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SN

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

SN/T 5255-2020

Determination of lead, cadmium, arsenic, mercury, copper, chromium, nickel, manganese, barium, selenium, zinc, tin, titanium, antimony, cobalt, aluminum, beryllium, vanadium, silver and molybdenum in coal by inductively coupled plasma mass spectrometry

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(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 organizations of this standard. Qingdao Customs of the People's Republic of China, Shijiazhuang Customs of the People's Republic of China.

1 Scope

This standard specifies the determination of lead, cadmium, arsenic, mercury, copper, chromium, nickel, and copper in coal by microwave digestion-inductively coupled plasma mass spectrometry.

Manganese, barium, selenium, zinc, tin, titanium, antimony, cobalt, aluminum, beryllium, vanadium, silver, and molybdenum content of 20 elements.

This standard applies to lead, cadmium, arsenic, mercury, copper, chromium, nickel, manganese, barium, selenium, zinc, tin, titanium, antimony, cobalt, aluminum,

Determination of 20 elements of beryllium, vanadium, silver and molybdenum. 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. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 474 Coal sample preparation method GB/T 602 Preparation of standard solutions for impurity determination of chemical reagents GB/T 6682 Analytical laboratory water specifications and test methods GB/T 8170 Numerical rounding rules and the expression and determination of limit values

3 Principles of the method

After the sample is digested by microwave, it is introduced into an inductively coupled plasma mass spectrometer for measurement. Using online internal standard, the mass spectrum signal of the element to be measured is compared with the intensity ratio of the internal standard element mass spectrum signal is proportional to the concentration of the element to be measured for quantitative analysis.

4 Reagents and materials

Unless otherwise specified, only use the reagents above the superior grade and the water is the second grade water that meets the requirements of GB/T 6682.

4.1

Hydrochloric acid ($\rho=1.19$ g/mL).

4.2

Hydrofluoric acid ($\rho=1.30$ g/mL).

4.3

Nitric acid ($\rho=1.42$ g/mL).

4.4

Nitric acid solution (2:98, volume ratio). Take an appropriate amount of nitric acid (4.3) and ultrapure water and mix with a volume ratio of 2:98.

4.5

Standard storage of lead, cadmium, arsenic, mercury, copper, chromium, nickel, manganese, barium, selenium, zinc, tin, titanium, antimony, cobalt, aluminum, beryllium,

vanadium, silver, and molybdenum Preparation solution (1 000 µg/mL). Prepare according to GB/T 602 or directly purchase certified reference materials.

4.6

Standard solution series. Use standard stock solution (4.5) to prepare mixed standard working solution series according to Table 2.

Note: The medium is 2% nitric acid.

4.7

Mass spectrometry tuning solution. 1 µg/L mixed standard solution of lithium, yttrium, cerium, thallium, and cobalt, prepared according to GB/T 602 or directly purchased with a certified standard substance.

4.8

Mixed internal standard solution. 10 mg/L mixed standard solution of scandium, lithium, indium, bismuth, germanium, and rhodium, prepared according to GB/T 602 or purchased directly Certified reference materials.

4.9

Internal standard solution (online internal standard solution). take 2.5 mL of mixed internal standard solution (4.8) and dilute to 50 with 2% nitric acid (volume fraction) mL to obtain a standard internal standard solution with a concentration of 500 µg/L.

4.10

Argon gas. purity $\geq 99.99\%$.

4.11

Helium. purity $\geq 99.99\%$.

5 Apparatus and equipment

5.1

Inductively Coupled Plasma Mass Spectrometer (ICP-MS). equipped with hydrofluoric acid solution atomization injection system. See Appendix A for instrument operating parameters.

5.2

High temperature pressure microwave digestion instrument. equipped with high temperature resistant pressure sealed digestion tank and the digestion tank must be able to reach above 205 °C in actual operation.

See Appendix B for instrument operating parameters.

5.3

Analytical balance. Sensitivity 0.1 mg.

5.4

Pipette. The pipette has a volume of 100 μL , 1,000 μL , and 10 mL.

6 Sample preparation

Prepare samples in accordance with GB/T 474, pass through a 0.2 mm sieve, bake at 105 °C for 2 h, then place in a desiccator and cool to the room Warm spare.

7.1 Sample

Weigh 0.10 g sample to the nearest 0.0001 g.

7.2 Number of determinations

Weigh two samples for parallel measurement, and take the average of the results.

7.3 Blank test

Except that the sample to be tested is not added, the blank test shall be carried out in accordance with 7.4.

7.4 Digestion of the sample

Add 2 mL of hydrochloric acid (4.1), 1 mL of hydrofluoric acid (4.2), and 7 mL of nitric acid (4.3) to the sample, shake well, tighten the sealed digestion tank, and place into the microwave digestion apparatus. Refer to the working conditions of microwave digestion in Appendix B for microwave digestion. Take it out, cool to room temperature, open the digestion tank, Transfer the digested solution directly to a 100 mL plastic volumetric flask, rinse the digestion tank and lid with deionized water 3 to 5 times, and combine the washing liquids in the mother liquor, dilute to the mark with 2% nitric acid, mix well, and stand for measurement.

7.5.1 Drawing the standard curve

Use the tuning solution (4.7) to tune the various indicators of the instrument to meet the measurement requirements, establish the analysis program according to the instrument manual, and edit the measurement Method (see Table A.1 for the working parameters of the inductively coupled plasma mass spectrometer), select the elements to be measured and the corresponding internal standard elements (see Table A.1) A.2), the standard series of solutions are measured in the order of concentration from low to high, the signal intensity of each measured element and its internal standard element signal intensity value the ratio