

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

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Replacing GB/T 11225-1989; GB/T 11219.1-1989; GB/T 11219.2-1989

Radiochemical analysis of plutonium in water and soil samples

水和土壤样品中钚的放射化学分析方法

(English Translation)

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Table of Contents

Preface

1 Scope of application

2 Normative reference documents

3 Extraction chromatography

3.1 Principle of the method

3.2 Reagents and materials

3.3 Instruments and equipment

3.4 Samples

3.5 Analysis Step

3.6 Results Calculation

3.7 Determination of full radiochemical recovery of plutonium

3.8 Method validation

4 Ion exchange method

4.1 Principle of the method

4.2 Reagents and Materials

4.3 Instruments and equipment

4.4 Samples .9

4.5 Analysis steps

4.6 Results Calculation

Foreword

In order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Radioactive Pollution, Human health, regulate environmental monitoring methods, the development of this standard.

This standard specifies the method for the determination of plutonium in water and soil samples.

This standard is the "analysis method of plutonium in water" (GB 11225-89), "determination of plutonium in soil - extraction chromatography" (GB 11219.1-89), "determination of plutonium in soil - ion exchange method" (GB 11219.2-89) three standards of the integrated revision, the use of the analysis Principle and the original standard is basically the same.

The three standards were first released in 1989, the original standard drafting unit for the China Atomic Energy Research Institute. This is the first revision.

The main contents of the amendment are as follows.

- the integration of the three original standards, merged into a standard;
- the contents of the original standard on the sampling part are deleted;
- increased individual steps in the pretreatment and analysis of water and soil samples;
- Added ^{242}Pu as a tracer to determine the full radiochemical recovery rate;
- increased the requirements for blank experiments;
- Added instructions for the use of the sample amount and the chemical yield indicator.

Since the implementation of this standard, the former State Environmental Protection Agency approved on March 16, 1989, issued three environmental protection standards "plutonium in water (GB 11225-89), "Determination of plutonium in soil - Extraction chromatography" (GB 11219.1-89), "plutonium in soil of the determination - ion exchange method "(GB 11219.2-89) abolished.

Appendix A and Appendix B of this standard are informative.

This standard is organized by the Ministry of Environmental Protection Nuclear Safety Management Division, Science and Technology Standards Division.

The main drafting of this standard. Ministry of Environmental Protection Radiation Environmental Monitoring Technology Center (Zhejiang Province, radiation environmental monitoring station).

The environmental protection department of this standard approved on October 12, 2016.

This standard has been implemented since November 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Radiochemical analysis of plutonium in water and soil samples

1 Scope of application

This standard specifies the radiochemical analysis methods for the determination of plutonium in groundwater, surface water and soil. Nuclear industry in wastewater the determination of plutonium may be referred to the present method.

The extraction chromatography method in this standard is suitable for the determination of plutonium in water and soil samples. The ion exchange method is suitable for soil Determination and determination of plutonium in.

The minimum detectable limit of this method is water sample $1 \times 10^{-5}\text{Bq/L}$, soil type $1.5 \times$

10-5Bq/g.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

GB 12997 water quality sampling program design technical specifications Technical Guidance for Water Quality Sampling GB/T 6379 Accuracy of measurement methods and results (accuracy and precision) Technical specification for preservation and management of samples for water quality sampling

3.1 Principle of the method

The pretreated samples were prepared into 6 ~ 8mol/L HNO₃ sample solution. After reduction, oxidation regulates the valence of plutonium After plutonium with Pu (NO₃)⁵⁻ or Pu (NO₃)⁶²⁻ Anionic form is present in the solution. With tri-n-octylamine-polychloroethyl chloride the enriched powder extraction layer adsorbs plutonium and is rinsed with hydrochloric acid and nitric acid to further purify plutonium. Dissolve with oxalic acid-nitric acid mixed solution.

The source of the electrodeposition is carried out at low acidity. Finally, the activity of plutonium was measured with a low background alpha spectrometer.

3.2 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents conforming to national standards are used in the analysis, and the test water is freshly prepared.

Ionized water or distilled water.

3.2.1

anhydrous calcium chloride. the content of not less than 96.0%.

3.2.2

magnesium chloride. the content of not less than 97.0%.

3.2.3

Ammonium sulfonate. HO · SO₂ · NH₂, the content of not less than 99.5%.

3.2.4

reduced iron powder. the content of not less than 97.0%.

3.2.5

sodium nitrite. the content of not less than 99.0%.

3.2.6

oxalic acid. $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, the content of not less than 99.8%.

3.2.7

Polychlorotrifluoroethylene powder. irradiation synthesis, 40 to 60 mesh.

3.2.8

nitric acid. mass fraction of 65.0% ~ 68.0%.

3.2.9 Hydrochloric acid. mass fraction of 36.0% ~ 38.0%

3.2.10 Ammonium hydroxide. mass fraction of 25.0% ~ 28.0%.

3.2.11 xylene. $\text{C}_6\text{H}_4(\text{CH}_3)_2$, the content of not less than 80.0%.

3.2.12 Ethanol. mass fraction of not less than 99.5%.

3.2.13 Hydrochloric acid. $c = 10 \text{ mol/L}$.

3.2.14 Nitric acid. (1 1).

3.2.15 nitric acid. $c = 3 \text{ mol/L}$.

3.2.16 nitric acid. $c = 0.1 \text{ mol/L}$.

3.2.17 Ammonium hydroxide. (1 1).

3.2.18 0.025 mol/L oxalic acid - 0.150 mol/L nitric acid solution.

3.2.19 sodium nitrite solution. $c = 4 \text{ mol/L}$.

3.2.20 Tri-n-octylamine (TOA). $[\text{CH}_3(\text{CH}_2)_7]_3\text{N}$, the content of not less than 95.0% to 99.0%.

3.2.21 Ferrous sulfamate solution

Weigh 3.0g of reduced iron powder (3.2.4) and 12.0 g of sulfamic acid (3.2.3), and dissolved about 40 mL of nitric acid (3.2.16) Solution, filtered to remove insoluble matter, the filtrate diluted with nitric acid (3.2.16) to 50mL brown volumetric flask, stored in the refrigerator, spare. The use of not more than 30d.

3.2.22 hydroiodic acid. mass fraction of not less than 45.0%.

3.2.23 0.4mol/L hydroiodic acid - 6.0mol/L hydrochloric acid solution.

3.2.24 precision test paper. measuring range of $\text{pH} = 0.5 \sim 5.0$.

3.2.25 sulfuric acid. mass fraction of 95.0% ~ 98.0%.