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GB/T 14506.30-2010

Methods for chemical analysis of silicate rocks - Part 30: Determination of 44 elements

硅酸盐岩石化学分析方法 第30部分：44个元素量测定

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1 Scope

GB/T 14506.30-2010 is the Chinese national standard on methods for chemical analysis of silicate rocks - part 30: determination of 44 elements, in the field of mining and minerals. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 10 November 2010 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 February 2011. Classification: ICS 73.080, CCS D53. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part of GB/T 14506 specifies a method for the determination of the contents of 44 elements in silicate rocks using closed-vessel acid digestion and inductively coupled plasma mass spectrometry (ICP-MS). This Part is applicable to the determination of the contents of 44 elements - lithium, beryllium, scandium, titanium, vanadium, manganese, cobalt, nickel, copper, zinc, gallium, arsenic, rubidium, strontium, yttrium, zirconium, niobium, molybdenum, cadmium, indium, cesium, barium, lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, lutetium, hafnium, tantalum, tungsten, thallium, lead, bismuth, thorium, and uranium - in silicate rocks. It is also applicable to the determination of the contents of the aforementioned elements in soil and sediment samples. This Part is not applicable to the determination of element contents in samples with an aluminum oxide content exceeding 20%. Determination range, see Annex A.

2 Normative references

The provisions in following documents become the provisions of this Part of

GB/T 14506 through reference in this Part. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Part, however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 14506.1, Methods for chemical analysis of silicate rocks -- Part 1. Determination of hygroscopic water content

3 Principle

The sample is dissolved using hydrofluoric acid and nitric acid in a closed digestion vessel. The hydrofluoric acid is evaporated off on a hot plate, followed by a second dissolution step with nitric acid in a sealed vessel. After dilution, the sample is analyzed directly by ICP-MS using the external standard method.

4 Reagents and solutions

4.1 Water is purified using an ion-exchange purification system. Prior to use, the content of the target elements in the water is verified to ensure it is below the method detection limit.

4.2 Nitric acid (rho

1.42 g/mL). guaranteed reagent or high-purity grade; purified by sub- boiling distillation before use.

4.3 Nitric acid (1+1).

4.4 Hydrofluoric acid (rho

1.16 g/mL). guaranteed reagent or high-purity grade, purified by sub-boiling distillation before use. WARNING -- Hydrofluoric acid is toxic and corrosive. Wear gloves during handling to prevent skin contact.

4.5 Single-element standard stock solution. for specific preparation details, refer to Annex B.

4.6 Multi-element mixed standard stock solution Prepare the following multi-element mixed standard stock solution by directly aliquoting single-element standard stock solutions (4.5). Alternatively, it may be obtained by diluting a commercially available multi-element mixed standard stock solution (see Table 1). NOTE. When preparing multi-element stock standard solutions, consider the compatibility and stability of the elements. Inspect the primary elemental stock standard solutions to ensure that impurities do not compromise the accuracy of the standards. Transfer newly prepared standard solutions into unused, acid-washed polypropylene bottles for storage, and periodically monitor their stability. Table 1 -- Multi-element mixed standard stock solution

4.7 Calibration standard solution Prepare calibration standard solutions by diluting the multi-element mixed standard stock solution (4.6). Transfer 100 µL of the multi-element mixed standard stock solution (4.6) into a 100 mL volumetric flask. Add 5 mL of nitric acid (4.2) and dilute to the mark with water (4.1). Mix well. Calibration standard solution 3 shall be prepared immediately before use, with the addition of

0.1 mL of hydrofluoric acid (4.4).

4.8 Mixed solution of internal standard elements Directly aliquot rhodium and rhenium single-element standard stock solutions (4.5) to prepare a mixed solution of internal standard elements, with rhodium and rhenium concentrations of 10 ng/mL each.

4.9 Blank solution

a) Calibration blank solution. Nitric acid solution (5+95);

b) Rinse blank solution. Nitric acid solution (2+98).

4.10 Single-element interference solution Single-element solutions of Ba, Ce, Pr, Nd, Zr, and Sn (each at a concentration of 1 µg/mL), Ti (10 µg/mL), and Fe and Ca (each at 250 µg/mL) are prepared to determine the interference coefficient k.

5.1 Inductively coupled plasma mass spectrometer

a) The instrument is capable of scanning within the mass range of 5 u~250 u. The minimum resolution is a peak width of 1 u at 5% of the peak height. Operating parameters for a quadrupole inductively coupled plasma mass spectrometer (ICP- MS), for example, are

provided in Annex C.

b) Argon. High-purity grade (argon mass fraction >= 99.99%).

5.2 Sealed sample digestion vessel. stainless steel outer shell, PTFE inner liner, 10 mL capacity.

5.3 Drying oven. Maximum temperature at 250°C.

5.4 Thermostatically controlled electric hot plate. maximum temperature at 250°C.

5.5 Analytical balance. Class II, readability of

0.01 mg.

5.6 Air-displacement pipettes. specifications of 10 µL~100 µL, 100 µL~1000 µL, and 1 mL~5 mL.

5.7 Disposable plastic bottles. capacity of 25 mL or 50 mL.

6 Specimen

6.1 The particle size of the specimen shall be less than 74 µm.

6.2 The specimen shall be pre-dried at 105°C for 2 h~to 4 h and then placed in a desiccator to cool to room temperature.

6.3 For rocks that readily absorb water, air-dried specimens shall be used. Concurrently with weighing the specimen, the content of adsorbed water shall be determined in accordance with GB/T 14506.1.The final results shall be calculated on a dry-state basis.

7 Analysis steps

7.1 Blank test Duplicate blank tests are to be conducted alongside the test materials. The reagents used must be taken from the same bottle and added in equal amounts.

7.2 Verification test Analyze a certified reference material of the same ore type and similar content alongside the test material.

7.3.1 Accurately weigh 25 mg or 50 mg (to the nearest

0.01 mg) of the test material into the inner vessel of a sealed digestion device.

7.3.2 Add 1 mL of hydrofluoric acid (4.4) and

0.5 mL of nitric acid (4.2), and seal the vessel. Place the dissolution vessel in an oven and heat for 24 h, maintaining the temperature at approximately 185°C ± 5°C.

7.3.3 After cooling, remove the inner vessel and place it on a hot plate to evaporate the