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YS

**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 568.1-2008

Replacing YS/T 568.1-2006

**Chemical analysis methods for zirconium oxide and hafnium
oxide. Determination of zirconium oxide and hafnium total
content. Mandelic acid gravimetric method**

氧化锆、氧化铪化学分析方法 氧化锆和氧化铪含量的测定 苦杏仁酸重量法

(English Translation)

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Foreword

YS/T 568 "zirconium, hafnium oxide chemical analysis method" is divided into 11 parts.

- Part 1. Determination of zirconium and hafnium oxide combined amount of mandelic acid gravimetric method;
- Part 2. Determination of iron content sulfonic acid spectrophotometry;
- Part 3. Determination of silicon content - Molybdenum blue spectrophotometric method;
- Part 4. Determination of aluminum content of chromium azure S- cypyrindinium chloride spectrophotometry;
- Part 5. Determination of sodium content by flame atomic absorption spectrometry;
- Part 6. Determination of the amount of titanium di Zhao DAM spectrophotometry;
- Part 7. Determination of phosphorus content of antimony salt - ascorbic acid - phosphorus molybdenum blue spectrophotometric method;
- Part 8. zirconia aluminum, calcium, magnesium, manganese, sodium, nickel, iron, titanium, zinc, molybdenum, vanadium, hafnium content - Inductively coupled plasma fat Emission spectrometry;
- Part 9. hafnium oxide of aluminum, calcium, magnesium, manganese, sodium, nickel, iron, titanium, zinc, molybdenum, vanadium, zirconium content - Inductively coupled plasma fat Emission spectrometry;
- Part 10. Determination of manganese content - Potassium periodate spectrophotometric method;
- Part 11. Determination of alpha- linked furoylhydrazone nickel dioxime

spectrophotometric method.

This is Part 1.

The partial replacement of YS/T 568.1-2006 "zirconium oxide, hafnium oxide, zirconium oxide and hafnium combined amount of determination" (formerly GB/T 2590.1-1981).

This portion of YS/T 568.1-2006 compared to the main changes are as follows.

--- Added "7.1 repeatability" clause;

--- Added "8 Quality assurance and control" clause.

The non-ferrous metal part by the National Standardization Technical Committee and centralized.

This part by the Beijing Nonferrous Metal Research Institute, China Nonferrous Metals Industry Institute of Standards Metrology and Quality is responsible for drafting.

1 Scope

This section provides zirconium oxide and hafnium oxide combined determination of the amount of zirconia and hafnium.

This section applies to the determination of zirconium oxide and zirconium oxide and hafnium oxide combined amount of hafnium. Measuring range. 75% to 99%.

2 Method summary

In 2.4mol/L ~ 3mol hot hydrochloric acid/L was added so as to generate an acid bitter almond bitter almond insoluble zirconium, hafnium white flocculent Precipitation, aging into a crystalline precipitate. Calcined at 900 °C precipitate, weighing as oxides.

3 Reagents

3.1

sulfate (rho1.84g/mL), pure class distinctions.

3.2

hydrofluoric acid (rho1.15g/mL), pure class distinctions.

3.3

hydrochloric acid (rho1.19g/mL), pure class distinctions.

3.4

Ammonia (rho0.90g/mL).

3.5

phenolphthalein solution (10g/L).

3.6

mandelic acid solution (160g/L), filtered use.

3.7

washing solution. 25g mandelic acid was dissolved in 500mL of water, and add 10mL hydrochloric acid (3.3).

4 Instrument

Box-type high-temperature furnace and high pressure sample dissolution apparatus.

5.1 Sample

Weigh the sample 0.20g, accurate to 0.0001g.

5.2 Determination of the number of Independent of the two measurements, the mean value.

5.3 Determination

5.3.1

The sample (5.1) into a high pressure sample dissolution apparatus Teflon crucible added 3mL of hydrofluoric acid (3.2), cover the crucible lid, spin Tight high-pressure units, at 140 °C insulation 50min, until the sample is completely dissolved (eg sample soluble in hydrofluoric acid, can do high pressure sample dissolution means for Platinum dish can). After cooling, the crucible of the sample solution was transferred to 100mL water platinum dish, add 2mL of sulfuric acid (3.1), added to the furnace Thermal evaporation to take strong white smoke, remove the cooling water purge platinum dish wall heating was continued to take to make white smoke, remove the cooling.

5.3.2

added 10mL of hydrochloric acid (3.3) in the platinum dish, and heated to a platinum dish, dissolved after drying, transferred to 250mL beaker with water and dilute Diluted to 100mL. Add 3 drops of phenolphthalein solution (3.5), was added dropwise aqueous ammonia (3.4) until the solution turns red, the number of excess drops, heated to boiling, removed.

5.3.3

hot medium speed quantitative filter paper, and washed in hot water with ammonia micro beaker of pH8. The precipitate was washed 7 times again. The precipitate together Filter

paper back into the original beaker, was added 25mL of hydrochloric acid (3.3), the precipitate was dissolved by heating and mashed filter, dilute with water to 100mL, heated to boiling, Remove. Mandelic acid solution was added 50mL (3.6) and continue heating to a boil, displacing the boiling water bath, heat 2h (without insulation on a boiling water bath,