Study on Analytical Methods for Cesium-137 Beta Counting in Fallout

Authors: Zhen Liying

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Abstract

This study established a β-counting method for cesium-137 in fallout by conducting digestion system selection experiments, ammonium phosphomolybdate adsorption system selection experiments, nitric acid-ammonium chloride detergent substitution experiments, interference elimination experiments, etc., referencing the instrument calibration and cesium bismuth iodide precipitation source preparation methods in HJ 816-2016, combined with low-background α/β measuring instruments for sample measurement. Researchers used this method to conduct comparative analysis with GB/T 16145-2022 on fallout samples, and the method comparison results showed no significant difference between the two. In method validation experiments, the precision and trueness of this method respectively met the requirements in HJ 61-2021 for parallel sample relative deviation ($ $30%) and spiked recovery rate (80%~120%). Given that there is currently a lack of analytical method standards for β-counting of cesium-137 in fallout domestically, this study can provide technical reference for the construction of relevant standard systems.

Full Text

Study on the Analytical Method for Beta Counting of Cesium-137 in Fallout

ZHEN Liying, QIN Hao, ZHU Shenhe, HE Gaopeng, LIANG Guoshuai, LI Meili, LI Qiaoqin, WANG Jiayue, LIN Shuqian
Guangdong Environmental Radiation Monitoring and Nuclear Emergency Response Technical Support Center, Guangzhou 510305, China

Abstract

This study establishes a reliable beta counting method for cesium-137 (Cs-137) detection in fallout samples through systematic experiments on digestion system selection, ammonium phosphomolybdate adsorption optimization, nitric acid-ammonium chloride detergent substitution, and interference elimination. Drawing upon the instrument calibration and cesium iodobismuthate precipitation source preparation protocols specified in HJ 816-2016, and employing low-background alpha/beta counting instruments for sample measurement, the developed method was compared against GB/T 16145-2022 using actual fallout samples, revealing no statistically significant differences between the two approaches. Method validation experiments demonstrated that both precision (relative deviation of parallel samples $ $30%) and accuracy (spiked recovery rates of 80%~120%) meet the requirements stipulated in HJ 61-2021. Given the current absence of a dedicated analytical standard for Cs-137 beta counting in fallout samples in China, this research provides valuable technical references for developing relevant standardization frameworks.

Keywords: Cesium-137; Fallout; Beta counting method

Cesium-137 is a significant artificial radionuclide originating from nuclear fuel cycle activities and nuclear technology applications. It undergoes beta decay, releasing two beta particles with energies of 0.512 MeV (94.0%) and 1.17 MeV (6.0%), decaying to barium-137m, which subsequently releases a 0.662 MeV gamma ray through isomeric transition [1~5]. With chemical properties similar to the essential element potassium, Cs-137 readily enters the human body through the food chain and primarily accumulates in bone and muscle tissues. Its relatively long physical half-life (30.17 years) and biological half-life (approximately 100 days), combined with high-energy beta and gamma emissions, can cause inflammatory lesions in tissues and organs, and potentially induce genetic mutations leading to cancer or hereditary effects. HJ 61-2021 explicitly requires monitoring of Cs-137 in fallout, and national radiation environmental quality monitoring mandates annual composite samples to be analyzed using beta counting methods, making it a critical target in radiation environmental monitoring [6].

Currently, China lacks a dedicated analytical standard for the beta counting method of Cs-137 in fallout. The relevant method "Gamma-ray spectrometry method for the determination of radionuclides in environmental and biological samples" (GB/T 16145-2022) has relatively high detection limits, making accurate measurement of low-activity-concentration Cs-137 in fallout difficult and insufficient to meet national radiation environmental quality monitoring quality assurance requirements [7]. Considering China’ s vast territory and potential regional variations in fallout composition due to climatic differences, this study utilized representative fallout samples from northeastern, northwestern, and southern China. Samples were reduced in volume, concentrated, and ashed at 450°C for 2 hours. Through digestion system selection experiments, ammonium phosphomolybdate adsorption system optimization, nitric acid-ammonium chloride detergent substitution experiments, and interference elimination studies, and by referencing the instrument calibration and cesium iodobismuthate precipitation source preparation methods in HJ 816-2016 [8] combined with low-background alpha/beta measurement instruments, a beta counting method suitable for environmental fallout Cs-137 analysis was established. Method comparison and validation studies were conducted, with results providing technical references for developing China’ s analytical standard system for fallout Cs-137 beta counting.

1.1 Instruments and Equipment

Low-background alpha/beta counter; high-purity germanium gamma spectrometer; electronic analytical balance; electric thermostatic drying oven; muffle furnace; electric stirrer; temperature-adjustable electric hot plate; G4 glass sand core funnel; detachable funnel; porcelain evaporating dish or polytetrafluoroethylene beaker; and other commonly used laboratory instruments and equipment.

1.2 Reagents and Consumables

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

2.1 Digestion System Selection Experiment

Based on Cs-137 analysis standards and literature for solid materials such as surface soils [8~11], five different digestion systems were selected: concentrated nitric acid + hydrofluoric acid + perchloric acid (System I), concentrated nitric acid + hydrogen peroxide (System II), aqua regia + hydrogen peroxide (System III), concentrated nitric acid (System IV), and 6 $\mathrm{mol \cdot L^{-1}}$ hydrochloric acid (System V). Two types of experiments were conducted: (1) Metal ion leaching rate assessment to evaluate digestion effectiveness by verifying sodium and potassium leaching rates after different digestion treatments, indirectly assessing the leaching efficiency of trace Cs-137 from the same group; and (2) Cs-137 digestion efficiency verification using IAEA surface soil reference material (batch number: IAEA-447, calibration certificate: IAEA-447 Rev.01), a post-Chernobyl surface moss soil containing known Cs-137 levels that had entered the biosphere cycle, providing more experimental representativeness than spiked standard materials.

(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; for aqua regia + hydrogen peroxide system, 15 mL aqua regia and 5 mL hydrogen peroxide; and for concentrated nitric acid + hydrofluoric acid + perchloric acid system, 10 mL concentrated nitric acid, 5 mL hydrofluoric acid, and 5 mL perchloric acid. After slow addition, samples were evaporated to dryness on a low-temperature hot plate, then ashed in a muffle furnace at 450°C for 2 hours (samples containing hydrofluoric acid were not subjected to additional ashing). The residue was leached with 50–80 mL of 1.5 $\mathrm{mol \cdot L^{-1}}$ nitric acid solution, filtered, and the filtrate was retained, diluted to 100 mL, and analyzed for potassium and sodium content. Simultaneous analysis of potassium and sodium in the original fallout ash samples allowed calculation of leaching rates to indirectly assess Cs-137 leaching efficiency.

(2) Cs-137 Digestion Efficiency Verification Experiment
To further verify Cs-137 digestion effectiveness, IAEA surface moss soil reference material containing Cs-137 was used. The same five digestion systems were applied. The IAEA reference material was ashed at 450°C for 2 hours, and precise aliquots were placed in evaporating dishes or polytetrafluoroethylene beakers, moistened with deionized water, spiked with 1.0 mL cesium carrier, and digestion solution was added slowly. After evaporation on a low-temperature hot plate, samples were ashed at 450°C for 2 hours (except hydrofluoric acid-treated samples). Residues were leached twice with 50–80 mL of 1.5 $\mathrm{mol \cdot L^{-1}}$ nitric acid solution, filtered while hot, and filtrates were retained in 250 mL beakers. After adding 1.0 g ammonium phosphomolybdate, stirring for approximately 30 minutes, and standing for over 1 hour, cesium iodobismuthate precipitation was performed following HJ 816-2016 procedures, and Cs-137 activity concentration was measured to compare relative errors between measured and reference values across the five acid systems.

2.2 Ammonium Phosphomolybdate Adsorption System Selection Experiment

Studies by Gu Ruomei and Sha Lianmao [12,13] demonstrate that ammonium phosphomolybdate quantitatively adsorbs cesium under both nitric and hydrochloric acid conditions with minimal constraint from acidic conditions, achieving over 90% Cs-137 adsorption even in concentrated nitric acid solutions. This enables direct cesium adsorption from sample nitric acid leachates. Based on literature review, adsorption system selection experiments were conducted using 100 mL nitric acid solutions at concentrations of 0.1, 1.0, 1.5, 3.0, 6.0, and 9.0 $\mathrm{mol \cdot L^{-1}}$ in 250 mL beakers, each spiked with 1.0 mL cesium carrier (22.4 mg cesium). After stirring for approximately 30 minutes and standing for over 1 hour, solutions were filtered through G4 glass sand core funnels, containers were washed with corresponding nitric acid concentrations, filtrates were discarded, and precipitates were retained. Following HJ 816-2016 procedures (sections 8.2–8.5), cesium iodobismuthate precipitates were weighed to calculate chemical recovery rates.

2.3 Nitric Acid-Ammonium Chloride Detergent Substitution Experiment

Ammonium ions (NH_4^+) have similar ionic radius and hydration energy to potassium ions (K^+) and can displace potassium from resins or adsorbents through competitive exchange reactions. Research indicates that washing ammonium phosphomolybdate with ammonium nitrate solution removes potassium interference, though high concentrations may cause cesium loss. Using 30 mL of 0.25 $mol \cdot L^{-1}$ ammonium nitrate to wash ammonium phosphomolybdate reduces potassium adsorption from 8×10^{-3} to 2×10^{-3} with minimal cesium loss [11]. HJ 816-2016 also employs approximately 0.1 mol/L nitric acid-ammonium nitrate detergent, but ammonium nitrate is classified as an explosive precursor chemical with restricted purchase and use. To verify the necessity of detergent use for potassium removal in fallout Cs-137 beta counting analysis, potassium and cesium contents in representative fallout samples from northeastern, northwestern, and southern China were first analyzed using YJ/T0568-2020 methods to assess detergent necessity. Subsequently, nitric acid-ammonium chloride detergent substitution experiments were conducted.

The substitution experiment involved: (1) In 50 mL of 1.5 $mol \cdot L^{-1}$ nitric acid solution, 60.0 mg potassium carrier and 22.3 mg cesium carrier were added with 1.0 g ammonium phosphomolybdate, stirred for 30 minutes, and stood for over 1 hour. The precipitate was filtered through a G4 glass sand core funnel and washed with 50 mL of 0.1, 0.15, or 0.25 $mol \cdot L^{-1}$ nitric acid-ammonium chloride or nitric acid-ammonium nitrate solutions. Wash solutions were retained, diluted 50,000-fold, and analyzed for potassium content to verify washing effectiveness. (2) In 50 mL of 1.5 $mol \cdot L^{-1}$ nitric acid solution, 720 mg potassium carrier and 22.3 mg cesium carrier were added with 1.0 g ammonium phosphomolybdate, processed similarly, and wash solutions were diluted 1,000-fold for cesium analysis to verify cesium loss during washing.

2.4 Interference and Elimination Experiments

Analysis of major metal ion contents in representative fallout samples from Changchun (northeast), Gansu and Xi’ an (northwest), and Yangjiang (south) was conducted based on literature review to investigate iron and potassium ion interference elimination. The iron interference elimination experiment involved: (1) In 100 mL of 1.5 $mol \cdot L^{-1}$ nitric acid solution, cesium carrier, iron carrier, and Cs-137 standard solution were added, followed by 1.0 g ammonium phosphomolybdate. After stirring and cesium iodobismuthate precipitation per HJ 816-2016, samples were measured using a low-background alpha/beta counter. (2) In 100 mL of 1.5 $mol \cdot L^{-1}$ nitric acid solution with cesium carrier, iron carrier, and Cs-137 standard solution, pH was adjusted to 8-9 using 2 $mol \cdot L^{-1}$ sodium hydroxide, heated near boiling, and filtered while hot. The filtrate was adjusted to pH 1 with nitric acid, 1.0 g ammonium phosphomolybdate was added, and cesium iodobismuthate precipitation was performed for measurement.

Potassium interference elimination experiments involved dissolving analytical-grade KCl powder in 100 mL water, adding 1.0 mL cesium carrier solution and appropriate Cs-137 standard solution, mixing uniformly, then adding 1.0 g ammonium phosphomolybdate. After 30 minutes stirring and filtration through a G4 glass sand core funnel with nitric acid-ammonium chloride wash solution, filtrate A was retained and analyzed for potassium-40 and Cs-137 content using gamma spectrometry. The precipitate was dissolved in sodium hydroxide solution to obtain solution B, which was similarly analyzed to determine potassium-40 and Cs-137 adsorbed on ammonium phosphomolybdate.

2.5 Method Comparison Experiment

The developed beta counting method was compared against GB/T 16145-2022 using fallout ash samples spiked with Cs-137 powder reference material (certificate number: GFJGJL1005250002865) from the China Institute of Atomic Energy. Seven sample groups with activity levels ranging from 0.12 to 2.67 $\mathrm{Bq} \cdot \mathrm{g}^{-1}$ were prepared through matrix spiking. Both methods were applied to analyze the seven sample groups, yielding paired measurement data. Significance testing of the seven paired datasets was conducted according to HJ 168-2020 requirements.

2.6 Method Validation Experiment

Three activity concentration levels of fallout samples (0.044, 0.12, and 2.60 $\mathrm{Bq} \cdot \mathrm{g}^{-1}$) were analyzed with six parallel measurements each to calculate relative standard deviations for precision validation. Two reference fallout samples (0.44 and 9.9 $\mathrm{Bq} \cdot \mathrm{g}^{-1}$) were analyzed with six parallel measurements each to calculate spike recovery rates for trueness validation. The method detection limit was verified under typical conditions (sampling period: 180 days, collection area: 0.25 m^2, measurement time: 1440 minutes, chemical recovery: 80%) by measuring blank background count rates with the low-background alpha/beta counter.

2.7 Calculation Methods

The activity concentration of Cs-137 in fallout is calculated using Equation (1):

$$A = \frac{(N_x - N_b) \times 60}{m \times E_x \times Y_{Cs} \times 100}$$

where:
A = Cs-137 activity concentration, $\mathrm{Bq} \cdot \mathrm{m}^{-2} \cdot \mathrm{d}^{-1}$ or $\mathrm{Bq} \cdot \mathrm{g}^{-1}$
N = sample count rate, min^{-1}
N = background count rate, min^{-1}
60 = conversion coefficient from seconds to minutes, $\mathrm{s} \cdot \mathrm{min}^{-1}$

m = sample amount, $m^2 \cdot d$ or g
E = instrument detection efficiency (obtained from Cs-137 efficiency-mass curve), %
$Y\_\{Cs\}$ = cesium chemical recovery, %

The detection limit for fallout Cs-137 is calculated using Equation (2):

$$MDC = ...$$

where:
MDC = minimum detectable concentration, $Bq \cdot m^{-2} \cdot d^{-1}$ or $Bq \cdot g^{-1}$
t = background measurement time, min
Other symbols are as defined in Equation (1).

3.1 Digestion System Selection Experiment Results

(1) Metal Ion Leaching Rate Experiment
As shown in [Figure 1: see original paper], potassium and sodium leaching rates exceeded 90% in the concentrated nitric acid + hydrofluoric acid + perchloric acid system, while the other four systems achieved leaching rates above 68.5%. Judging by the average of potassium and sodium leaching rates, the aqua regia + hydrogen peroxide system was slightly inferior to the hydrofluoric acid-containing system but superior to the other three systems.

(2) Cs-137 Digestion Efficiency Verification Experiment
Results in show that the concentrated nitric acid + hydrofluoric acid + perchloric acid and aqua regia + hydrogen peroxide systems produced relative errors of -7.93% to 8.54% (all <10%), while single-acid systems showed incomplete digestion. Subsequent investigations revealed that hydrofluoric acid use is restricted in some provincial radiation monitoring institutions, and perchloric acid is also an explosive precursor chemical. Therefore, when equally effective alternatives exist, their use should be avoided. In summary, the aqua regia + hydrogen peroxide system is most suitable for fallout Cs-137 beta counting analysis.

3.2 Ammonium Phosphomolybdate Adsorption System Selection Experiment Results

As shown in , ammonium phosphomolybdate quantitatively adsorbs cesium across different nitric acid concentrations, with minimal impact on chemical recovery rates. Considering literature review findings and the adsorption acid system specified in HJ 816-2016 ((1+9) nitric acid solution) [7], a 1.5 $mol \cdot L^{-1}$ nitric acid solution was selected as the optimal system for ammonium phosphomolybdate adsorption of Cs-137 in fallout beta counting analysis.

3.3 Nitric Acid-Ammonium Chloride Detergent Substitution Experiment Results

The distribution coefficient of ammonium phosphomolybdate is 6000 for cesium and 3.4 for potassium [14]. shows that potassium content in representative fallout samples from Changchun (northeast), Gansu and Xi’ an (northwest), and Yangjiang (south) exceeds cesium content by 10^4 times. Considering the distribution coefficients and the substantial potassium excess, detergent use remains necessary to reduce potassium interference in ammonium phosphomolybdate-based Cs-137 analysis.

demonstrates that both nitric acid-ammonium nitrate and nitric acid-ammonium chloride detergents effectively wash potassium from ammonium phosphomolybdate, with similar performance across different concentrations. shows that cesium loss rates remain below 3% after washing ammonium phosphomolybdate with either detergent. Therefore, based on theoretical and experimental verification of ammonium ion displacement of potassium, nitric acid-ammonium chloride detergent is a viable substitute, with 0.1 $\text{mol} \cdot \text{L}^{-1}$ nitric acid-ammonium chloride solution selected as the optimal washing solution.

3.4 Interference and Elimination

Analysis revealed that representative fallout samples from Changchun, Gansu, Xi’ an, and Yangjiang primarily contain the metal elements listed in (silicon refers to soluble silicon), with relatively stable concentrations varying slightly by region. Elemental concentrations from highest to lowest are: iron, calcium, aluminum, magnesium, sodium, potassium, soluble silicon, rubidium, and cesium.

Iron interference test results in indicate that iron addition within experimental ranges did not significantly affect Cs-137 chemical recovery or spike recovery rates. For example, in Experiment E, iron removal did not impact Cs-137 spike recovery but slightly reduced cesium chemical recovery. Therefore, iron ions do not significantly interfere with the Cs-137 beta counting method and may not require consideration. However, for actual fallout samples with large ash quantities and high iron content, iron removal steps may be considered to reduce potential interference.

As shown in , potassium-40 content in filtrate A after enrichment and separation accounted for 99.8% of the added amount, demonstrating effective potassium-cesium separation and excellent potassium-40 interference removal after ammonium phosphomolybdate enrichment and nitric acid-ammonium chloride washing.

Literature reports distribution coefficients of ammonium phosphomolybdate as 6000 for cesium, 230 for rubidium, 3.4 for potassium, and 0 for sodium [14]. Coexistence of aluminum, sodium, calcium, magnesium, and nickel has minimal impact on cesium adsorption [15]. shows rubidium content is twice that of cesium in fallout. Theoretically, ammonium phosphomolybdate' s cesium distribution coefficient is 26 times higher than rubidium' s, and beta counting analysis typically adds stable cesium carrier (~20 mg), making cesium content at least four orders of magnitude higher than rubidium. Considering both concentration and distribution coefficient differences, rubidium interference is negligible in fallout Cs-137 beta counting analysis.

3.5 Method Comparison Experiment Results

Method comparison results are presented in . Following the recommended method in Appendix B of HJ 168-2020, significance testing yielded t = -1.15 and P = 0.295. With degrees of freedom (df = 6) and significance level $\alpha = 0.05$, the critical value from the t-distribution table is 2.447, indicating no significant difference between the two analytical methods.

3.6 Method Validation Experiment Results

Precision and trueness measurement results are shown in and , respectively. Relative standard deviations from six parallel measurements ranged from -13.9% to 6.14%, with spike recovery rates of 90.3%~101%, meeting HJ 61-2021 control requirements (relative deviation of parallel samples $ $30%, spike recovery 80%~120%).

The method detection limit depends on sample quantity, detection efficiency, background count rate, and measurement time. Under typical conditions (sampler collection area: 0.25 m^2, sampling period: 180 days, detector sensitive area diameter: 50 mm, measurement time: 1440 minutes, chemical recovery: 80%), typical detection limit values are shown in , with a minimum detection limit of 1.2$×10^{-4}$ $\mathrm{Bq \cdot m^{-2} \cdot d^{-1}}$.

Conclusion

Based on systematic experiments for digestion system selection, ammonium phosphomolybdate adsorption optimization, nitric acid-ammonium chloride detergent substitution, and interference elimination, and considering practical restrictions on chemical reagents (ammonium nitrate as an explosive precursor, hydrofluoric acid restrictions in some institutions), a reliable beta counting method for fallout Cs-137 was established. The method employs aqua regia and hydrogen peroxide for joint digestion of fallout ash samples, selective cesium adsorption by ammonium phosphomolybdate in 1.5 $\mathrm{mol \cdot L^{-1}}$ nitric acid system, nitric acid-ammonium chloride solution as detergent, and follows HJ 816-2016 for instrument calibration and cesium iodobismuthate precipitation source preparation, combined with low-background alpha/beta counting for sample measurement.

The study confirmed that iron ions do not significantly interfere with Cs-137 beta counting, while potassium interference can be eliminated through enrichment, separation, and nitric acid-ammonium chloride washing. In practice, iron removal steps may be considered for samples with large ash quantities and high iron content. Literature review indicates that interference from rubidium, sodium, calcium, and other elements is also negligible. Comparative analysis with GB/T 16145-2022 showed no significant differences, while method precision and trueness met HJ 61-2021 requirements. The detection limit can reach 1.2$×10^{-4}$ $\mathrm{Bq} \cdot \mathrm{m}^{-2} \cdot \mathrm{d}^{-1}$ under typical conditions. As China currently lacks a dedicated analytical standard for fallout Cs-137 beta counting, these research findings provide valuable technical references for relevant standard system development.

Author Contributions

ZHEN Liying: research design, data compilation, and manuscript writing; QIN Hao, ZHU Shenhe, HE Gaopeng, LIANG Guoshuai, LI Meili, LI Qiaoqin: experimental analysis and data processing; WANG Jiayue, LIN Shuqian: sample collection and reference material management.

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