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

SN/T 5266-2020

**Determination of copper, iron, arsenic, zinc, cadmium, mercury
and silver content in mixtures-inductively coupled plasma
emission spectrometry**

混合中铜、铁、砷、锌、镉、汞和银含量的测定 电感耦合等离子体发射光谱法
(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Nanjing Customs of the People's Republic of China.

1 Scope

This standard specifies the use of inductively coupled plasma emission spectrometer to determine copper, iron, arsenic, zinc, cadmium, mercury and Method of silver content.

This standard applies to the determination of copper, iron, arsenic, zinc, cadmium, mercury and silver content in mixed lead-zinc ore. The measurement range is shown in Table 1.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated

reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 602 Preparation of standard solutions for impurity determination of chemical reagents
GB/T.2007.2 General Rules for Sampling and Sample Preparation of Bulk Mineral Products
Manual Sample Preparation Method GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

Add nitric acid, hydrochloric acid, and fluoroboric acid to the sample for digestion in a microwave digestion apparatus, and use an inductively coupled plasma emission spectrometer for digestion.

Perform determination and determine the content of each element according to the calibration curve.

4 Reagents and materials

Unless otherwise specified, only secondary water or equivalent purity water that meets the requirements of GB/T 6682 is used in the analysis.

4.1

Hydrochloric acid ($\rho=1.19$ g/mL), excellent grade pure.

4.2

Nitric acid ($\rho=1.42$ g/mL), excellent grade pure.

4.3

Nitric acid (1:20).

4.4

Fluoroboric acid ($\rho=1.84$ g/mL), excellent grade pure.

4.5

Standard stock solutions of copper, iron, arsenic, zinc, cadmium, mercury, and silver. prepare copper, iron, arsenic, zinc, the stock solutions of cadmium, mercury, and silver have a mass concentration of 1,000 $\mu\text{g/mL}$; or directly purchase a certified standard solution (the solution medium is nitric acid).

4.6

Mixed standard solutions of copper, iron, arsenic and zinc. Pipette 10 mL of standard solutions (4.5) of copper, iron, arsenic, and zinc into 100 mL respectively.

In a volumetric flask, dilute to the mark with nitric acid (4.3) and mix well.

4.7

Mixed standard solutions of cadmium, mercury, and silver. Pipette 1 mL of standard solutions (4.5) of cadmium, mercury, and silver into a 100 mL volumetric flask.

Dilute to the mark with nitric acid (4.3) and mix well.

5 Instruments

5.1

Inductively coupled plasma emission spectrometer. see Appendix A for the working conditions of the instrument.

5.2

Microwave digestion apparatus. the volume of the digestion tank is 50 mL ~ 100 mL.

5.3

Analytical balance. Sensitivity is 0.1 mg.

5.4

Oven. The temperature control accuracy is $\pm 5^{\circ}\text{C}$.

6.1 Sample preparation

According to the relevant regulations of GB/T.2007.2, the maximum particle size of the sample is 180 μm , and it should be mixed thoroughly before use. Before sample analysis Dry for 2 h at $105^{\circ}\text{C} \sim 110^{\circ}\text{C}$, and cool to room temperature in a desiccator.

6.2 Sample digestion

Weigh 0.2 g sample (6.1), accurate to 0.1 mg, place it in the digestion tank, add 6 mL hydrochloric acid (4.1), 2 mL nitric acid (4.2), 1 mL fluoroboric acid (4.4), after the sample reacts with the acid, close the digestion tank, put it into the microwave digestion apparatus, and operate according to the procedure of the instrument (see Appendix B), after the sample is digested completely, take it out and cool to room temperature, open the digestion tank, transfer all the samples to a 100 mL volumetric flask, and use Constant volume of water, shake well, filter, to be tested.

Note 1: In order to prevent cross-contamination, all the utensils in contact with the sample should be soaked in 10% HNO_3 for 24 hours (except those that cause corrosion), and rinsed with ultrapure water for 3 times.

Let dry for later use.

Note 2: After the sample is digested, make sure the volume and then filter.

Note 3: The maximum temperature of microwave digestion has a significant impact on the sample

digestion, please operate strictly in accordance with the requirements.

6.3 Blank test

Carry out a blank test with the sample.

6.4 Preparation of working curve solution

Preparation of working curves for copper, iron, arsenic, zinc, cadmium, mercury, and silver. pipette 0.00 mL, 0.25 mL, 0.50 mL, 2.50 mL, and 5.00 mL, 10.00 mL copper, iron, arsenic, zinc mixed standard solution (4.6) and 0.00 mL, 0.25 mL, 0.50 mL, 1.00 mL, 2.50 mL, 5.00 mL cadmium, mercury, and silver mixed standard solution (4.7) is diluted to the mark with nitric acid (4.3) in a series of 50 mL volumetric flasks, and mixed. this the content of the standard series of solutions is shown in Table 2.

6.5 Determination

According to the analytical conditions of the inductively coupled plasma emission spectrometer (see Appendix A), measure the working curve solution (6.4) of each element Spectral intensity, when the correlation coefficient of the working curve is greater than or equal to 0.995, the blank solution and the sample solution are measured, and the calibration curve the line determines the content of each measured element. If the concentration of the measured element is found to exceed the range of the calibration curve after the measurement, the sample solution can be used with water Re-measure after proper dilution.

7 Result calculation

The content of the measured element is calculated as the mass fraction ω , calculated according to formula (1).

8 Results presentation

Take the average of the results of two parallel determinations. If the calculated result is greater than or equal to 0.1%, the result shall be kept to two decimal places; if calculated the result is less than 0.1%, and the result is to four decimal places.

9 Precision

The precision was measured by 11 laboratories on 3 levels of samples, and the data was determined after statistics. The results are shown in Table 3.