬的测定范围下限由0.0005%改为0.0001%（见表2）。

——Changethe lower limit for the determination range of lead and nickel from 0.0002% to 0.0001% in method 2;

Change the lower limit for the determination range of zinc and aluminum from 0.0005% to 0.0003% in method 2;

Change the lower limit of chromium range from 0.0005% to 0.0001% in method 2.(Seen in Table 2)

本部分由全国稀土标准化技术委员会（SAC/TC 229）提出并归口。

This partis proposed byChina Rare Earth Standardization Technical Committee（SAC/TC 229）,of which this part is under the jurisdiction.

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The previous versions of this part are:

——GB/T 8762.4-1998,GB/T 8762.6-1988；

——GB/T 11074.4-1989；

——GB/T 12690.14-1990, GB/T 12690.19-1990,GB/T 12690.24-1990；

——GB/T 12690.5-2003.

稀土金属及其氧化物中非稀土杂质化学分析方法 第5部分：钴、锰、铅、镍、铜、锌、铝、铬、 镁、镉、钒、铁量的测定

**Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides**

**Part 5: Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium andiron contents**

1. **范围Scopeand field of application**

本方法规定了稀土金属中钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒、铁含量及其氧化物中氧化钴、氧化锰、氧化铅、氧化镍、氧化铜、氧化锌、氧化铝、氧化铬、氧化镁、氧化镉、氧化钒、氧化铁含量的测定方法。

This standard method specifies the determination ofcobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron in the rare earthmetals and their oxides in the rare earth oxides.

本方法适用于稀土金属中钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒、铁含量及其氧化物中氧化钴、氧化锰、氧化铅、氧化镍、氧化铜、氧化锌、氧化铝、氧化铬、氧化镁、氧化镉、氧化钒、氧化铁含量的测定。共包含两个方法：方法1电感耦合等离子体发射光谱法，方法2电感耦合等离子体质谱法。方法1测定范围见表1，方法2测定范围见表2。

This standard method is applicable to determination ofcobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium,magnesium, cadmium, vanadium and iron in the rare earthmetals and their oxides in the rare earthoxides. This part consists of two methods:

* Method 1: inductively coupled plasma atomic emission spectrometric method
* Method 2: inductively coupled plasma mass spectrometric method

The determination ranges for Method 2 are shown in Table 2.

表1

|  |  |  |  |
| --- | --- | --- | --- |
| 氧化物 | 测定范围（质量分数）/% | 氧化物 | 测定范围（质量分数）/% |
| 氧化钴 | 0.0010～0.10 | 氧化铝 | 0.0010～0.10 |
| 氧化锰 | 0.0010～0.10 | 氧化铬 | 0.0010～0.10 |
| 氧化铅 | 0.0010～0.10 | 氧化镁 | 0.0002～0.10 |
| 氧化镍 | 0.0010～0.10 | 氧化镉 | 0.0010～0.10 |
| 氧化铜 | 0.0010～0.10 | 氧化钒 | 0.0010～0.10 |
| 氧化锌 | 0.0010～0.10 | 氧化铁 | 0.0010～0.50 |

Table 1

|  |  |  |  |
| --- | --- | --- | --- |
| RE oxides | Ranger (Mass fraction)/% | RE oxides | Ranger (Mass fraction)/% |
| Cobalt oxide | 0.0010～0.10 | Aluminum oxide | 0.0010～0.10 |
| Manganese oxide | 0.0010～0.10 | Chromium oxide | 0.0010～0.10 |
| Lead oxide | 0.0010～0.10 | Magnesium oxide | 0.0002～0.10 |
| Nickel oxide | 0.0010～0.10 | Cadmium oxide | 0.0010～0.10 |
| Copper oxide | 0.0010～0.10 | Vanadium oxide | 0.0010～0.10 |
| Zinc oxide | 0.0010～0.10 | Iron oxide | 0.0010～0.50 |

表2

|  |  |  |  |
| --- | --- | --- | --- |
| 氧化物 | 测定范围（质量分数）/% | 氧化物 | 测定范围（质量分数）/% |
| 氧化钴 | 0.0001～0.050 | 氧化铝 | 0.0003～0.050 |
| 氧化锰 | 0.0001～0.050 | 氧化铬 | 0.0001～0.050 |
| 氧化铅 | 0.0001～0.050 | 氧化镁 | 0.0001～0.050 |
| 氧化镍 | 0.0001～0.050 | 氧化镉 | 0.0001～0.050 |
| 氧化铜 | 0.0001～0.050 | 氧化钒 | 0.0001～0.050 |
| 氧化锌 | 0.0003～0.050 | - | - |

Table 2

|  |  |  |  |
| --- | --- | --- | --- |
| RE oxides | Mass fraction/% | RE oxides | Mass fraction/% |
| Cobalt oxide | 0.0001～0.050 | Aluminum oxide | 0.0003～0.050 |
| Manganese oxide | 0.0001～0.050 | Chromium oxide | 0.0001～0.050 |
| Lead oxide | 0.0001～0.050 | Magnesium oxide | 0.0001～0.050 |
| Nickel oxide | 0.0001～0.050 | Cadmium oxide | 0.0001～0.050 |
| Copper oxide | 0.0001～0.050 | Vanadium oxide | 0.0001～0.050 |
| Zinc oxide | 0.0003～0.050 | - | - |

2 方法1：电感耦合等离子体发射光谱法 Method 1: Inductively coupled plasma atomic emission spectrometry

2.1 方法原理 Principle

试样以硝酸溶解，在稀硝酸介质中，直接以氩等离子体光源激发，进行光谱测定。

The sample is dissolved in nitric acid, and then directly excited by the light source of argon inductively coupled plasma in the medium of dilute nitric acid.

2.2 试剂和材料 Reagents

2.2.1 过氧化氢（30%），优级纯。Hydrogen peroxide, H2O2, 30%, GR.

2.2.2 盐酸（ρ 1.19 g/mL），优级纯。 Hydrochloric acid (conc), HCl, ρ 1.19 g/mL, GR.

2.2.3 硝酸（ρ 1.42 g/mL），UL级。Nitric acid (conc), HNO3, ρ 1.42 g/mL, UL.

2.2.4 盐酸（1+1），优级纯。Hydrochloric acid, HCl (1+1), GR.

2.2.5 硝酸（1+1），UL级。Nitric acid, HNO3 (1+1), UL.

2.2.6钴标准贮存溶液：准确称取0.2000g金属钴[*w*（Co）≥99.9％]于200mL烧杯，加10mL水，加20mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200mL容量瓶中，用水稀释至刻度，摇匀。此溶液1mL含1.0mg钴。

2.2.6 Cobalt standard stock solution, 1 mL = 1.0 mg of cobalt: Weigh out 0.2000 g of cobalt metal (99.9% minimum) into a 200 ml beaker, add 10 ml of water and 20 ml of nitric acid (2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.7 锰标准贮存溶液：准确称取0.2000g金属锰[*w*（Mn）≥99.9％]于200mL烧杯，加10mL水，加20mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200mL容量瓶中，用水稀释至刻度，摇匀。此溶液1mL含1mg锰。

2.2.7 Manganese standard stock solution, 1 mL = 1.0 mg of manganese: Weigh out 0.2000g of manganese metal (99.9% minimum) into a 200 ml beaker, add 10 ml of water and 20 ml of nitric acid (2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.8 镍标准贮存溶液：准确称取0.2000 g金属镍粉[*w*（Ni）≥99.9％]于200 mL烧杯，加10mL水，加20 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1mg镍。

2.2.8 Nickel standard stock solution, 1 mL = 1.0 mg of nickel: Weigh out 0.2000g of nickel metal powder (99.9% minimum) into a 200ml beaker, add 10 ml of water and 20 ml of nitric acid (2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.9 铜标准贮存溶液：准确称取0.2000 g金属铜[*w*（Cu）≥99.9％]于200 mL烧杯，加10mL水，加20 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg铜。

2.2.9 Copper standard stock solution, 1 mL = 1.0 mg of copper: Weigh out 0.2000g of copper metal (99.9% minimum) into a 200ml beaker, add 10 ml of water and 20 ml of nitric acid (2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.10 铁标准贮存溶液：准确称取0.2000 g金属铁粉[*w*（Fe）≥99.9％]于200 mL烧杯，加10mL水，加20 mL盐酸（2.2.4），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1mg铁。

2.2.10 Iron standard stock solution, 1 mL = 1.0 mg of iron: Weigh out 0.2000g of metal iron powder (99.9% minimum) into a 200ml beaker, add 10 ml of water and 20 ml of hydrochloric acid (2.2.4), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.11 铝标准贮存溶液：准确称取0.2000 g金属铝箔[*w*（Al）≥99.9％]（预先用稀盐酸浸泡，经无水乙醇清洗，用红外灯烘干）于200 mL烧杯中，加10mL水，加20 mL盐酸（2.2.4），滴加2 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg铝。

2.2.11 Aluminum standard stock solution, 1 mL = 1.0 mg aluminum: Weigh out 0.2000g of aluminum foil (99.9% minimum) (pretreated by soaking in dilute hydrochloric acid, then washing with absolute ethyl alcohol and drying under infrared lamp)into a 200ml beaker, add 10 ml of water,20 ml of hydrochloric acid(2.2.4) and 2ml nitric acid(2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.12锌标准贮存溶液：准确称取0.2000 g 金属锌粒[*w*（Zn）≥99.9％]，置于300 mL烧杯，加20 mL盐酸（2.2.4），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg锌。

2.2.12 Zinc standard stock solution, 1 mL = 1.0 mg zinc: Weigh out 0.2000g of metal zinc granule (99.9% minimum) into a 300ml beaker, add 20 ml of hydrochloric acid(2.2.4), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask,dilute to volume with water and mix well.

2.2.13 铬标准贮存溶液：准确称取0.2000 g金属铬[*w*（Cr）≥99.9％] 于200 mL烧杯，加10mL水，加20 mL盐酸（2.2.4），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1mg铬。

2.2.13 Chromium standard stock solution, 1 mL = 1.0 mg chromium: Weigh out 0.2000g of chromium metal (99.9% minimum) into a 200ml beaker, add 10 ml of water and 20 ml of hydrochloric acid (2.2.4), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.14铅标准贮存溶液：准确称取0.2000 g金属铅[*w*（Pb）≥99.9％] ]于200 mL烧杯，加10mL水，分次加入20 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1mg铅。

2.2.14 Lead standard stock solution, 1 mL = 1 mg lead: Weigh out 0.2000g of metal lead powder (99.9% minimum) into a 200ml beaker, add 10 ml of water, and add 20 ml of nitric acid (2.2.5) for several times, heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.15镁标准贮存溶液：称取0.3317 g经800 ℃灼烧的氧化镁[*w*（MgO）≥99.9％]于200 mL烧杯中，加10 mL盐酸(2.2.4)，加热至溶解完全，冷却至室温。移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg镁。

2.2.15 Magnesium standard stock solution, 1 mL = 1.0 mg magnesium: Weigh out 0.3317g of magnesium oxide (99.9% minimum),which is previously ignited at 800 °C, into a 200ml beaker, add 10 ml of hydrochloric acid(2.2.4), heat until completely dissolved. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.16 镉标准贮存溶液：准确称取0.2000 g金属镉[*w*（Cd）≥99.9％]于200 mL烧杯，加10mL水，加20 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg镉。

2.2.16 Cadmium standard stock solution, 1 mL = 1.0 mg cadmium: Weigh out 0.2000g of cadmium metal(99.9% minimum) into a 200ml beaker, add 10 ml of water and 20 ml of nitric acid(2.2.5), heat gently at moderate temperature. Cool to room temperature. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.17钒标准贮存溶液：称取0.4592 g偏钒酸铵（优级纯）[*w*（NH4VO3）≥99.9％]于200 mL烧杯，溶于适量水中，用硝酸（2.2.5）中和至酸性，溶液移入200 mL容量瓶中，用水稀释至刻度，混匀。此溶液1 mL含1 mg钒。

2.2.17 Vanadium standard stock solution, 1 mL = 1.0 mg vanadium: Weigh out 0.4592g of ammonium metavanadate (G.R.,99.9%minimum) into a 200ml beaker, dissolve in moderate water, and add nitric acid(2.2.5) to neutralize the solution to be acidic. Transfer into a 200 ml volumetric flask, dilute to volume with water and mix well.

2.2.18 稀土基体溶液：称取10.0000g经950℃灼烧1h的单一稀土氧化物[*w*（REO/∑REO）≥99.99%，*w*（∑REO）≥99.5%，*w*（Co）＜0.0001%，*w*（Mn）＜0.0001%，*w*（Pb）＜0.0001%，*w*（Ni）＜0.0001%，*w*（Cu）＜0.0001%，*w*（Zn）＜0.0001%，*w*（Al）＜0.0001%，*w*（Cr）＜0.0001%，*w*（Mg）＜0.0001%，*w*（Cd）＜0.0001%，*w*（V）＜0.0001%，*w*（Fe）＜0.0001%]，置于500mL烧杯中，加50mL硝酸(2.2.5)，低温加热至溶解完全，取下冷却。移入100mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含100mg单一稀土氧化物。

2.2.18 Rare earth matrix solution 1 mL = 100 mg of single rare earth oxide: Weigh out 10.0000g of single rare earth oxide (w(REO/∑REO) ≥ 99.99%, w(∑REO) ≥ 99.5%, w(Co) < 0.0001%, w(Mn) < 0.0001%, w(Pb) <0.0001%, w(Ni) < 0.0001%, w(Cu) < 0.0001%, w(Zn) < 0.0001%, w(Al) < 0.0001%, w(Cr) < 0.0001%, w(Mg) < 0.0001%, w(Cd) < 0.0001%, w(V) < 0.0001%, w(Fe) < 0.0001%), which is previously ignited at 950 ºC for 1 h, into a 500ml beaker, add 50 ml of nitric acid (2.2.5) and heat gently at a moderate temperature until completely dissolved. Cool to room temperature. Transfer into a 100 ml volumetric flask, dilute to volume with water and mix well.

2.2.19混合标准溶液I：分别移取钴标准贮存溶液（2.2.6）、锰标准贮存溶液（2.2.7）、镍标准贮存溶液（2.2.8）、铜标准贮存溶液（2.2.9）、铝标准贮存溶液（2.2.11）、锌标准贮存溶液（2.2.12）、铬标准贮存溶液（2.2.13）、铅标准贮存溶液（2.2.14）、镁标准贮存溶液（2.2.15）、镉标准贮存溶液（2.2.16）、钒标准贮存溶液（2.2.17）各10.00 mL溶液移入200 mL容量瓶中，加20mL硝酸(2.2.5),用水稀释至刻度，摇匀。此溶液为混合标准溶液I，1 mL含钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒各50 μg。

2.2.19 Mixed standard solution I, 1 mL = 50 µg of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium: Transfer 10.00 ml of each standard stock solution of cobalt (2.2.6) , manganese (2.2.7), nickel (2.2.8), copper (2.2.9), aluminum (2.2.11), zinc (2.2.12), chromium (2.2.13),lead (2.2.14),magnesium (2.2.15), cadmium (2.2.16), vanadium (2.2.17) to a 200 ml volumetric flask, add 20 ml of nitric acid (2.2.5),dilute to volume with water and mix well.

2.2.20混合标准溶液II：移取混合标准溶液I(2.2.19) 20.00 mL于100 mL容量瓶中，加10mL硝酸(2.2.5),用水稀释至刻度，摇匀。此溶液为混合标准溶液II，1 mL含钴、锰、铅、镍、铜、锌、铝、铬、镁、镉、钒各10ug。

2.2.20 Mixed standard solution II, 1 mL = 10 µg of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium: Transfer 10.00 ml of mixed standard solution I (2.2.19) to a 100 ml volumetric flask, add 10 ml of nitric acid (2.2.5), dilute to volume with water and mix well.

2.2.21 铁标准溶液I：移取铁标准贮存溶液(2.2.10) 20.00 mL于200 mL容量瓶中，加20mL硝酸(2.2.5),用水稀释至刻度，摇匀。此溶液为铁标准溶液I，1 mL含铁100μg。

2.2.21 Iron standard solution I, 1 mL = 100 µg of iron: Transfer 20.00 ml of iron standard stock solution (2.2.10) to a 200 ml volumetric flask, add 20 ml of nitric acid (2.2.5), dilute to volume with water and mix well.

2.2.22铁标准溶液II：移取10.00 mL铁标准溶液I(2.2.21)于100 mL容量瓶中，加10mL硝酸(2.2.5),用水稀释至刻度，摇匀。此溶液为铁标准溶液II，1 mL含铁10 μg。

2.2.22 Iron standard solution II, 1 mL = 10 µg of iron: Transfer 10.00 ml of iron standard stock solution (2.2.21) to a 100 ml volumetric flask, add 10 ml of nitric acid (2.2.5), dilute to volume with water and mix well.

2.3 仪器设备Apparatus

2.3.1 电感耦合等离子体发射光谱仪，分辨率<0.006nm（200nm处）。

2.3.1 Inductively coupled plasma atomic emission spectrometer, with resolution<0.006 nm (at 200 nm).

2.3.2 氩等离子体光源，使用功率不大于2kw。

2.3.2 Argon inductively coupled plasma light source, the operating power is less than 2kw.

2.4 试样 Test sample

2.4.1氧化物试样于105 ℃烘1 h，置于干燥器中，冷却至室温，立即称样。

2.4.1 The oxide test sample is dried at 105 ºC for 1 h, and then kept in a desiccator, cooled to room temperature, weighed immediately.

2.4.2 金属试样应去掉表面氧化层，取样后立即称样。

2.4.2 The metal test sample is peeled off the oxide surface, weighed immediately after sampling.

2.5 分析步骤 Analytical procedures

2.5.1 试料Test portion

称取0.5g试样（试样2.4），精确到0.0001g。

Weigh out 0.5g test sample (2.4), accurate to 0.0001g.

2.5.2 测定次数Parallel determination

称取两份试料（2.5.1）进行平行测定，取其平均值。

Weigh two test portions (2.5.1) for parallel determination. Take their average value.

2.5.3 空白试验 Blank

随同试料做空白试验。

Blank is prepared along with the test sample.

2.5.4 分析试液的制备 Preparation of analytical test solution

2.5.4.1 将试料（2.5.1）（除氧化铈外）置于100mL烧杯中，加水润湿，加入5mL硝酸（2.2.5），低温加热至溶解完全，冷却至室温，移入50mL容量瓶中，用水稀释至刻度，摇匀。待测。

2.5.4.1 Place the test portion (2.5.1, except cerium oxide) into a 100 ml beaker, add water to make the sample wet and add 5 ml of nitric acid (2.2.5), heat gently at moderate temperature until completely dissolved. Cool to room temperature. Transfer into a 50 ml volumetric flask, dilute to the mark with water and mix well.

2.5.4.2 将试料（2.5.1）（氧化铈）置于100mL烧杯中，加水润湿，加入10mL硝酸（2.2.5），滴加过氧化氢（2.2.1）低温加热至溶解完全并蒸至溶液呈黄色，不再有小气泡出现，冷却至室温，移入50mL容量瓶中，用水稀释至刻度，摇匀。待测。

2.5.4.2 Place the test portion (2.5.1)(cerium oxide) into a 100 ml beaker, add some water to make the sample wet and add 10 ml of nitric acid (2.2.5) and hydrogen peroxide (3.1) dropwise, heat gently at moderate temperature until completely dissolved. Boil the solution till no more small bubbles and yellow color appears. Cool to room temperature. Transfer into a 50 ml volumetric flask, dilute to the mark with water and mix well.

2.5.4.3 被测元素质量分数>0.0050%时，移取10.00mL（2.5.4.1或2.5.4.2）溶液于100mL容量瓶中，用水稀释至刻度，摇匀。

2.5.4.3 When mass fraction of test element is over 0.0050%, 10.00mL of solution (2.5.4.1or 2.5.4.2) is transferred into a 100 ml volumetric flask, dilute to the mark with water and mix well.

2.5.5 系列标准溶液的配制 Preparation of standard series solutions

将混合标准溶液I（2.2.19）、II（2.2.20）和铁标准溶液I（2.2.21）、II（2.2.22）及稀土基体溶液（2.2.18）按表3、表4分别移入100mL容量瓶中，加入10mL硝酸（2.2.5），用水稀释至刻度，混匀，制得各标准溶液，待测。

To each of six 100 ml volumetric flasks, transfer certain quantities of mixed standard solution I (2.2.19), II(2.2.20), iron standard solution I(2.2.21), II (2.2.22) and rare earth matrix solution (2.2.18) as described in table 3 and table 4. Add 10 ml nitric acid (2.2.5), dilute to the mark with water and mix well to prepare the standard series solutions.

表3

|  |  |  |
| --- | --- | --- |
| 标液标号 | 基体浓度/（μg/mL） | 各被测元素浓度/（μg/mL） |
| Fe | Co | Mn | Pb | Ni | Cu | Zn | Al | Cr | Mg | Cd | V |
| 1 | 10000 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 10000 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| 3 | 10000 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |
| 4 | 10000 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| 5 | 10000 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 6 | 10000 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |

Table 3

|  |  |  |
| --- | --- | --- |
| No. | Mass concentration of matrix（μg/mL） | Mass concentration of each test element（μg/mL） |
| Fe | Co | Mn | Pb | Ni | Cu | Zn | Al | Cr | Mg | Cd | V |
| 1 | 10000 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 10000 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| 3 | 10000 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |
| 4 | 10000 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| 5 | 10000 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 6 | 10000 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |

当被测元素质量分数>0.0050%时，使用按表4配制的标准曲线系列。

When mass fraction of test element is over >0.0050%, standard series solutions are prepared as described in table4.

表4

|  |  |  |
| --- | --- | --- |
| 标液标号 | 基体浓度/（μg/mL） | 各被测元素浓度/（μg/mL） |
| Fe | Co | Mn | Pb | Ni | Cu | Zn | Al | Cr | Mg | Cd | V |
| 1 | 1000 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 1000 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 |
| 3 | 1000 | 0.2 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 |
| 4 | 1000 | 0.5 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 |
| 5 | 1000 | 1.0 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 |
| 6 | 1000 | 5.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |

Table 4

|  |  |  |
| --- | --- | --- |
| No. | Mass concentration of matrix（μg/mL） | Mass concentration of each test element（μg/mL） |
| Fe | Co | Mn | Pb | Ni | Cu | Zn | Al | Cr | Mg | Cd | V |
| 1 | 1000 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2 | 1000 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 | 0.05 |
| 3 | 1000 | 0.2 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 | 0.10 |
| 4 | 1000 | 0.5 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 | 0.20 |
| 5 | 1000 | 1.0 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 | 0.50 |
| 6 | 1000 | 5.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |

2.5.6 测定 Determination

推荐分析线见表5。

Recommended analytical lines are listed in table 5.

表5 Table 5

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素Elements | La | Ce | Pr |  Nd | Sm |
| 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm |
| Co | 237.862 | 228.616, 238.892 | 238.892, 228.616 | 237.862, 228.616 | 238.892 |
| Mn | 259.373 | 259.373, 257.610 | 257.610, 259.373 | 293.930, 257.610 | 257.610, 260.569 |
| Pb | 280.200 | 280.200 | 280.200, 283.306 | 280.200, 283.306 | 261.418 |
| Ni | 222.547 | 222.547, 232.504 | 232.504, 222.547 | 232.504 | 232.504, 222.547 |
| Cu | 324.754 | 213.598, 224.700 | 224.700, 213.598 | 224.700, 204.379 | 324.754, 204.379 |
| Zn | 213.856 | 213.856 | 206.200, 213.856 | 213.856 | 213.856 |
| Al | 167.020, 309.271, 396.152 | 167.020, 237.312, 257.510 | 167.020, 226.909 | 167.020, 308.215, 226.909 | 167.020, 237.312, 308.215 |
| Cr | 205.552 | 206.149, 267.716276.259 | 267.716, 206.149 | 205.552, 267.716 | 206.149, 205.552 |
| Mg | 279.553, 280.270 | 280.270 | 279.553, 280.270 | 279.553, 280.270 | 279.553, 280.270 |
| Cd | 226.502, 214.438 | 214.438 | 214.438, 226.502 | 214.438, 228.802 | 214.438, 226.502 |
| V | 311.071, 292.402 | 292.402 | 292.402 | 292.402, 311.071 | 292.402 |
| Fe | 259.940 | 240.488 | 259.940, 240.488 | 259.940, 238.204 | 259.940, 238.204 |

表5（续）Table 5 (Continued)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素Elements | Eu | Gd | Tb | Dy  | Ho  |
| 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm |
| Co | 228.616, 238.892 | 228.616, 238.892 | 237.862 | 238.892, 237.862 | 237.862, 228.616 |
| Mn | 259.373, 260.569 | 259.373, 257.610 | 259.373 | 257.610, 260.569 | 257.610, 260.569 |
| Pb | 220.353, 280.200 | 220.353 | 280.200 | 280.200 | 283.306, 405.783280.200 |
| Ni | 232.504, 222.547 | 222.547, 232.504 | 232.504 | 232.504 | 216.556 |
| Cu | 204.379, 199.969, 327.396 | 324.754, 224.700 | 324.754 | 224.700, 324.754 | 224.700, 213.589 |
| Zn | 206.200, 213.856 | 206.200, 213.856 | 213.856 | 213.856 | 213.856 |
| Al | 167.020, 237.312, 396.152 | 167.020, 237.312, 396.152 | 167.020, 237.312 | 167.020, 237.312, 257.510 | 167.020, 396.152, 237.312 |
| Cr | 205.552 | 206.149, 267.716 | 205.552, 267.716 | 267.716, 206.149 | 206.149, 205.552 |
| Mg | 279.553, 280.270 | 279.553, 280.270 | 279.553 | 279.553, 280.270 | 279.553, 280.270 |
| Cd | 226.502, 214.438 | 226.502, 228.802 | 226.502 | 214.438, 228.802 | 226.502, 228.802 |
| V | 311.071 | 311.071, 292.402 | 292.402 | 292.402 | 311.071, 292.402 |
| Fe | 259.940 | 240.488, 259.940 | 238.204, 259.940 | 259.940 | 259.940, 238.204 |

表5（续）Table 5 (Continued)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素Elements | Er  | Tm  | Yb |  Lu |  Y |
| 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm | 分析线/nmAnalytical lines/nm |
| Co | 237.862, 238.892 | 238.892, 237.862 | 238.892, 237.862 | 237.862, 228.616 | 238.892, 237.862 |
| Mn | 257.610, 260.569 | 259.373, 257.610 | 259.373, 260.569 | 259.373, 257.610 | 259.373, 257.610 |
| Pb | 280.200 | 220.353 | 220.353 | 405.783, 283.306 | 280.200, 283.306 |
| Ni | 232.504, 216.556 | 222.547, 232.504 | 232.504, 222.547 | 232.504, 216.556 | 232.504, 222.547 |
| Cu | 224.700 | 213.598, 204.379 | 324.754, 213.598 | 324.754, 213.598 | 213.598, 224.700 |
| Zn | 206.200 | 206.200, 213.856 | 206.200, 213.856 | 206.200, 213.856 | 206.200, 213.856 |
| Al | 167.020, 237.312, 396.152 | 167.020, 396.152, 226.909 | 167.020, 226.909, 396.152 | 167.020, 237.312, 226.909 | 167.020, 396.152, 237.336 |
| Cr | 267.716, 205.552 | 206.149, 205.552 | 267.716, 205.552 | 206.149, 205.552 | 205.552, 267.716 |
| Mg | 280.270 | 279.553, 280.270 | 279.553, 280.270 | 279.553, 280.270 | 279.553, 280.270 |
| Cd | 226.502, 228.802 | 214.438, 226.502 | 214.438, 226.502 | 228.802, 214.438 | 228.802, 214.438 |
| V | 292.402 | 311.071 | 311.071 | 292.402, 311.071 | 292.402, 311.071 |
| Fe | 238.204, 259.940 | 238.204, 240.488 | 259.940, 238.204 | 259.940, 238.204 | 259.940, 238.204 |

2.6 分析结果的计算 Expression of results

如待测元素为金属，按式（1）计算样品中待测元素的质量分数*w*（%）：

If the test sample is a metal, the content of each element, expressed as a mass fraction w(%), is calculated according to equation (1):

$ω\left(X\right)=\frac{(ρ-ρ\_{0})∙V\_{0}∙V\_{2}×10^{-6}}{m\_{0}∙V\_{1}}×100\%$………………………………（1）

式中：Where

*w(X)*——试料中被测元素的质量分数（%）；The mass fraction (%) of the determinand in test sample;

*p*——计算机输出的分析试液（2.5.4）中被测元素的浓度，单位为微克每毫升（μg/mL）；The mass concentration, in micrograms per milliliter (μg/ml), of the determinand in the analytical test solutions (2.5.4)acquired from standard solution curve;

*p0*——自工作曲线上查得空白溶液中待测元素的质量浓度，单位为微克每毫升（μg/mL）；The mass concentration, in micrograms per milliliter (μg/ml), of the blank test solution acquired from standard solution curve;

*V0*——试液的总体积，单位为毫升（mL）；The gross volume, in milliliters (ml), of the test solution;

*V*1——试液的分取体积，单位为毫升（mL）；The volume, in milliliters (ml), of the section taken from the test solution;

*V2*——试液的测定体积，单位为毫升（mL）；The volume, in milliliters (ml), of the analytical test solution;

*m0*——试料的质量，单位为克（g）；The mass, in grams (g), of the test portion;

如待测元素为氧化物，按式（2）计算样品中待测元素的质量分数*w*（%）：

If the test sample is an oxide, the content of each element, expressed as a mass fraction w (%), is calculated according to equation (2):

$ω\left(X\right)=\frac{k∙(ρ-ρ\_{0})∙V\_{0}∙V\_{2}×10^{-6}}{m\_{0}∙V\_{1}}×100\%$………………………………（2）

式中：where

*w(X)*——试料中被测元素的质量分数（%）；The mass fraction (%) of the determinand in test sample;

*p*——计算机输出的分析试液（2.5.4）中被测元素的浓度，单位为微克每毫升（μg/mL）；The mass concentration, in micrograms per milliliter (μg/ml), of the determinand in the analytical test solutions (2.5.4) outputted from computer;

*p0*——自工作曲线上查得空白溶液中待测元素的质量浓度，单位为微克每毫升（μg/mL）；The mass concentration, in micrograms per milliliter (μg/ml), of the blank test solution acquired from standard solution curve;

*V0*——试液的总体积，单位为毫升（mL）；The gross volume, in milliliters (ml), of the test solution;

*V1*——试液的分取体积，单位为毫升（mL）；The volume, in milliliters (ml), of the section taken from the test solution;

*V2*——试液的测定体积，单位为毫升（mL）；The volume, in milliliters (ml), of the analytical test solution;

*m0*——试料的质量，单位为克（g）；The mass, in grams (g), of the test portion;

*k*——各元素氧化物与其单质的换算系数，见表6。The conversion factor of each oxide to its metal.

表6 Table 6

|  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 元素Elements | Co(Co2O3) | Mn(MnO2) | Pb(PbO) | Ni(NiO) | Cu(CuO) | Zn(ZnO) | Al(Al2O3) | Cr(Cr2O3) | Mg(MgO) | Cd(CdO) | V(V2O5) | Fe(Fe2O3) |
| k | 1.4072 | 1.5825 | 1.0772 | 1.2726 | 1.2518 | 1.2447 | 1.8895 | 1.4616 | 1.6583 | 1.1423 | 1.7852 | 1.4296 |

2.7 精密度 Precision

2.7.1 重复性 Repeatability

在重复性条件下获得的两次独立测试结果的测定值，在以下给出的平均值的范围内，这两个测试结果的绝对差值不超过重复性限（r），超过重复性限（r）的情况下不超过5%，重复性限（r）按表7数据采用线性内插法求得。

The absolute difference between the determined values of the two independent determination results obtained under the repeatability conditions is not greater than the repeatability limits (r) in the range of the following average values, under 95% confidence probability. The repeatability limit (r) is calculated by linear interpolation method on the basis of the data listed in table 7.

表7

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素Elements | 质量分数Mass fraction/% | 重复性限Repeatability limit（r）/% | 元素Elements | 质量分数Mass fraction/% | 重复性限Repeatability limit（r）/% |
| 氧化钴Cobalt oxide | 0.0009 | 0.0001 | 氧化铝Aluminum oxide | 0.0008 | 0.0002 |
| 0.0045 | 0.0003 | 0.0041 | 0.0007 |
| 0.0089 | 0.0006 | 0.0084 | 0.0011 |
| 0.054 | 0.007 | 0.053 | 0.005 |
| 0.080 | 0.008 | 0.081 | 0.007 |
| 氧化锰Manganese oxide | 0.0011 | 0.0003 | 氧化铬Chromium oxide | 0.0006 | 0.0003 |
| 0.0057 | 0.0007 | 0.0035 | 0.0003 |
| 0.0114 | 0.0019 | 0.0068 | 0.0011 |
| 0.055 | 0.004 | 0.056 | 0.008 |
| 0.098 | 0.007 | 0.085 | 0.014 |
| 氧化铅Lead oxide | 0.0011 | 0.0004 | 氧化镁Magnesium oxide | 0.0010 | 0.0004 |
| 0.0046 | 0.0007 | 0.0045 | 0.0005 |
| 0.0094 | 0.0008 | 0.0088 | 0.0009 |
| 0.044 | 0.012 | 0.043 | 0.005 |
| 0.080 | 0.015 | 0.074 | 0.006 |
| 氧化镍Nickel oxide | 0.0009 | 0.0001 | 氧化镉Cadmium oxide | 0.0005 | 0.0001 |
| 0.0049 | 0.0004 | 0.0025 | 0.0002 |
| 0.0098 | 0.0009 | 0.0050 | 0.0005 |
| 0.050 | 0.007 | 0.024 | 0.003 |
| 0.082 | 0.008 | 0.063 | 0.005 |
| 氧化铜Copper oxide | 0.0009 | 0.0002 | 氧化钒Vanadium oxide | 0.0008 | 0.0001 |
| 0.0048 | 0.0008 | 0.0044 | 0.0003 |
| 0.0099 | 0.0017 | 0.0091 | 0.0006 |
| 0.048 | 0.006 | 0.042 | 0.006 |
| 0.083 | 0.009 | 0.078 | 0.007 |
| 氧化锌Zinc oxide | 0.0008 | 0.0004 | 氧化铁Iron oxide | 0.0011 | 0.0003 |
| 0.0047 | 0.0007 | 0.0056 | 0.0009 |
| 0.0096 | 0.0010 | 0.011 | 0.002 |
| 0.049 | 0.005 | 0.093 | 0.005 |
| 0.079 | 0.007 | 0.27 | 0.02 |
|  |  | 0.33 | 0.03 |

2.7.2 允许差 Tolerance

实验室之间分析结果的差值应不大于表8所列的允许差。

The difference of the analytical results among laboratories should not be greater than the tolerances listed in table 8.

表8 Table 8

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| 元素Elements | 氧化物质量分数 Mass fraction of oxides /% | 允许差 Tolerance /% | 元素Elements | 氧化物质量分数 Mass fraction of oxides /% | 允许差 Tolerance /% |
| 氧化钴Cobalt oxide | 0.0010~0.0050 | 0.0008 | 氧化铝Aluminum oxide | 0.0010~0.0050 | 0.0008 |
| >0.0050~0.010 | 0.002 | >0.0050~0.010 | 0.002 |
| >0.010~0.050 | 0.007 | >0.010~0.050 | 0.007 |
| >0.050~0.10 | 0.009 | >0.050~0.10 | 0.009 |
| 氧化锰Manganese oxide | 0.0010~0.0050 | 0.0008 | 氧化铬Chromium oxide | 0.0010~0.0050 | 0.0008 |
| >0.0050~0.010 | 0.002 | >0.0050~0.020 | 0.002 |
| >0.010~0.050 | 0.007 | >0.020~0.050 | 0.005 |
| >0.050~0.10 | 0.009 | >0.050~0.10 | 0.015 |
| 氧化铁Iron oxide | 0.0010~0.0050 | 0.0008 | 氧化镁Magnesium oxide | 0.0002~0.0010 | 0.0002 |
| >0.0050~0.010 | 0.002 | >0.0010~0.0050 | 0.0008 |
| >0.010~0.050 | 0.007 | >0.0050~0.010 | 0.002 |
| >0.050~0.10 | 0.01 | >0.010~0.050 | 0.007 |
| >0.10~0.30 | 0.03 | >0.050~0.10 | 0.009 |
| >0.30~0.50 | 0.04 |  |  |
| 氧化镍Nickel oxide | 0.0010~0.0050 | 0.0008 | 氧化镉Cadmium oxide | 0.0010~0.0050 | 0.0008 |
| >0.0050~0.010 | 0.002 | >0.0050~0.010 | 0.002 |
| >0.010~0.050 | 0.007 | >0.010~0.050 | 0.007 |
| >0.050~0.10 | 0.009 | >0.050~0.10 | 0.009 |
| 氧化铜Copper oxide | 0.0010~0.0050 | 0.0008 | 氧化钒Vanadium oxide | 0.0010~0.0050 | 0.0008 |
| >0.0050~0.010 | 0.002 | >0.0050~0.010 | 0.002 |
| >0.010~0.050 | 0.007 | >0.010~0.050 | 0.007 |
| >0.050~0.10 | 0.009 | >0.050~0.10 | 0.009 |
| 氧化锌Copper oxide | 0.0010~0.0050 | 0.0008 | 氧化铅Lead oxide | 0.0010~0.0050 | 0.0008 |
| >0.0050~0.010 | 0.002 | >0.0050~0.010 | 0.002 |
| >0.010~0.050 | 0.007 | >0.010~0.040 | 0.007 |
| >0.050~0.10 | 0.009 | >0.040~0.10 | 0.016 |

**3 方法2：电感耦合等离子体质谱法**

**3 Method 2: Inductively Coupled Plasma Mass Spectrometric Method**

3.1 方法原理Principle

试样以硝酸溶解，在稀硝酸介质中，直接以氩等离子体光源激发，进行质谱测定。采用内标法校正基体效应。

The test portionis dissolved withnitric acid, and then directly excited by the light source of argon inductively coupled plasma. Carry themass spectrometric determination in the medium of dilute nitric acid. The internal standard method is used to correct matrix effect on the determination.

3.2 试剂和材料Reagents

3.2.1过氧化氢（30%），MOS级纯

Hydrogen peroxide（30%），with purity of MOS-level

3.2.2硝酸（ρ 1.42 g/mL），MOS级纯

Nitric acid（ρ 1.42 g/mL），with purity of MOS-level

3.2.3硝酸（1+1）

Nitric acid，diluted1+1

3.2.4 钴标准贮存溶液：准确称取0.2000g金属钴[*w*（Co）≥99.9％]于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，冷却至室温，溶液移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg钴。

Cobalt standard stock solution(1 mL = 1 mgCo) : Weight out 0.2000 g of cobalt metal[*w*（Co）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature.After cooling off to room temperature, transfer the cobalt solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.5 锰标准贮存溶液：准确称取0.2000g金属锰[*w*（Mn）≥99.9％]，[预先用稀硫酸洗涤除去表面上的氧化物，再用水洗涤除酸，烘干冷却后使用] 于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，冷却至室温，溶液移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg锰。

Manganese standard stock solution (1 mL = 1 mgMn):Weight out 0.2000 g of manganese metal[*w*（Mn）≥99.9％],[ wash with dilute sulfuric acidto remove the oxide on the surface, and then wash with water and dry before use ] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature. After cooling off to room temperature, transfer the manganese solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.6铅标准贮存溶液：准确称取0.2000g金属铅[*w*（Pb）≥99.9％]于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，待完全溶解后，加热除去二氧化氮，取下冷却，移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg铅。

Lead standard stock solution (1 mL = 1 mgPb) : Weight out 0.2000 g of lead metal[*w*（Pb）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature. After complete dissolution heat the lead solution to remove nitrogen dioxide. After cooling offto room temperature, transfer the lead solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.7镍标准贮存溶液：准确称取0.2000g金属镍[*w*（Ni）≥99.9％] 于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，冷却至室温，溶液移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg镍。

Nickel standard stock solution (1 mL = 1 mgNi) : Weight out 0.2000 g of nickel metal[*w*（Ni）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature. After cooling offto room temperature, transfer the nickel solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.8铜标准贮存溶液：准确称取0.2000g金属铜[*w*（Cu）≥99.9％] 于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，冷却至室温，溶液移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg铜。

Copper standard stock solution (1 mL = 1 mgCu) : Weight out 0.2000 g of copper metal[*w*（Cu）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature. After cooling offto room temperature, transfer the copper solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.9锌标准贮存溶液：准确称取0.2000 g 金属锌粒[*w*（Zn）≥99.9％]，置于300 mL烧杯，加20 mL盐酸，低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg锌。

Zinc standard stock solution (1 mL = 1 mgZn) : Weight out 0.2000 g of zinc metal[*w*（Zn）≥99.9％] into a 300-mL beaker,add 10 mL of water and 20 mL of hydrochloric acid,heat gently at moderate temperature. After cooling offto room temperature, transfer the zinc solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.10铝标准贮存溶液：准确称取0.2000 g金属铝箔[*w*（Al）≥99.9％],[预先用稀盐酸浸泡，经无水乙醇清洗，用红外灯烘干]于200 mL烧杯中，加10mL水，加20 mL盐酸，滴加2 mL硝酸（2.2.5），低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1 mg铝。

Aluminum standard stock solution (1 mL = 1 mgAl) :Weight out 0.2000 g of aluminum metal[w（Al）≥99.9％], [soak in dilute hydrochloric acid, wash with anhydrous ethanol, and dry under infrared lamp before use] into a 200-mL beaker,add 10 mL of water and 20 mL of hydrochloric acidwith drop wise 2 mLof nitric acid（3.2.3）, heat gently at moderate temperature. After cooling offto room temperature, transfer the aluminum solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.11铬标准贮存溶液：准确称取0.2000 g金属铬[*w*（Cr）≥99.9％] 于200 mL烧杯，加10mL水，加20 mL 盐酸，低温溶解，冷却至室温，溶液移入200 mL容量瓶中，用水稀释至刻度，摇匀。此溶液1 mL含1mg铬。

Chromium standard stock solution (1 mL = 1 mgCr) : Weight out 0.2000 g of chromium metal[w（Cr）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of hydrochloric acid,heat gently at moderate temperature. After cooling offto room temperature, transfer the chromium solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.12镁标准贮存溶液: 称取0.3317 g经800 ℃灼烧的氧化镁[*w*（MgO）≥99.9％]于200 mL烧杯中，加10 mL盐酸，加热至溶解完全，冷却至室温。移入200 mL容量瓶中， 用水稀释至刻度，摇匀。 此溶液1 mL含1 mg镁。

Magnesium standard stock solution (1 mL = 1 mgMg):Weight out0.3317 gofmagnesium oxide ([*w*（MgO）≥99.9％], previously ignited at 800℃) into a 200-mL beaker,add 10 mL of hydrochloric acid,heat gently at moderate temperature. After cooling offto room temperature, transfer the magnesium solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.13镉标准贮存溶液：称取0.2000g金属镉[*w*（Cd）≥99.9％] 于200mL烧杯中，加10mL水，加20mL硝酸（3.2.3），低温溶解，冷却至室温，溶液移入200mL容量瓶中，以水稀释至刻度，混匀。此溶液1 mL含1mg镉。

Cadmium standard stock solution (1 mL = 1 mgCd) : Weight out 0.2000 g of cadmium metal[*w*（Cd）≥99.9％] into a 200-mL beaker,add 10 mL of water and 20 mL of nitric acid（3.2.3）, heat gently at moderate temperature. After cooling offto room temperature, transfer the cadmium solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.14钒标准贮存溶液：称取0.4592 g偏钒酸铵（优级纯）[w（NH4VO3）≥99.9％] 于200 mL烧杯，溶于适量水中，用硝酸（3.2.2）中和至酸性，溶液移入200 mL容量瓶中，用水稀释至刻度，混匀。此溶液1 mL含1 mg钒。

Vanadium standard stock solution (1 mL = 1 mg V) : Weight out 0.4592 g of ammonium metavanadate(GR) [w（NH4VO3）≥99.9％] into a 200-mL beaker, dissolve in amount of water and neutralizewith nitric acid（3.2.2）to PH-value smaller than 7. Transfer the vanadium solution to a 200-mL volumetric flask, dilute to the mark with water and mix well.

3.2.15混合标准贮存溶液(I)：分别移取标准贮存溶液（3.2.4~3.2.14）各5.00mL于100mL容量瓶中，加入10mL硝酸（3.2.2）以水稀释至刻度，混匀。此溶液1mL含50μg各元素。

Mixed standard solution (I) (1 mL = 50 μg various elements):Respectively transfer 5.00 mL of standard stock solution (3.2.4~3.2.14) into a 100-mL volumetric flaskwith calibrated pipettes.Add 10 mL of nitric acid（3.2.3）, dilute to the mark with water and mix well.

3.2.16混合标准贮存溶液(II)：分取5.00mL混合标准贮存溶液(I) ( 3.2.15) 至250mL容量瓶中，加入25mL硝酸（3.2.3），定容。此溶液1mL含有1μg各元素。

Mixed standard solution (II) (1 mL = 1 μg various elements): Transfer 5.00 mL mixed standardsolution (I) ( 3.2.15) into a 250-mL volumetric flaskwith a calibrated pipette, add 25 mL of nitric acid（3.2.3）, dilute to the mark with water and mix well.

3.2.17铯内标贮存溶液(I)：准确称取1.26675g氯化铯（优级纯，在110℃烘干过），溶于少量水后，将溶液移入1000mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg铯。

Cesium internal standard stock solution (I) (1 mL = 1 mg Cs): Weight out 1.26675 g ofcesium chloride (GR) (dried at 110℃ ), dissolve in amount of water, and transfer to a 1000-mL volumetric flask, dilute to the mark with water and mix well.

3.2.18铯内标贮存溶液(II)：分取5.00mL铯内标贮存溶液(I)( 3.2.17)至250mL容量瓶中，加入25mL硝酸（3.2.3），定容。此溶液1mL含有20μg铯。

Cesium internal standard stock solution (II) (1 mL = 20 μg Cs): Transfer 5.00 mL cesium standard stock solution (I) ( 3.2.17) into a 250-mL volumetric flaskwith a calibrated pipette, add 25 mL of Nitric acid（3.2.3）dilute to the mark with water and mix well.

3.2.19铯内标溶液：分取5.00mL铯内标贮存溶液(II)( 3.2.18) 至100mL容量瓶中，加入5mL硝酸（3.2.3），定容。此溶液1mL含1μg铯的内标溶液。

Cesium internal standard solution (1 mL = 1 μg Cs): Transfer 5.00 mL cesium standard stock solution (II) ( 3.2.18) into a 100-mL volumetric flaskwith a calibrated pipette, add 5 mL of nitric acid（3.2.3）dilute to the mark with water and mix well.

3.2.20铟内标贮存溶液（I）：准确称取1.0000g金属铟[*w*（In）≥99.9％]于300mL烧杯中，加入20mL盐酸（1+1），置于水浴上加热，至样品完全溶解。将溶液移入1000mL容量瓶中，以水稀释至刻度，混匀。此溶液1mL含有1mg铟。

Indium internal standardstock solution (I) (1 mL = 1 mg In): Weight out 1.0000 g of indium metal[*w*（In）≥99.9％] into a 300-mL beaker, add20mL of hydrochloric acid diluted1+1 ,heat on a water bath. After complete dissolution transfer the indium solution to a 1000-mL volumetric flask, dilute to the mark with water and mix well.

3.2.21铟内标贮存溶液(II)：分取5.00mL铟内标贮存溶液(I)（3.2.20）至250mL容量瓶中，加入25mL硝酸（3.2.3），定容。此溶液1mL含有20μg铟。

Indium internal standard stock solution (II) (1 mL = 20 μg In): Transfer 5.00 mLindium standard stock solution (I) ( 3.2.20) into a 250-mL volumetric flaskwith a calibrated pipette, add 25 mL of nitric acid（3.2.3）, dilute to the mark with water and mix well.

3.2.22铟内标溶液：分取5.00mL铟内标贮存溶液(II)（3.2.21）至100mL容量瓶中，加入5mL硝酸（3.2.3），定容。此溶液1mL含有1μg铟。

Indium internal standard solution (1 mL = 1 μg In): Transfer 5.00 mLindium standard stock solution (II) ( 3.2.21) into a 100-mL volumetric flaskwith a calibrated pipette, add 5 mL of nitric acid（3.2.3）, dilute to the mark with water and mix well.

3.2.23氩气[φ（Ar）≥99.99％]。

Argon gas [φ（Ar）≥99.99％]。

3.3 仪器Apparatus

电感耦合等离子体质谱仪：质量分辨率不低于0.8±0.1（amu）。

Inductively coupled plasma mass spectrometer: Mass resolution is not less than 0.8±0.1（amu）.

3.4 试样Sample

3.4.1氧化物试样于105℃烘1h，置于干燥器中，冷却至室温，立即称量。

For rare earth oxide, dry at 105℃for 1 hour, keep in desiccator, Cool to room temperature and weighimmediately.

3.4.2金属试样去掉氧化层，取样后，立即称量。

For rare earth metal,weigh immediately after remove the oxide layer.

3.5 分析步骤Procedure

3.5.1 试料Test portion

称取0.25g试样（3.4），精确至0.0001g。

Weigh 0.25g ofsample (3.4)to the nearest 0.0001g.

3.5.2测定次数Parallel determination

称取两份试料进行平行测定，取其平均值。

Carry out the parallel determination for two test portions. Average the two results.

3.5.3空白试验Blank Test

随试料做空白试验。

Carry out a blank test at the same timewith the test portion

3.5.4 分析试液的制备Preparation of the test solution

3.5.4.1将除二氧化铈外的试料（3.5.1）置于100mL聚四氟乙烯烧杯中，加入5mL硝酸（3.2.3），低温加热至溶解完全，冷却至室温，移入100mL容量瓶，定容。按照表9进行分取、内标加入及定容操作。

Place the test portion（3.5.1）(Except cerium dioxide) into a 100-mL PTFE beaker, add 5mL of nitric acid（3.2.3）, heat gently at moderate temperature,dissolvecompletely.Cool to room temperature, transfer into a 100-mL volumetric flask, dilute to the mark with water and mix well. Transfer upper solutionwith a calibrated pipette, add internal standard and nitric acid according to table 9. Dilute to the mark with water and mix well.

3.5.4.2将二氧化铈试料（3.5.1）置于100mL聚四氟乙烯烧杯中，加入5mL硝酸（3.2.3）和1.5mL过氧化氢（3.2.1），低温加热至溶解完全并赶尽气泡，冷却至室温。移入100mL容量瓶，定容。按照表9进行分取、内标加入及定容操作。

Place thecerium dioxidetest portion（3.5.1）into a 100-mL PTFE beaker, add 5mL of nitric acid（3.2.3）and 1.5mL of hydrogen peroxide（3.2.1）, heat gently at moderate temperature,dissolvecompletely. After removing of all bubbles, cool to room temperature, transfer into a 100-mL volumetric flask, dilute to the mark with water and mix well. Transfer upper solutionwith a calibrated pipette, add internal standard and nitric acid according to table 9. Dilute to the mark with water and mix well.

表 9 Table 9

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Determination range/% | Volume of transfered solution (3.5.4.1,3.5.4.2）/mL | Volume of test solution/mL | Volume ofinternal standardsolution（3.2.19 or 3.2.21）/mL | Volume ofsupplemented Nitric acid(3.2.3) /mL |
| 0.0001~0.010 | 10.00 | 50.00 | 0.50 | 2 |
| >0.010~0.050 | 5.00 | 50.00 | 0.50 | 2 |

3.5.5 标准系列溶液的配制Preparation of standard series solutions

移取适量混合标准溶液（3.2.16）于7个100mL容量瓶中，加入1.00mL内标溶液（3.2.19, 3.2.21），加入4mL硝酸溶液（3.2.3），以水稀释至刻度，混匀，待测。此标样系列溶液浓度见表10。

According to table 10, transfer amount ofmixed standard solution (II)（3.2.16）respectivelyto 7 100-mL volumetric flasks, add 1.00mL of internal standardsolution（3.2.19, 3.2.22） and 4mL of nitric acid（3.2.3）,dilute to volume with water, and mix well. The concentrations of the standard series solutions areshown in table 10.

表 10 Table 10

|  |  |
| --- | --- |
| Solution Nr. | 各被测元素质量浓度， ng/mLConcentrations of thetargetelements**,** ng/mL |
| Cobalt | Manganese | Lead | Nickel | Copper | Zinc | Aluminum | Chromium | Magnesium | Cadmium | Vanadium |
| 1234567 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 | 02.005.0010.0020.0050.00150.00 |

3.5.6 测定Determination

3.5.6.1推荐测量同位素见表11。The recommended isotopesareshown in table 11.

 **表 11** Table 11

|  |  |  |  |
| --- | --- | --- | --- |
| Element | isotope | Element | isotope |
| CoMnPbNiCuZnAl | 595520860636427 | CrMgCdVCs In | 522411451133115 |

3.5.6.2将分析试液（3.5.4）与标准系列溶液（3.5.5）同时进行等离子体质谱测定。

Determinethe test solution（3.5.4）together with the standard series solutions (3.5.5) by the plasma mass spectrometry.

3.6 分析结果的计算**Expression of results**

如待测元素为金属，按式（3）计算样品中待测元素的质量分数*w*（%）：

The content of rare earthmetals, expressed as percentage by mass, is calculated by formula (3):

$w(X)=\frac{(ρ-ρ\_{0})∙V\_{2}∙V\_{0}×10^{-9}}{m∙V\_{1}}×100$………………………………（3）

式中：

*ρ——*自工作曲线上查得分析试液(3.5.4)中待测元素的质量浓度，单位为纳克每毫升（ng/mL）；

*ρ0——*自工作曲线上查得空白溶液中待测元素的质量浓度，单位为纳克每毫升（ng/mL）；

*V0——*第一次定容体积，单位为毫升（mL）；

*V1——*分取试液体积，单位为毫升（mL）；

*V2——*第二次定容体积，单位为毫升（mL）；

*m——*试料的质量，单位为克（g）。

Where:

*ρ*is the concentration, in nanogramper milliliter , of the target element, acquired from the calibration curve ;

*ρ0*is the concentration, in nanogramper milliliter, of the blank test, acquired from the calibration curve;

*V0*is the volume, in milliliter,of the grosssolution;

*V1*is the volume, in milliliter, of the transfered solution;

*V2*is the volume, in milliliter,of the test solution;

*m——*is the mass, in grams, of the test portion.

如待测元素为氧化物，按式（4）计算样品中待测元素的质量分数*w*（%）：

The contentof rare earth oxides, expressed as percentage by mass, is calculated by formula (4):

$w(X)=\frac{k∙(ρ-ρ\_{0})∙V\_{2}∙V\_{0}×10^{-9}}{m∙V\_{1}}×100$…………………………………(4)

式中：

*ρ——*自工作曲线上查得分析试液(3.5.4)中待测元素的质量浓度，单位为纳克每毫升（ng/mL）；

*ρ0——*自工作曲线上查得空白溶液中待测元素的质量浓度，单位为纳克每毫升（ng/mL）；

*V0——*第一次定容体积，单位为毫升（mL）；

*V1——*分取试液体积，单位为毫升（mL）；

*V2——*第二次定容体积，单位为毫升（mL）；

*m——*试料的质量，单位为克（g）。

*k* *——*各元素氧化物与其单质的换算系数，见表12。

*ρ*is the concentration, in nanogramper milliliter, of the target element, acquired from the calibration curve ;

*ρ0*is the concentration, in nanogramper milliliter, of the blank test, acquired from the calibration curve;

*V0*is the volume, in milliliter, of the grosssolution;

*V1*is the volume, in milliliter, of the transfered solution;

*V2*is the volume, in milliliter, of the test solution;

*m*is the mass, in grams, of the test portion.

*k ——*is theconversion factor of each metal to its oxide, shown in table 12.

表 12 Table 12

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| element | Co(Co2O3) | Mn(MnO2) | Pb(PbO) | Ni(NiO) | Cu(CuO) | Zn(ZnO) | Al(Al2O3) | Cr(Cr2O3) | Mg(MgO) | Cd(CdO) | V(V2O5) |
| *k* | 1.4072 | 1.5825 | 1.0772 | 1.2726 | 1.2518 | 1.2447 | 1.8895 | 1.4616 | 1.6583 | 1.1423 | 1.7852 |

3.7 精密度Precision

3.7.1 重复性Repeatability

在重复性条件下获得的两次独立测试结果的测定值，在以下给出的平均值范围内，这两个测试结果的绝对差值不超过重复性限（*r*），超过重复性限（*r*）的情况不超过5%，重复性限（*r*）按表13数据采用线性内插法求得；超过表13中含量的测定值，其重复性限（*r*）用外推法计算求得。

Two independent determinations are carried out under the repeatability conditions. In the range of the following average values in Table 13, the absolute difference of the two determined values shall not be greater than the repeatability limit (*r*) under 95% confidence probability.

The repeatability limit (*r*) should be calculated by linear interpolation method in accordance with the data listed in Table 13.

表 13 Table 13

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Element | Mass fraction/% | Repeatabilitylimit（*r*）/% | Element | Mass fraction/% | Repeatabilitylimit（*r*）/% |
| Cobalt oxide | 0.0004 | 0.0001 | Aluminum oxide | 0.0004 | 0.0004 |
| 0.0028 | 0.0002 | 0.0045 | 0.0006 |
| 0.0085 | 0.0009 | 0.015 | 0.001 |
| 0.019 | 0.001 | - | - |
| 0.035 | 0.002 | - | - |
| Manganese oxide | 0.0004 | 0.0001 | Chromic oxide | 0.0004 | 0.0001 |
| 0.0028 | 0.0002 | 0.0006 | 0.0002 |
| 0.0085 | 0.0009 | 0.0071 | 0.0006 |
| 0.019 | 0.001 | 0.019 | 0.002 |
| 0.036 | 0.002 | 0.035 | 0.003 |
| Lead oxide | 0.0005 | 0.0001 | Magnesium oxide | 0.0005 | 0.0001 |
| 0.0023 | 0.0002 | 0.0025 | 0.0005 |
| 0.010 | 0.001 | 0.0064 | 0.0006 |
| 0.014 | 0.001 | 0.010 | 0.001 |
| 0.033 | 0.002 | 0.032 | 0.003 |

表 13（续）Table 13（Continue）

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Element | Mass fraction/% | Repeatability limit（*r*）/% | Element | Mass fraction/% | Repeatability limit（*r*）/% |
| Nickel oxide | 0.0003 | 0.0002 | Cadmium oxide | 0.0004 | 0.0002 |
| 0.0026 | 0.0003 | 0.0016 | 0.0003 |
| 0.0080 | 0.0006 | 0.0044 | 0.0006 |
| 0.019 | 0.001 | 0.011 | 0.001 |
| 0.035 | 0.002 | 0.061 | 0.003 |
| Copper oxide | 0.0004 | 0.0002 | Vanadium oxide | 0.0008 | 0.0001 |
| 0.0026 | 0.0003 | 0.0016 | 0.0002 |
| 0.0052 | 0.0005 | 0.0077 | 0.0003 |
| 0.0095 | 0.0009 | 0.015 | 0.001 |
| 0.041 | 0.002 | - | - |
| Zinc oxide | 0.0018 | 0.0003 |  |  |  |
| 0.0045 | 0.0005 |  |  |
| 0.0065 | 0.0006 |  |  |
| 0.016 | 0.001 |  |  |
| 0.034 | 0.002 |  |  |
| 注：重复性限（*r*）为2.8×S*r*，S*r*为重复性标准差。Note:*r=*2.8×S*r,* andS*r* is the standard devition. |

3.7.2 允许差Tolerance

实验室之间分析结果的差值应不大于表14所列允许差。

The difference of the analytical results among laboratories should not be greater than the tolerances listed in Table 14.

表 14 Table 14

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Element | Determination range/% | Tolerance /% | Element | Determination range/% | Tolerance /% |
| Cobalt oxide | 0.0001～0.0003 | 0.0001 | Alumina | 0.0003～0.0010 | 0.0004 |
| >0.0003~0.0010 | 0.0002 | >0.0010~0.0030 | 0.0008 |
| >0.0010~0.0030 | 0.0003 | >0.0030~0.010 | 0.001 |
| >0.0030~0.010 | 0.0009 | >0.010~0.025 | 0.002 |
| >0.010~0.025 | 0.002 | >0.025~0.050 | 0.003 |
| >0.025~0.050 | 0.003 | - | - |
| Manganese oxide | 0.0001～0.0003 | 0.0001 | Chromic oxide | 0.0001～0.0003 | 0.0001 |
| >0.0003~0.0010 | 0.0002 | >0.0003~0.0010 | 0.0002 |
| >0.0010~0.0030 | 0.0003 | >0.0010~0.0030 | 0.0003 |
| >0.0030~0.010 | 0.0009 | >0.0030~0.010 | 0.0006 |
| >0.010~0.025 | 0.002 | >0.010~0.025 | 0.002 |
| >0.025~0.050 | 0.003 | >0.025~0.050 | 0.003 |
| Lead oxide | 0.0001～0.0003 | 0.0001 | Magnesium oxide | 0.0001～0.0003 | 0.0001 |
| >0.0003~0.0010 | 0.0002 | >0.0003~0.0010 | 0.0002 |
| >0.0010~0.0030 | 0.0003 | >0.0010~0.0030 | 0.0008 |
| >0.0030~0.010 | 0.001 | >0.0030~0.010 | 0.001 |
| >0.010~0.025 | 0.002 | >0.010~0.025 | 0.002 |
| >0.025~0.050 | 0.003 | >0.025~0.050 | 0.003 |

表 14（续）Table 14（Continue）

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Element | Determination range/% | Tolerance /% | Element | Determination range/% | Tolerance /% |
| Nickel oxide | 0.0001～0.0003 | 0.0001 | Cadmium oxide | 0.0001～0.0003 | 0.0001 |
| >0.0003~0.0010 | 0.0002 | >0.0003~0.0010 | 0.0002 |
| >0.0010~0.0030 | 0.0003 | >0.0010~0.0030 | 0.0003 |
| >0.0030~0.010 | 0.0006 | >0.0030~0.010 | 0.0006 |
| >0.010~0.025 | 0.002 | >0.010~0.025 | 0.002 |
| >0.025~0.050 | 0.003 | >0.025~0.050 | 0.004 |
| Copper oxide | 0.0001～0.0003 | 0.0001 | Vanadium oxide | 0.0001～0.0003 | 0.0001 |
| >0.0003~0.0010 | 0.0002 | >0.0003~0.0010 | 0.0002 |
| >0.0010~0.0030 | 0.0003 | >0.0010~0.0030 | 0.0003 |
| >0.0030~0.010 | 0.0008 | >0.0030~0.010 | 0.0009 |
| >0.010~0.025 | 0.002 | >0.010~0.025 | 0.002 |
| >0.025~0.050 | 0.003 | >0.025~0.050 | 0.003 |
| Zinc oxide | 0.0003～0.0010 | 0.0003 |  |  |  |
| >0.0010~0.0030 | 0.0004 |  |  |
| >0.0030~0.010 | 0.001 |  |  |
| >0.010~0.025 | 0.002 |  |  |
| >0.025~0.050 | 0.003 |  |  |
| - | - |  |  |

3.8 质量保证和控制Quality control

每周用自制的控制标样（如有国家级或行业级标样时，应首先使用）校核一次本标准分析方法的有效性。当过程失控时，应找出原因，纠正错误，重新进行校核。

Check the validity of this standard method with reference material each week. National or industrial grade reference materials shall be used prior to the self-made controlling sample. Find cause and correct in case that the process is out of control. Then check again.