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Iron ores - Determination of multiple trace elements - Inductively coupled plasma mass spectrometric method

铁矿石 多种微量元素含量的测定 电感耦合等离子体质谱法

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Iron ores - Determination of multiple trace elements - Inductively coupled plasma mass spectrometric method

WARNING -- Personnel using this document shall have regular laboratory work experience. This document does not point out all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions specified in relevant national regulations.

1 Scope

This Part of GB/T 6730 specifies the method for determining the content of multiple trace elements in iron ores by inductively coupled plasma mass spectrometric method.

This Part applies to the determination of the content of lead, cadmium, arsenic, mercury, barium, cobalt, thallium, molybdenum, yttrium, lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, dysprosium, holmium, erbium, thulium, ytterbium elements in iron ores. The determination range is shown in Table 1.

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 10322.1 Iron ores - Sampling and sample preparation procedures

GB/T 12806 Laboratory glassware - One-mark volumetric flasks

3 Principle

After microwave digestion, the sample is introduced into an inductively coupled plasma mass spectrometer for determination. Use on-line internal standard, and carry out quantitative analysis on the basis that the intensity ratio of the mass spectrum signal of the element to be determined to the mass spectrum signal of the internal standard element is proportional to the concentration of the element to be determined.

4 Reagents and materials

Unless otherwise stated, only approved guaranteed reagents and secondary water complying with GB/T 6682 or water equivalent to its purity are used in the analysis.

4.1 Hydrochloric acid, ρ is about 1.19 g/mL.

4.2 Hydrofluoric acid, ρ is about 1.30 g/mL.

4.3 Nitric acid, ρ is about 1.42 g/mL.

4.4 Nitric acid, 2 + 98. TAKE an appropriate amount of nitric acid (see 4.3) and water, MIX them in a volume ratio of 2:98.

4.5 Lead, cadmium, arsenic, mercury, barium, cobalt, thallium, molybdenum, yttrium, lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, dysprosium, holmium, erbium, thulium, ytterbium standard stock solutions (1000 μg/mL): PREPARE according to GB/T 602 or use certified standard solutions.

4.6 Series of standard solutions: USE the standard stock solutions (see 4.5) to prepare a series of standard solutions according to Table 2. The medium is 2 % nitric acid (see 4.4).

4.8 Mixed internal standard solution: 10 mg/L mixed standard solution of lutetium, indium, bismuth, germanium, rhodium, and terbium. Prepare according to GB/T 602 or use a certified standard solution.

4.9 Internal standard solution (on-line internal standard solution): Take 2.5 mL of the mixed internal standard solution (see 4.8), adjust the volume to 50 mL with nitric acid (see 4.4), to obtain a standard internal standard solution with a concentration of 500 μg/L.

4.10 Argon gas: purity ≥ 99.99 %.

4.11 Helium: purity ≥ 99.99 %.

5 Instruments

Unless otherwise stated, use general laboratory instruments in the analysis. One-mark volumetric flasks shall comply with the provisions of GB/T 12806.

5.1 Inductively coupled plasma mass spectrometer (ICP-MS): equipped with a hydrofluoric acid solution resistant atomization sample injection system. See Annex A for working parameters of the instrument.

5.2 Microwave digestion instrument: see Annex B for working parameters.

5.3 Analytical balance: the scale value is 0.1 mg.

5.4 Pipette: the pipette range is 100 μL, 1000 μL, and 10 mL.

6 Sampling and sample preparation

6.1 Laboratory samples

Sampling and preparation shall be carried out in accordance with GB/T 10322.1. Generally, the sample particle size is less than 100 μm. If the content of combined water or easy oxides in the sample is high, the particle size shall be less than 160 μm.

For provisions regarding high combined water and easy oxides content, see GB/T 6730.1.

6.2 Pre-dried samples

Thoroughly mix the laboratory samples and take samples using the sample splitting method. According to the provisions of GB/T 6730.1, dry the sample at a temperature of 105 ℃ ± 2 ℃, and cool it to room temperature in a desiccator for later use.

7 Analysis steps

7.1 Number of determinations

According to Annex C, the same pre-dried sample shall be determined independently at least twice, and the average value of the results shall be taken.

NOTE: "Independent" means that the results of the second and any subsequent determination are not affected by the results of the previous determinations. In this analytical method, this condition means that the same determined object is independently and repeatedly determined in a short period of time by the same operator using the same equipment and the same test method in the same laboratory, including using appropriate recalibration.

7.2 Sample mass

Weigh 0.10 g of pre-dried sample (see 6.2), accurate to 0.0001 g. The sample weighing operation shall be carried out as quickly as possible to prevent the sample from absorbing moisture again.

7.3 Blank test and verification test

7.3.1 Blank test

Except for not adding the sample to be determined, the blank test shall be carried out in accordance with 7.4. All reagents shall be taken from the same reagent bottle. When analyzing multiple samples, it can use a blank value.

7.3.2 Verification test

Along with the sample analysis, carry out the verification test for reference materials of the same type.

7.4 Digestion of sample

ADD 7 mL of hydrochloric acid (see 4.1), 1 mL of hydrofluoric acid (see 4.2), and 2.5 mL of nitric acid (see 4.3) to the sample, SHAKE well, TIGHTEN the sealed digestion tank, and PUT it into the microwave digestion instrument. Refer to the microwave digestion working conditions in Annex 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 water 3 ~ 5 times, COMBINE the washing liquid into the solution, DILUTE with nitric acid (see 4.4) to the mark, MIX well, and LET STAND for later test.

7.5 Quantitative determination

7.5.1 Plotting of calibration curve

Inter-laboratory precision is used to evaluate the consistency between the final results reported by two laboratories. After the two laboratories report the results according to the same steps specified in 8.2.2, calculate according to formula (2):

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \qquad \cdots\cdots (2)$$

where:

μ_{12} - the average value of the final results;

μ_1 - the final result reported by Laboratory 1;

μ_2 - the final result reported by Laboratory 2.

If $|\mu_1 - \mu_2| \leq R$, the final results of the two laboratories are consistent.

8.2.4 Correctness check

The correctness check uses a certified reference material (CRM) or a reference material (RM) for verification. The final laboratory result is compared with the standard value A_c of CRM or RM. There will be two possibilities.

a) $|\mu_c - A_c| \leq C$, in this case, there is no significant difference between the determined value and the standard value.

b) $|\mu_c - A_c| > C$, in this case, there is a significant difference between the determined value and the standard value.

where:

μ_c - the determined value of CRM or RM;

A_c - the standard value of CRM or RM;

C - the value depends on the type of CRM / RM used.

The C value of the certified reference material (CRM) or reference material (RM) determined by multiple laboratories is calculated according to formula (3):

$$C = \frac{1}{\sqrt{2}} \sqrt{R^2 - \frac{n-1}{n} r^2 + 8u^2} \qquad \cdots\cdots (3)$$

where:

R - the inter-laboratory reproducibility limit;

r - the in-laboratory repeatability limit;

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