

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Soil and sediment - Determination of aqua regia extracts of 12
metal elements-Inductively coupled plasma mass spectrometry
(English Translation)**

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1 Scope

This Standard specifies the inductively coupled plasma mass spectrometry method for the determination of 12 metal elements in aqua regia extracts of soil and sediment.

This Standard applies to the determination of 12 metal elements -- cadmium (Cd), cobalt (Co), copper (Cu), chromium (Cr), manganese (Mn), nickel (Ni), lead (Pb), zinc (Zn), vanadium (V), arsenic (As), molybdenum (Mo), and antimony (Sb) -- in soil and sediment. If verified, this Standard can also be applied to the determination of other metal elements.

When the sampling volume is 0.10g and the constant volume after digestion is 50ml, the detection limits and lower determination limits of the 12 metal elements are shown in Table 1.

Translator NOTE: In the table, A: cadmium B: cobalt C: copper D: chromium E: manganese F:

nickel G: lead H: zinc I: vanadium J: arsenic K: molybdenum L: antimony.

2 Normative references

This Standard refers to the following documents or their clauses. For undated references, the latest edition of the referenced document applies.

HJ/T 166, Technical specification for soil environmental monitoring

HJ 613, Soil - Determination of dry matter and water content - Gravimetric method

GB 17378.3, The specification for marine monitoring - Part 3: Sample collection, storage and transportation

GB 17378.5, The specification for marine monitoring - Part 5: Sediment analysis

3 Method principle

Soil and sediment samples are digested by a mixed solution of hydrochloric acid/nitric acid (aqua regia) on an electric heating plate or microwave digestion apparatus. Use an inductively coupled plasma mass spectrometer to test. Qualification is based on the mass spectrum or characteristic ions of the element. Use internal standard method for quantification.

The specimen is brought into the atomization system by the carrier gas for atomization.

The target element goes into the axial channel of the plasma in the form of aerosol. It is fully evaporated, dissociated, atomized and ionized at high temperature and inert gas.

It is then converted into charged positive ion. It enters the mass spectrometer through the ion acquisition system. The mass spectrometer separates and analyzes qualitatively and quantitatively according to the mass-to-charge ratio of the ion. Within a certain concentration range, the response value corresponding to the mass-to-charge ratio of an ion is proportional to its concentration.

4.1 Mass spectrum interference

Mass spectrum interference mainly includes polyatomic ion interference, isobaric interference, oxide and doubly charged ion interference.

Polyatomic ion interference is the most important source of interference in ICP-MS. It can use interference correction equations, instrument optimization, and collision- reaction cell techniques to resolve. See Table A.1 in Annex A for common polyatomic ion interferences. Isobaric interferences can be corrected using the interference correction equation. Or before the analysis, eliminate the sample by chemical separation and other methods. See Table A.2 in Annex A for the main interference correction equations. Oxide interference and double charge interference can be reduced by adjusting the instrument parameters.

4.2 Non-mass spectrum interference

It can use high-purity metals (purity is greater than 99.99%) or metal salts (baseline or high-purity reagents) to prepare. Commercially available certified reference materials can also be purchased for preparation. The medium is nitric acid solution (5.4).

5.7.5 Internal standard reference use solution: $\rho = 100\mu\text{g/L}$.

Use nitric acid solution (5.4) to dilute the internal standard reference stock solution (5.7.4) and prepare the internal standard reference use solution. Due to the different diameters of peristaltic pump tubes used in different instruments, when adding internal standard online, the concentration added is also different. Therefore, when preparing the internal standard reference use solution, the concentration of the internal standard element in the specimen shall be 10~50 $\mu\text{g/L}$.

5.7.6 Tuning solution: $\rho = 10\mu\text{g/L}$.

It is advisable to choose a solution containing Li, Be, Mg, Y, Co, In, Tl, Pb and Bi elements as the tuning solution of the mass spectrometer. It can use high-purity metals (purity is greater than 99.99%) or corresponding metal salts (baseline or high-purity reagents) to prepare. Commercially available certified reference materials can also be purchased

NOTE 1: Reference solutions of all elements shall be stored in sealed polyethylene or polypropylene bottles after preparation.

5.8 Slow quantitative filter paper.

5.9 Carrier gas: Argon, purity $\geq 99.999\%$.

6 Instruments and equipment

6.1

Inductively coupled plasma mass spectrometer

The mass range that can be scanned is 5~250u. The peak width at a resolution of 10% peak height shall be between 0.6~0.8u.

6.2

Temperature control electric heating plate

The control accuracy is $\pm 0.2^\circ\text{C}$. The maximum temperature can be set up to 250°C .

6.3

Microwave digester

The output power is 1000~1600W. It has programmable control function. It can carry out full monitoring of temperature, pressure and time (heating time and holding time). It has safety protection function.

6.4

Analytical balance

The accuracy is 0.0001g.

6.5

Teflon closed digestion vessel

It can resist pressure, acid and corrosion. It has the function of pressure relief.

6.6

Erlenmeyer flask

100ml.

6.7

Glass funnel.

6.8

Volumetric flask

50ml.

6.9

Nylon sieve

0.15mm (100 mesh).

6.10

General laboratory instruments and equipment.

7.1 Sample collection and storage

Collect and preserve soil samples in accordance with the relevant regulations of HJ/T

166. Collect and preserve sediment samples in accordance with the relevant provisions of GB 17378.3. Contamination and loss of analyte elements shall be avoided during sample collection, transportation and storage.

7.2 Determination of moisture

Determination of dry matter of soil samples is carried out in accordance with HJ 613.

The moisture content of sediment samples shall be carried out in accordance with GB 17378.5.

7.3 Sample preparation

Remove sticks, leaves, stones and other foreign objects in the sample. According to the requirements of HJ/T 166 and GB 17378.5, the collected samples are air-dried, coarsely