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Soil and stream sediment - Determination of iodine content - Pressurized ammonia extraction inductively coupled plasma mass spectrometry

土壤、水系沉积物 碘含量的测定 氨水封闭溶解-电感耦合等离子体质谱法

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Warning – Personnel using this document must have practical experience in formal laboratory work. This document does not address all possible security issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions which are set by the relevant national regulations.

1 Scope

This document describes a method for the determination of iodine content in soil and stream sediment by pressurized ammonia extraction inductively coupled plasma mass spectrometry.

This document is applicable to the determination of iodine content in soil and steam sediment with the method detection limit and measurement range as follows:

-- method detection limit 0.07 μg/g;

-- measurement range 0.23 μg/g ~ 100 μg/g.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 6041, General rules for mass spectrometric analysis

GB/T 6379.2, Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 6379.4, Accuracy (trueness and precision) of measurement methods and results - Part 4: Basic methods for the determination of the trueness of a standard measurement method

GB/T 6682, Water for analytical laboratory use - Specification and test methods

GB/T 14505-2010, Method for chemical analysis of rocks and ores - General rules and regulations

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

GB/T 33087, Ultra pure water for instrumental analysis specification and test methods

JJF 1159, Calibration specification for quadrupole inductively coupled plasma mass spectrometers

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Principle

The sample is heated with dilute ammonia in a sealed sample dissolver to extract iodine. The sample solution is measured using an inductively coupled plasma mass spectrometer and qualitatively determined by the iodine isotope ^{127}I. Within a certain concentration range, the iodine element mass spectrum count value is proportional to the mass concentration of the iodine element. The iodine content in the sample is calculated by measuring the iodine element mass spectrum count value.

5 Test conditions

The test conditions such as temperature, humidity, voltage and frequency during inductively coupled plasma mass spectrometer detection shall comply with the relevant requirements of GB/T 6041 and JJF 1159.

6 Reagents or materials

Unless otherwise stated, the reagents used in the analysis are analytical reagents, and the water is grade-1 water specified in GB/T 6682 or high-purity water for instrumental analysis specified in GB/T 33087.

6.1 Ammonia [$\omega(NH_3) = 25\% \sim 28\%$].

6.2 Ammonia solution (1+9).

6.3 Ammonia solution (1+99).

7.4 Centrifuge: the maximum speed is 10 000 r/min.

7.5 Sealed sample dissolver: stainless steel outer sleeve, polytetrafluoroethylene inner tank, volume 15 mL.

7.6 Graduated test tube: 10 mL.

8 Sample

8.1 Prepare the sample in accordance with the relevant provisions of 4.1 in GB/T 14505-2010. The particle size of the sample shall be less than 74 μm.

8.2 After the sample is naturally air-dried at room temperature, place it in a desiccator for later use.

8.3 Weigh 0.1 g of the sample, accurate to 0.1 mg, which is the test portion.

8.4 Measure the adsorbed water of the sample dried at room temperature according to the method specified in GB/T 14506.1.

9 Test steps

9.1 Blank test

Carry out duplicate blank tests along with the test portion. The reagents used shall be taken from the same bottle of reagents and added in equal amounts.

9.2 Verification test

Analyze standard materials of the same type and similar content along with the test portion.

9.3 Sample decomposition

9.3.1 Place the test portion (8.3) in the polytetrafluoroethylene inner tank of the sealed sample dissolver (7.5); add 5 mL of ammonia solution (6.2); cover the crucible lid; put it into the steel sleeve; tighten it; put it in the digital temperature control oven (7.3); control the temperature at 185 °C ± 5 °C and heat for 15 h.

9.3.2 After cooling, open the lid, and take out the inner tank; use water to transfer the solution in the inner tank together with the precipitate into a 10 mL graduated test tube (7.6); use water to dilute to the mark; shake well.

9.3.3 Leave to settle or use a centrifuge (7.4) to centrifuge. This is the test portion solution.

9.4 Determination

Start the instrument according to the conditions specified in the instrument operating instructions. For working conditions, see Table A.1 in Appendix A. Select the analysis element isotope and the internal standard element isotope. The relevant isotopes are shown in Table A.2. Prepare the sample analysis table. The instrument shall be stable for at least 30 minutes after ignition. Use the mixed mass spectrometry tuning solution (6.4) to optimize the instrument parameters. Introduce the rhenium internal standard solution (6.9) online through the tee; measure the concentration values of the iodine element in the calibration solution (6.8), full reagent blank solution (9.1), standard material solution (9.2) and test portion solution (9.3.3) respectively.

9.5 Drawing of the calibration curve

Taking the mass concentration of iodine element in the calibration solution series as the abscissa and the relative intensity of the mass spectrum signals of iodine element and internal standard element as the ordinate, establish a calibration curve. Find the concentration value of iodine element in the test portion solution from the calibration curve.

10 Test data processing

The content of iodine (w_i) to be measured in the sample is measured as mass fraction [in micrograms per gram (μg/g)], and calculated according to Formula (1):

$$w_i = \frac{(\rho_i - \rho_{i0})V_0}{[1 - w(H_2O^-)]m_i \times 1\ 000} \quad \cdots\cdots\cdots\cdots (1)$$

Where:

ρ_i – the mass concentration of the component to be measured in the test portion measurement solution, in nanograms per milliliter (ng/mL);

ρ_{i0} – the mass concentration of the component to be measured in the blank test solution (9.1), in nanograms per milliliter (ng/mL);

V_0 – the total volume of the test portion solution, in milliliters (mL);

$w(H_2O^-)$ – the mass fraction of water adsorbed by the sample, calculated as percentage (%);

m_i – test portion mass, in grams (g).

Express the results obtained in accordance with the provisions of GB/T 14505-2010: ××× μg/g, ××.× μg/g, ×.×× μg/g, 0.×× μg/g.

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