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Methods for chemical analysis of tungsten concentrates - Part 11: Determination of impurity element contents - Inductively coupled plasma atomic emission spectrometry

钨精矿化学分析方法 第11部分：杂质元素含量的测定 电感耦合等离子体原子发射光谱法

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Table of Contents

Foreword

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.

5.4 g) and

0.1 g strontium chloride (

5.5 g). Place the silver crucible in a muffle furnace, heat from a low temperature to 700 °C, and melt for 15 minutes. Remove and cool.

8.4.2.2 Immerse the cooled silver crucible in a 250mL beaker pre-filled with 100mL of hot water, and heat at a low temperature until it just boils, then wait for it to melt. After the block is completely dissolved, wash the crucible with water, boil it on an electric furnace, then remove it and cool it to room temperature. Filter it using medium-speed quantitative filter paper and use sodium carbonate solution. Wash the beaker 2 to 3 times with liquid (5.18) and the precipitate 4 to 5 times, then discard the filtrate.

8.4.2.3 Place the precipitate and filter paper back into the original beaker, add 20 mL of nitric acid (5.12) and 5 mL of perchloric acid (5.14). Cover with a watch glass and add... Heat until it emits concentrated perchloric acid fumes, and continue heating until nearly dry. Remove and let cool slightly. Add 5 mL of nitric acid (5.12). Rinse the watch glass and cup walls with water, and boil to dissolve. Dissolve the salts, cool to room temperature, transfer

to a 100 mL volumetric flask, dilute to the mark with water, and mix well. Filter dry.

8.4.2.4 Based on the barium content in the sample, transfer the test solution (8.4.2.3) to a 100 mL volumetric flask according to Table 2, add 5 mL of nitric acid (5.12), and use... Dilute with water to the mark and mix well.

8 Test Procedure

8.1 Samples Weigh the sample according to Table 2 (Chapter 7), accurate to 0.0001g.

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

8.3 Blank Test A blank test was conducted along with the sample.

8.4 Preparation of analytical solutions

8.4.1 Preparation of analytical solutions for bismuth, copper, lead, zinc, manganese, and iron

8.4.1.1 Place the sample (8.1) in a 250mL beaker, moisten it with a small amount of water, add 50mL of hydrochloric acid (5.9), and heat it in a boiling water bath. After 1 hour, remove and cool. Add 20 mL of nitric acid (5.11) and 2 mL of perchloric acid (5.14), and heat on an electric furnace until thick white fumes are produced and the volume is approximately [missing value]. 1 mL, after slightly cooling, rinse the beaker and watch glass with water to bring the solution volume to 50 mL, heat to dissolve the salts, and then remove and cool. Filter quantitatively using a slow filter. The solution was filtered through paper into a 100 mL volumetric flask. The beaker and precipitate were washed several times with nitric acid (5.16). 5 mL of nitric acid (5.11) was then added to the test solution. Dilute with water to the mark and mix well.

8.4.1.2 Based on the content of each element to be tested in the sample, transfer the test solution (8.4.1.1) to a 100 mL volumetric flask according to Table 2, and add 5 mL of nitric acid. (5.11) Dilute with water to the mark and mix well.

8.4.2 Preparation of Barium Analysis Solution Warning

---Caution required when handling molten sodium hydroxide. Wear safety goggles and gloves are recommended.

8.4.2.1 Place the sample (8.1) in a 30 mL silver crucible pre-filled with 2 g of anhydrous sodium carbonate (5.2), and cover it with 1 g of sodium hydroxide (5.3). 1 g sodium peroxide (

8.4.3 Preparation of Tin Analysis Solution

8.4.3.1 Place the sample (8.1) in a 10mL porcelain crucible pre-lined with 2g of zinc powder (5.6), stir well with a glass rod, and gently brush any residue adhering to the glass. Add 2g

of sodium hydroxide (5.3) to the sample on the glass rod, cover with 2g of sodium chloride (5.7), place the crucible in a 500°C high-temperature furnace, and wait for the furnace temperature to rise. When the temperature reaches 600°C, turn off the power, let it cool slightly, remove the crucible, and allow it to cool completely.

8.4.3.2 Place the crucible in a.200mL beaker, add 45mL of hydrochloric acid (5.17), heat until the sintered block is completely dissolved, rinse the watch glass, and while... Add 0.3g of potassium permanganate (5.8g) while hot, stir the solution continuously, and keep it in a hot water bath for 5 minutes until the solution turns completely yellow. Then cool. Then, transfer the test solution and precipitate together into a 100mL volumetric flask, dilute with water to the mark, mix well, and filter dry.

8.4.3.3 Take a portion of the test solution (8.4.3.2) according to Table 2 into a 250 mL beaker, add 2 mL of yttrium chloride solution (5.20), and add ammonia water (5.15) dropwise until... No further precipitate formed, so add 20 mL in excess. Heat to a gentle boil, remove from heat, filter using medium-speed quantitative filter paper, and wash the beaker with hot ammonia solution (5.19). Twice, settle three times, then wash once with water each time, and filter dry.

8.4.3.4 Place the precipitate and filter paper back into the original beaker, add 15 mL of hydrochloric acid (5.10), crush the filter paper, add 20 mL of water, and heat until the filter paper... It is in the form of pulp. Remove it, cool it to room temperature, transfer it to a 100mL volumetric flask, dilute it with water to the mark, mix well, and filter dry.

8.4.3.5 When the tin content (mass fraction) in the sample is greater than 0.10%,

5.00 mL of the test solution (8.4.3.2) can also be aliquoted into 100 mL. Add 10 mL of hydrochloric acid (5.10) to a volumetric flask, dilute with water to the mark, and mix well.

8.4.4 Preparation of Phosphorus Analysis Solution

8.4.4.1 Place the sample (8.1) in an iron crucible [containing 4g of sodium peroxide that has been preheated to remove moisture in a high-temperature electric furnace with a graphite ring]. In (5.4), cover with 1g of sodium hydroxide (5.3), heat on an electric furnace to remove moisture, and place the crucible in a high-temperature furnace at 700°C~750°C. Melt for 10-15 minutes, then remove and let cool slightly.

8.4.4.2 Place the crucible in a 300mL beaker containing 80mL of water, wash the crucible with water, and add 20mL of hydrochloric acid dropwise while stirring continuously. (5.10) Acidify the solution, cool it, transfer it to a.200mL volumetric flask, dilute it to the mark with water, and mix well.

8.4.4.3 Transfer

10.00 mL of the supernatant solution (8.4.4.2) into a 100 mL volumetric flask, dilute with

water to the mark, and mix well. Preparation of

8.5 series standard solutions

8.5.1 Preparation of standard solutions of bismuth, copper, lead, zinc, manganese and iron series Transfer the corresponding standard stock solutions (5.22~5.28) from Table 3 into.200mL volumetric flasks, add 10mL of nitric acid (5.11), and dilute with water. Dilute to the mark and mix well. The mass concentrations of each element in this series of standard solutions are shown in Table 4.

8.5.3 Preparation of Tin Series Standard Solutions

8.5.3.1 Carrier separation-inductively coupled plasma atomic emission spectrometry determination of a series of standard solutions. 0 mL,

10.00 mL of tin standard solution B (5.33), and

10.00 mL of tin standard solution A (5.32) were dispensed into six 100 mL containers. Add 15 mL of hydrochloric acid (5.10) to each volumetric flask, dilute to the mark with water, and mix well. The mass concentrations of tin in this series of standard solutions are as follows: 0μg/mL, 0.20μg/mL, 0.50μg/mL, 1.00μg/mL, 5.00μg/mL, 10.00μg/mL.

8.5.3.2 Direct determination - Inductively coupled plasma atomic emission spectrometry (ICP-AES) for the determination of a series of standard solutions. Transfer 0 mL,

10.00 mL of tin standard solution B (5.33), and

5.00 mL of tin standard solution A (5.32) were dispensed into six 100 mL volumes. Add 5 mL of blank solution and 10 mL of hydrochloric acid (5.10) to each bottle, dilute to the mark with water, and mix well. The mass of tin in this series of standard solutions... The concentrations were 0 μg/mL, 0.20 μg/mL, 0.50 μg/mL, 1.00 μg/mL, 2.00 μg/mL, and 5.00 μg/mL, respectively.

8.5.4 Preparation of Phosphorus Series Standard Solutions Transfer 0 mL,

0.50 mL,

10.00 mL of phosphorus standard solution (5.34) to a set of 100 mL containers. In a volumetric flask, add

1.0 mL of hydrochloric acid (5.10) and

2.5 mL of sodium hydroxide solution (5.21), dilute to the mark with water, and mix well. This series of standard solutions... The phosphorus concentrations were 0 μg/mL, 0.50 μg/mL, 1.00 μg/mL, 2.00 μg/mL, 5.00 μg/mL, and 10.00 μg/mL, respectively.

8.6 Measurement After the inductively coupled plasma atomic emission spectrometer has stabilized, analyze the spectral lines according to the wavelengths recommended in Table 5, and apply the results to a series of standard solutions. (8.5) The determination was