Analytical Methods for Cesium-137 Beta Counting in Sediment

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Abstract

This study established a beta counting method for Cesium-137 in environmental fallout through digestion system selection experiments, ammonium phosphomolybdate adsorption system selection experiments, nitric acid-ammonium chloride wash solution substitution experiments, and interference elimination experiments, referencing the instrument calibration and sample precipitation source preparation methods in HJ 816-2016, combined with sample measurement using a low-background alpha/beta counter. Comparative analysis of fallout samples using the method established in this study and GB/T 16145-2022 showed no significant difference. Method validation demonstrated that both precision and accuracy satisfied the relevant requirements of the 'Technical Specifications for Radiation Environmental Monitoring' (HJ 61-2021). Currently, China lacks method standards for the beta counting method of Cesium-137 in fallout, and this study can provide valuable technical reference for the construction of the standard system for this method.

Full Text

Research on Analytical Method for Cesium-137 in Fallout Using Beta Counting Method

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Abstract

This study established a beta counting method suitable for quantitative analysis of Cesium-137 in environmental fallout samples. The methodology development involved systematic experiments, including digestion system selection, ammonium phosphomolybdate adsorption system optimization, evaluation of nitric acid-ammonium chloride washing solution alternatives, and interference elimination. Instrument calibration and sample precipitate source preparation followed the procedures outlined in HJ 816-2016. Sample radioactivity was measured using a low-background α/β counter. Validation of the developed method was conducted through comparative analysis of fallout samples against the standard gamma spectrometry method GB/T 16145-2022, yielding results with no statistically significant difference. Method performance verification demonstrated that both precision and trueness meet the requirements stipulated in the Technical Specifications for Radiation Environmental Monitoring (HJ 61-2021). Currently, China lacks a standardized method for Cesium-137 determination in fallout samples using beta counting. This work provides valuable technical reference for establishing a standardized beta counting methodology for Cesium-137 analysis in fallout within China.

Keywords: Cesium-137; Fallout; Beta counting method

Cesium-137 is an important artificial radionuclide that undergoes β^- decay, releasing two β particles with energies of 0.512 MeV (94.0%) and 1.17 MeV (6.0%). The decay product is Barium-137m, which subsequently releases a γ-ray with energy of 0.662 MeV through isomeric transition [1]. Cesium-137 has chemical properties similar to potassium, an essential element for life, and can easily enter the human body through the food chain, primarily accumulating in bone and muscle tissues. With a relatively long physical half-life (30.17 years) and biological half-life (approximately 100 days), the high-energy β and γ radiation emitted by Cesium-137 can cause inflammatory lesions in tissues and organs, and may even lead to genetic mutations causing cancer or hereditary effects. HJ 61-2021 explicitly requires monitoring of Cesium-137 in fallout, and annual composite samples in national radiation environmental quality monitoring must be analyzed for Cesium-137 using beta counting, making it one of the important objects of radiation environmental monitoring [2].

Currently, China lacks a standardized analytical method for Cesium-137 beta counting in fallout. The related method “Gamma-ray spectrometry method for the determination of radionuclides in environmental and biological samples” (GB/T 16145-2022) has a relatively high detection limit, making accurate measurement of low-activity-concentration Cesium-137 in fallout difficult and insufficient to meet the quality assurance requirements of national radiation environmental monitoring [3]. This study investigated ash samples of fallout collected from northeastern, northwestern, and southern China regions after volume reduction, concentration, and calcination at 450°C for 2 hours. Through systematic experiments including digestion system selection, ammonium phosphomolybdate adsorption system optimization, nitric acid-ammonium chloride washing solution substitution, and interference elimination, and by referencing the instrument calibration and sample precipitate source preparation methods in HJ 816-2016 [4] combined with low-background α/β measurement, we established a beta counting method suitable for environmental fallout Cesium-137. Method comparison and validation were also conducted, providing technical reference for the establishment of a standardized beta counting methodology for fallout Cesium-137 in China.

1.1 Instruments and Equipment

Low-background α/β counter (instrument performance meeting Grade II or higher requirements of GB/T 11682); High-purity germanium γ spectrometer; Analytical balance (division value 0.1 mg); Electric thermostatic drying oven; Muffle furnace (capable of maintaining constant temperature at 450$±$10°C); Electric stirrer; Temperature-adjustable electric hot plate; G4 glass sand core funnel; Detachable funnel; Porcelain evaporating dish or polytetrafluoroethylene beaker; and other common laboratory instruments and equipment.

1.2 Reagents and Consumables

30% hydrogen peroxide, nitric acid, hydrochloric acid, hydrofluoric acid, perchloric acid, glacial acetic acid, anhydrous ethanol, ammonium phosphomolybdate hydrate, aqua regia, citric acid, sodium hydroxide, sodium iodide, bismuth iodide, ammonium chloride, ammonium nitrate, cesium chloride, certified reference materials for Cesium-137 and potassium chloride, etc. All chemical reagents used were analytical grade or superior purity meeting national standards, and experimental water was freshly prepared deionized water.

2.1 Digestion System Selection Experiment

Based on Cesium-137 analysis standards and literature regarding digestion systems for solid materials such as surface soil [4-7], five different systems were selected for two types of digestion system selection experiments: concentrated nitric acid, 6 mol/L hydrochloric acid, concentrated nitric acid + hydrogen peroxide, aqua regia + hydrogen peroxide, and concentrated nitric acid + hydrofluoric acid + perchloric acid. The first experiment evaluated digestion effectiveness by measuring metal ion dissolution rates, while the second experiment validated Cesium-137 digestion effectiveness using IAEA surface soil reference material.

(1) Metal Ion Leaching Rate Experiment

Approximately 0.8 g (accurate to 0.1 mg) of fallout ash sample was placed in a clean porcelain evaporating dish or polytetrafluoroethylene beaker (for systems containing hydrofluoric acid), moistened slightly with deionized water, and digestion solution was added slowly. For single-acid systems, 20 mL of digestion solution was added; for concentrated nitric acid + hydrogen peroxide system, 15 mL concentrated nitric acid and 5 mL hydrogen peroxide were added; for aqua regia + hydrogen peroxide system, 15 mL aqua regia and 5 mL hydrogen peroxide were added; for concentrated nitric acid + hydrofluoric acid + perchloric acid system, 10 mL concentrated nitric acid, 5 mL hydrofluoric acid, and 5 mL perchloric acid were added. After slow addition, the mixture was evaporated to dryness on a low-temperature hot plate. The sample in the evaporating dish was then calcined in a muffle furnace at 450°C for 2 hours (samples treated with hydrofluoric acid were not calcined). The residue after calcination or hydrofluoric acid digestion was leached with 50-80 mL of 1.5 mol/L nitric acid solution and filtered. The filtrate was retained, diluted to 100 mL, and potassium and sodium contents were measured. Simultaneously, potassium and sodium contents in the original fallout ash sample were measured. The leaching rates of potassium and sodium ions after digestion were calculated by comparing their concentrations in the digestion solution and original ash sample, indirectly assessing the leaching rate of trace cesium in the same group.

(2) Cesium-137 Digestion Effectiveness Validation Experiment
To further validate Cesium-137 digestion effectiveness, IAEA surface moss soil reference material containing Cesium-137 (Chernobyl accident-contaminated nuclear power plant vicinity surface soil) was used as the test sample (note: no suitable fallout Cesium-137 reference material is available domestically). The same five digestion systems were employed as in the metal ion leaching experiment. The IAEA soil sample was calcined at 450°C for 2 hours, and a precise amount was placed in an evaporating dish or polytetrafluoroethylene beaker (for hydrofluoric acid systems), moistened with deionized water, and 1.00 mL of cesium carrier was added before digestion solution was added slowly. The mixture was evaporated to dryness on a low-temperature hot plate and calcined at 450°C for 2 hours (hydrofluoric acid systems were not calcined). The residue was leached twice with 50-80 mL of 1.5 mol/L nitric acid solution with heating, filtered while hot, and the filtrate was retained in a 250 mL beaker. One gram of ammonium phosphomolybdate was added, stirred for approximately 30 minutes, and left to stand for over 1 hour. Following the analytical procedure in HJ 816-2016, Cesium-137 activity concentration was measured as cesium bismuth iodide precipitate. The relative error between measured and reference values was compared across the five acid systems.

2.2 Ammonium Phosphomolybdate Adsorption System Selection Experiment

Studies by Gu Ruomei and Sha Lianmao [8,9] demonstrated that ammonium phosphomolybdate can quantitatively adsorb cesium under both nitric acid and hydrochloric acid conditions, with minimal constraint from acidic conditions. Even in concentrated nitric acid solution, the adsorption rate of Cesium-137 exceeds 90%, enabling direct cesium adsorption in nitric acid leachate. Based on literature review, an ammonium phosphomolybdate adsorption system selection experiment was conducted. The experimental method involved measuring 100 mL of 0.1 mol/L, 1 mol/L, 1.5 mol/L, 3 mol/L, 6 mol/L, and 9 mol/L nitric acid solutions into separate 250 mL beakers, accurately adding 1.00 mL of cesium carrier (22.37 mg cesium) to each, stirring for approximately 30 minutes, and allowing to stand for over 1 hour. The mixture was filtered using a G4 glass sand core funnel, the container was washed with nitric acid solution of the corresponding concentration, the filtrate was discarded, and the precipitate was retained. Following steps 8.2-8.5 in HJ 816-2016, the weight of cesium bismuth iodide was measured and the chemical recovery rate of cesium was calculated.

2.3 Nitric Acid-Ammonium Chloride Washing Solution Substitution Experiment

Ammonium ion (NH_4^+) has similar ionic radius and hydration energy to potassium ion (K^+) and can displace potassium ions from resins or adsorbents through competitive exchange reactions. Research indicates that washing ammonium phosphomolybdate with ammonium nitrate solution can remove potassium interference, though high concentrations may cause cesium loss. Using 30 mL of 0.25 mol/L ammonium nitrate to wash ammonium phosphomolybdate reduces potassium adsorption from 8$×10^{-3}$ to 2$×10^{-3}$ while causing minimal cesium loss [10]. HJ 816-2016 also employs nitric acid-ammonium nitrate washing solution at approximately 0.1 mol/L concentration. However, ammonium nitrate is currently classified as an explosive precursor chemical with restricted purchase and use. To verify whether washing solution is necessary to remove potassium interference in fallout Cesium-137 beta counting analysis, we first analyzed potassium and cesium contents in fallout ash samples collected from northeastern, northwestern, and southern China regions using the YJ/T0568-2020 method to discuss and validate the necessity of washing solution. Subsequently, experiments substituting nitric acid-ammonium chloride for nitric acid-ammonium nitrate washing solution were conducted.

The nitric acid-ammonium chloride substitution experiment involved: (1) In 50 mL of 1.5 mol/L nitric acid solution, 60 mg potassium carrier and 22.34 mg cesium carrier were added, followed by 1 g ammonium phosphomolybdate. After stirring for approximately 30 minutes and standing for over 1 hour, the mixture was filtered using a G4 glass sand core funnel. The ammonium phosphomolybdate was washed with 50 mL of 0.1 mol/L, 0.15 mol/L, or 0.25 mol/L nitric acid-ammonium chloride or nitric acid-ammonium nitrate solution, and the washing solution was retained. After diluting the washing solution 50,000-fold, potassium ion content was measured to verify potassium washing effectiveness. (2) In 50 mL of 1.5 mol/L nitric acid solution, 720 mg potassium carrier and 22.34 mg cesium carrier were added with 1 g ammonium phosphomolybdate. After stirring and standing, the mixture was filtered, and the ammonium phosphomolybdate was washed with 50 mL of 0.1 mol/L nitric acid-ammonium chloride or nitric acid-ammonium nitrate solution. The washing solution was retained, diluted 1,000-fold, and cesium content was measured to verify cesium loss after washing.

2.4 Interference Elimination Experiment

Analysis of representative fallout samples from Changchun (northeastern region), Gansu and Xi'an (northwestern region), and Yangjiang (Guangdong region) revealed the main metal ion contents shown in Table 6 . Although contents varied slightly, they were generally stable, with elements in descending order of concentration: iron, calcium, aluminum, magnesium, sodium, potassium, soluble silicon, rubidium, and cesium.

The iron interference elimination experiment involved: (1) In 100 mL of 1.5 mol/L nitric acid solution, specific amounts of cesium (20.84 mg) and iron carriers (60 mg, 120 mg, 200 mg, 500 mg) were added with Cesium-137 standard solution and 1 g ammonium phosphomolybdate. Following HJ 816-2016, cesium bismuth iodide precipitate was formed and measured using a low-background α/β counter. (2) In 100 mL of 1.5 mol/L nitric acid solution, cesium (20.84 mg) and iron (60 mg) carriers were added with 0.2035 Bq Cesium-137 standard solution. The pH was adjusted to 8-9 using 2 mol/L sodium hydroxide, heated to near boiling, and filtered while hot. The filtrate was collected, pH was adjusted to approximately 1 with nitric acid, 1 g ammonium phosphomolybdate was added, and cesium bismuth iodide precipitate was formed and measured.

The potassium interference elimination experiment involved dissolving a specific amount of high-purity KCl standard powder in 100 mL water, adding 1.00 mL cesium carrier solution and appropriate Cesium-137 standard solution, and mixing uniformly. One gram of ammonium phosphomolybdate was added and stirred for 30 minutes. The mixture was filtered using a G4 glass sand core funnel, the container was washed with nitric acid-ammonium chloride washing solution, and filtrate A was retained. Potassium-40 and Cesium-137 contents in filtrate A were measured using a γ spectrometer. The precipitate was dissolved in sodium hydroxide solution to collect dissolution liquid B, and Potassium-40 and Cesium-137 contents in dissolution liquid B were measured to determine the amounts adsorbed on ammonium phosphomolybdate precipitate.

2.5 Method Comparison Experiment

The developed fallout Cesium-137 beta counting method was compared with GB/T 16145-2022. The comparison samples used fallout ash as the matrix, with Cesium-137 powder reference material purchased from the China Institute of Atomic Energy as the standard source. Matrix spiking was employed to obtain seven sample groups with activity levels ranging from 0.12 Bq/g to 2.67 Bq/g. Both the developed beta counting method and the gamma spectrometry method of GB/T 16145-2022 were used to analyze and measure the seven sample groups, yielding seven paired measurement datasets for Cesium-137. Significance testing of the paired data was conducted according to HJ 168-2020 requirements.

2.6 Method Validation Experiment

Fallout samples with activity concentrations of 0.044 Bq/g, 0.12 Bq/g, and 2.60 Bq/g were used as uniform samples. Each activity level was analyzed in six parallel measurements to calculate relative standard deviation and verify method precision. Two fallout reference samples with reference values of 0.44 Bq/g and

9.9 Bq/g were used as validation samples, with six parallel measurements at each activity level to calculate spiked recovery rates and verify method trueness. The low-background α/β counter was used to measure blank disk background count rates. Using typical conditions (sampling time 180 days, sample collection area 0.25 m^2, measurement time 1440 minutes, chemical recovery rate 80%), the detection limit was calculated according to the detection limit formula.

2.2 Calculation Methods

The activity concentration of Cesium-137 in fallout was calculated using formula (1):

$$A = \frac{(N_x - N_b) \times 60}{m \times E_x \times Y_{Cs}} \tag{1}$$

where:
- A = Cesium-137 activity concentration, $\mathrm{Bq/(m^2 \cdot d)}$ or Bq/g
- N_x = sample count rate, min^{-1}
- N_b = background count rate, min^{-1}
- 60 = conversion coefficient from seconds to minutes, s/min
- m = sample amount, $\mathrm{m^2 \cdot d}$ or g
- E_x = instrument detection efficiency, obtained from Cesium-137 detection efficiency-mass curve, %
- Y_{Cs} = cesium chemical recovery rate, %

The detection limit for fallout Cesium-137 was calculated using formula (2):

$$MDC = \frac{2.78 + 3.29 \times \sqrt{N_b \times t_b}}{t_b \times m \times E_x \times Y_{Cs}} \times 60 \tag{2}$$

where:
- MDC = detection limit, $\mathrm{Bq/(m^2 \cdot d)}$ or Bq/g
- t_b = background measurement time, min
- Other symbols as defined in formula (1).

3.1 Digestion System Selection Experiment

(1) Metal Ion Leaching Rate Experiment

As shown in Figure 1 [Figure 1: see original paper], the leaching rates of potassium and sodium ions in the concentrated nitric acid + hydrofluoric acid + perchloric acid system exceeded 90%, while the other four digestion systems achieved leaching rates above 68.5%. Based solely on potassium or sodium leaching rates, no system was clearly superior. However, judging by the average leaching rates of potassium and sodium, the aqua regia + hydrogen peroxide system ranked second to the hydrofluoric acid-containing system but performed slightly better than the other three systems.

(2) Cesium-137 Digestion Effectiveness Validation Experiment

Table 1 shows that the concentrated nitric acid + hydrofluoric acid + perchloric acid and aqua regia + hydrogen peroxide systems produced relative errors of -7.62% to 8.54%, all less than 10%, while single-acid systems showed incomplete digestion. Subsequent literature review and investigation of domestic provincial radiation monitoring institutions revealed that hydrofluoric acid use is restricted in some institutions, and perchloric acid is classified as an explosive precursor chemical. Therefore, when equally effective alternatives are available, their use should be avoided. In conclusion, the aqua regia + hydrogen peroxide system is suitable as the digestion system for fallout Cesium-137 beta counting method.

3.2 Ammonium Phosphomolybdate Adsorption System Selection Experiment

Table 2 demonstrates that ammonium phosphomolybdate can quantitatively adsorb cesium across different nitric acid concentrations, with minimal impact on chemical recovery rate. Considering literature findings and the adsorption acid system used in HJ 816-2016 ((1+9) nitric acid solution) [4], a 1.5 mol/L nitric acid solution is selected as the ammonium phosphomolybdate adsorption system for fallout Cesium-137 beta counting analysis.

3.3 Nitric Acid-Ammonium Chloride Washing Solution Substitution Experiment

The distribution coefficients of ammonium phosphomolybdate are 6000 for cesium and 3.4 for potassium [10]. Table 3 shows that potassium content in representative fallout samples from Changchun (northeastern region), Gansu and Xi'an (northwestern region), and Yangjiang (Guangdong region) exceeds cesium content by 10^4 times. Considering the distribution coefficients and the fact that potassium content far exceeds cesium content in fallout, using washing solution to reduce potassium interference is necessary when ammonium phosphomolybdate is employed as the adsorbent for fallout Cesium-137 analysis.

Table 4 shows that both nitric acid-ammonium chloride and nitric acid-ammonium nitrate washing solutions can wash certain amounts of potassium from ammonium phosphomolybdate, with similar washing effectiveness across different concentrations. Table 5 indicates that cesium loss rates after washing ammonium phosphomolybdate adsorbed with cesium carrier are within 3% for both washing solutions.

In conclusion, based on the theory of ammonium ion displacing potassium ion and experimental verification, substituting nitric acid-ammonium chloride washing solution for nitric acid-ammonium nitrate is feasible. A 0.1 mol/L nitric acid-ammonium chloride washing solution is selected as the detergent.

3.4 Interference and Elimination

Analysis revealed that representative fallout samples from Changchun, Gansu, Xi'an, and Yangjiang mainly contained the metal elements listed in Table 6 , with slight variations but generally stable concentrations. Elements in descending order of concentration were iron, calcium, aluminum, magnesium, sodium, potassium, soluble silicon, rubidium, and cesium.

Table 7 shows that within the experimental range, iron ion addition did not significantly affect Cesium-137 chemical recovery or spiked recovery rates. For example, in sample E, the iron removal step did not affect Cesium-137 spiked recovery but slightly reduced cesium chemical recovery. In conclusion, iron ions did not significantly interfere with the Cesium-137 beta counting method, and iron interference can be considered negligible. However, in actual sample analysis, when the ash content is large and iron concentration is high, an iron removal step may be considered to reduce iron ion influence.

Table 8 shows that 99.8% of added Potassium-40 remained in filtrate A after enrichment and separation, indicating effective separation of potassium and cesium after ammonium phosphomolybdate enrichment and nitric acid-ammonium chloride washing, with good removal of Potassium-40 interference.

Literature reports distribution coefficients of ammonium phosphomolybdate as 6000 for cesium, 230 for rubidium, 3.4 for potassium, and 0 for sodium [10]. Coexisting aluminum, sodium, calcium, magnesium, and nickel have minimal effect on cesium adsorption by ammonium phosphomolybdate [11]. Table 6 shows rubidium content in fallout is double that of cesium. Theoretically, with a distribution coefficient 26 times higher for cesium than rubidium and the addition of stable cesium carrier (approximately 20 mg) in beta counting analysis, cesium content during analysis exceeds rubidium by at least four orders of magnitude. Considering both concentration and distribution coefficient differences, rubidium interference is negligible in fallout Cesium-137 beta counting analysis.

3.5 Method Comparison Experiment

Method comparison results are shown in Table 9 . According to the method recommended in Appendix B of HJ 168-2020, significance testing of the paired measurement results [12] yielded t = -1.15 and P = 0.295. Based on degrees of freedom (df = 6) and significance level $\alpha = 0.05$, the critical value from the t-distribution table is 2.447, indicating no statistically significant difference between the two analytical methods.

3.6 Method Validation Experiment

Method precision measurement results are shown in Table 10 , and trueness measurement results in Table 11 . The relative standard deviation from six parallel measurements ranged from -13.9% to 6.14%, and spiked recovery rates ranged from 90.3% to 101%, all meeting the control index requirements in HJ 61-2021.

The detection limit of this method depends on sample amount, detection efficiency, background count rate, and measurement time. Using typical conditions (sampler collection area 0.25 m^2, sampling period 180 days, measurement time 1440 minutes with a low-background α/β counter with 50 mm detector sensitive area diameter, and chemical recovery rate of 80%), typical detection limit values are shown in Table 12 . The minimum typical detection limit is 1.2×10^{-4} Bq/(m^2 · d).

Conclusion

Through digestion system selection, ammonium phosphomolybdate adsorption system optimization, nitric acid-ammonium chloride washing solution substitution, and interference elimination experiments, and considering restrictions on hazardous chemicals such as hydrofluoric acid and ammonium nitrate, we established a fallout Cesium-137 beta counting method. The method employs aqua regia and hydrogen peroxide for joint digestion of fallout ash samples, selective cesium adsorption by ammonium phosphomolybdate in 1.5 mol/L nitric acid system, nitric acid-ammonium chloride solution as washing agent, instrument calibration and sample precipitate source preparation according to HJ 816-2016, and sample measurement using low-background α/β counter.

This study confirmed that iron ions did not significantly interfere with Cesium-137 beta counting, and potassium interference can be eliminated through enrichment, separation, and nitric acid-ammonium chloride washing. In actual analysis, when fallout sample ash content is large and iron concentration is high, an iron removal step may be added to reduce iron influence. Literature review indicates that interference from rubidium, sodium, calcium, etc., is also minimal in this method.

The method established in this work showed no significant difference when compared with GB/T 16145-2022 for fallout sample analysis. Method precision and trueness meet the requirements of Technical Specifications for Radiation Environmental Monitoring (HJ 61-2021). Under typical conditions, the detection limit can reach 1.2×10^{-4} Bq/(m^2 · d).

Currently, China lacks a standardized method for fallout Cesium-137 beta counting. This work provides valuable technical reference for establishing a standardized beta counting methodology for Cesium-137 analysis in fallout.

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Note: Figure translations are in progress. See original paper for figures.