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GB/T 6150.16-2026

Replacing GB/T 6150.16-2009

Methods for chemical analysis of tungsten concentrates - Part 16: Determination of iron, manganese, silicon, calcium and tungsten contents - X-ray fluorescence spectrometry

钨精矿化学分析方法 第16部分：铁、锰、硅、钙和钨含量的测定 X射线荧光光谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1.Structure and Drafting Rules of Standardization Documents". Drafting. This document is Part 16 of GB/T 6150, "Chemical Analysis Methods for Tungsten Concentrate". GB/T 6150 has already published the following parts.

1.Determination of Tungsten Trioxide Content by Ammonium Tungstate Gravimetric Method (oncinnabar)

2.Determination of Tin Content by Potassium Iodate Titration and Inductively Coupled Plasma Atomic Emission Spectrometry;

3.Determination of Phosphorus Content. Spectrophotometry and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

4.Determination of Sulfur Content by High-Frequency Induction Infrared Absorption Method and Combustion-Iodometric Method;

---Determination of calcium content using EDTA volumetric method and flame atomic absorption spectrometry;

6.Determination of Moisture Content in Wet Storage by Gravimetric Method;

---Determination of tantalum and niobium content using plasma atomic emission spectrometry and spectrophotometry;

8.Determination of Molybdenum Content by Thiocyanate Spectrophotometry;

---Determination of Copper Content by Flame Atomic Absorption Spectrometry;

10.Determination of Lead Content by Hydride Generation Atomic Fluorescence Spectrometry and Flame Atomic Absorption Spectrometry;

11.Determination of Impurity Element Content by Inductively Coupled Plasma Atomic Emission Spectrometry;

12.Determination of Silica Content by Silicomolybdenum Blue Spectrophotometric and Gravimetric Methods;

5.4 Lithium bromide solution (400 g/L). Weigh 40 g ±

0.001 g of lithium bromide, dissolve it in 80 mL of water, and dilute to 100 mL.

5.5 Hafnium oxide. w(HfO2)>=99.9%, calcined at 700°C for 2h.

5.6 Ferric oxide. w(Fe2O3)>=99.99%, dried at 105°C~110°C for 2h.

5.7 Manganese dioxide. w(MnO2)>=99.99%, dried at 105°C~110°C for 2h.

5.8 Silica. w(SiO2)>=99.99%, dried at 105°C~110°C for 2h.

5.9 Calcium carbonate. w(CaCO3)>=99.99%, dried at 105°C~110°C for 2h.

5.10 Tungsten trioxide. w(WO3)>=99.99%, dried at 105°C~110°C for 2h.

10 Mixed Gas. 10% methane and 90% argon, with a volume fraction of not less than 99.95%.

6 Instruments and Equipment

6.1 X-ray fluorescence spectrometer. wavelength dispersive type.

6.2 Muffle furnace.

6.3 Melting furnace. It is temperature-controllable and can heat up to 1200°C, and has an automatic oscillation function.

6.4 Platinum-gold crucible (95% Pt 5% Au). Volume not less than 30 mL.

6.5 Electronic balance. graduation value 0.01mg.

7 Samples

7.1 The particle size of the sample should not exceed

0.074 mm.

7.2 The sample should be dried at 105°C~110°C for 2 hours and then cooled to room

temperature in a desiccator for later use.

8 Test Procedure

8.1 Samples Weigh 0.5000g ± 0.0001g of sample (Chapter 7).

8.2 Parallel determination Perform two parallel experiments and take the average value.

8.3 Preparation of test samples

8.3.1 Weigh the sample (8.1), 7.0000g ± 0.0001g mixed flux (5.2), and 0.1000g ± 0.0001g hafnium oxide (5.5) into a container. In the platinum-gold crucible (6.4), mix with a glass rod and gently brush the sample adhering to the glass rod with a brush.

8.3.2 Add

1.0 mL of lithium nitrate solution (5.3) dropwise evenly to a platinum-gold crucible containing the sample (8.1), place it on a hot plate, and let it dry before rotating. Transfer to a muffle furnace at 700°C and pre-oxidize for 10 min, then remove. After cooling, uniformly add

0.1 mL of lithium bromide solution (5.4), and transfer to... Melt in a furnace at 1150°C~1200°C, keeping the furnace rotating or shaking the platinum-gold crucible (6.4) during the melting process, so that the platinum-gold crucible adheres to the metal. Small molten beads on the crucible wall and the sample entered the melt. After 10 minutes, the sample was completely melted and the melt was homogeneous. After standing for 1 minute, the platinum-gold crucible was... (6.4) Remove and cool the molded sample. Demold the sample from the mold. The molded sample should be a homogeneous glass with a smooth, flat surface and no unmelted material. The material should not contain crystals or bubbles; otherwise, it should be prepared again.

8.3.3 Remove the sample sheet and store it in a desiccator. When measuring, hold the sample sheet by its edge and avoid contaminating the X-ray measurement surface.

8.4 Preparation of a series of standard samples Prepare standard samples according to Table 2 using an electronic balance (6.5). Weigh the oxides of each element (if feasible, priority should be given to the corresponding national standard). A series of standard or industry standard samples), 7.0000g ± 0.0001g mixed flux (5.2) and 0.1000g ± 0.0001g hafnium oxide (5.5) are mixed together. Place the sample in a platinum-gold crucible (6.4), mix thoroughly with a glass rod, and gently brush off any residue adhering to the glass rod with a brush. The preparation of the series of standard samples shall follow 8.3.2. Step

8.3.3 is performed. The content of each element or oxide in the series of standard samples is shown in Table 3.

8.5 Measurement

8.5.1 Measurement conditions The recommended analytical lines and measurement conditions for the instrument are shown in Table 4.

8.5.2 Plotting the Working Curve

8.5.2.1 Input the mass fraction of each element or oxide to be measured from the series of standard samples (8.4) into the computer and run the X-ray fluorescence spectrometer. After stabilization, the X-ray fluorescence intensity of the series of standard samples (8.4) was measured according to the instrument measurement parameters given in Table

4.The series of standard samples... (8.4) The content of each element or oxide is used as the abscissa and the X-ray fluorescence intensity value is used as the ordinate to plot the working curve.

8.5.2.2 The absorption-enhancement effect between elements is corrected using the theoretical alpha coefficient or the basic parameter method to obtain a quadratic equation for intensity versus concentration. Using a linear equation, determine the calibration curve constants a and b, and save them in the calculator's quantitative analysis software. The linear correlation coefficient of the working curve should not exceed [a certain value]. Less than 0.999.

8.5.3 Instrument Drift Correction Before each measurement, the X-ray fluorescence intensity of the instrument is checked for significant changes by monitoring the sample or fused standard sample. If significant drift occurs, perform drift correction or redraw the working curve.

8.5.4 Determination of Sample Pieces Under optimal instrument conditions, the X-ray fluorescence intensity of the analyte element or oxide in the sample (8.3) was measured using computer software. The content of each element or oxide is calculated according to formula (1).

10 Precision

10.1 Repeatability The measured values of two independent test results obtained under repeatability conditions, within the range of the average values given in Table 5, represent the two test results... The absolute difference does not exceed the repeatability limit (r), and the number of cases exceeding the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is determined by linear regression based on the data in Table 5. The precision data were obtained using interpolation or extrapolation. Statistical results for the precision data are shown in Appendix A.

10.2 Reproducibility The measured values of two independent test results obtained under reproducibility conditions, within the range of the average values given in Table 6, represent