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Research on the Rapid Analysis Method of ^{90}Sr in Environmental Soil Samples

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Abstract. As an important fission product generated in nuclear reactors, ^{90}Sr is characterized by its high fission yield, long half-life, and tendency to enter the human body through the food chain, making it a key nuclide of concern in environmental radioactivity assessment. To enable rapid detection of ^{90}Sr in environmental samples under nuclear emergency conditions, a fast analytical method for ^{90}Sr in environmental soil samples was developed based on triple quadrupole inductively coupled plasma mass spectrometry (ICP-MS). The method involves extracting Sr from soil via acid leaching, separating it from the matrix through co-precipitation with $Ca(OH)_2$, and further purifying Sr using Sr resin chromatography. The ^{90}Sr content was then determined by ICP-MS under a mixed gas flow of 1.0 ml/min NH_3 and 2.0 ml/min He. This method achieved a detection limit of 4.3 mBq/kg for ^{90}Sr in soil samples, with a total analysis time of 10 hours. Compared to traditional radiometric methods requiring 3–5 days for analysis, it better meets the need for rapid ^{90}Sr analysis under nuclear emergency response conditions.

1 Introduction

^{90}Sr,a typical artificial radionuclide,has become a key focus in environmental radioactivity assessment due to its high nuclear fission reaction yield, long physical half-life (28.90y), and long biological half-cycle (7y)[1-3].

Table 1. The content of Sr-90 in soils of different regions.

Sampling locations	Concentration of ^{90}Sr (Bq/kg)
Northwest of the Fukushima Nuclear Power Plant	52.1-73.5[4]
Near the Chernobyl Nuclear Power Plant in Ukraine	2.38-6.88×10^{4}[5]
Ivankiv District, Kyiv, Ukraine	288[6]
Inner Mongolia, China	1.2-7.6[7]

To illustrate the significance of ^{90}Sr in environmental research, Table 1 presents data on the concentrations of ^{90}Sr found in different environmental soil samples collected from representative regions, highlighting the variations in ^{90}Sr concentrations across different environmental matrices and geographical locations. Compared with ^{90}Sr in ordinary soil, the content of ^{90}Sr in soil directly affected by nuclear activities is higher. For instance, the ^{90}Sr level in soil near the Fukushima nuclear power plant is one order of magnitude higher than the soil baseline in the region, while the ^{90}Sr contamination within the Chernobyl exclusion zone is two to three orders of magnitude higher than that outside the zone. Analyzing different concentration ranges with different methods can provide information for risk assessment and remediation strategies.

The current standard method for analyzing ^{90}Sr in environmental and biological samples in China is "Radiochemical analysis of strontium-90 in Water and Biological Sample Ash" (HJ 815-2016)[8], which requires 2-3 column loading during the separation process. After the separation process is completed, it needs to be placed to allow ^{90}Sr/^{90}Y equilibrium before measurement, and is not suitable for rapid analysis of samples in case of emergency. Mass spectrometry for ^{90}Sr measurement does not require waiting for radioequilibrium time because it directly measures the number of ^{90}Sr atoms, but at the same time, a large amount of isobar ^{90}Zr in the sample and the tailed peak of stable ^{88}Sr will interfere with the accurate determination of ^{90}Sr in the mass spectrometry. Then consider introducing the collision reaction cell (CRC) technique into ICP-MS to suppress the formation of the tailing peaks of the same amount of isobar ^{90}Zr and stable ^{88}Sr, as well as other molecular ion peaks that interfere with ^{90}Sr measurement through a series of condition tests, in order to achieve high sensitivity and high accuracy in ^{90}Sr measurement[9-12].

In response to the increasingly severe nuclear environment situation today, we have higher requirements for nuclear emergency monitoring methods. On the one hand, they need to meet the demands of traditional environmental monitoring, separating target nuclides from complex environmental samples and having a detection limit that can accurately measure low radioactivity nuclides. On the other hand, due to the urgency of nuclear emergency response, it is necessary to significantly shorten the analysis cycle of individual samples while also having the capacity to process a large

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number of samples simultaneously. Therefore, we need to establish rapid and efficient analytical methods.

2 Pre-treatment and chemical separation of soil samples

At the beginning of the analysis process, the soil samples need to be pre-treated by dry ashing and acid leaching to ensure that the elements to be tested are effectively leached from the matrix and transferred to the solution system for operation. Chemical separation using a combination of selective precipitation and Sr resin can significantly improve the efficiency of Sr separation from a large number of matrix elements.

2.1. Instruments and reagents

The experiment was conducted using soil near the China Institute of Atomic Energy. The sampling depth was 0-5 cm on the surface layer. Stones (diameter >5 mm) and vegetation roots were removed. The remaining samples were dried to a constant weight, then re-weighed, ground, passed through a 200-mesh sieve and sealed for storage.

The specific instruments and reagents for the experiment are as follows: Sr extraction resin with a particle size of 50-100 µm, Eichrom LTD, USA.Electronic balance BS110S, Sartorius LTD. High-speed centrifuge TG18M, Changsha Pingfan Instrument & Meter Co., LTD. Constant temperature magnetic stirrer S10-3, Shanghai Sile Instrument Co., LTD. Milli-Q Ultrapure Water System A10, Merck Millipore GMBH, Germany; Vacuum pump ZK-26/25, Hangzhou Mio Instrument Co., LTD. A variety of pipettes, Eppendorf AG GMBH, Germany; Muffle furnace 1200X, Hefei Kejing Materials Technology Co., LTD.Heavy metal digestion instrument MDS-6G, Shanghai Xinyi Microwave Chemistry Co., LTD. ICP-OES PQ9000, Jena Analytical Instruments AG, Germany.0.5×5 cm chromatographic column 7370507, BIO-RAD, USA.

Ferrous sulfate ammonia solution, solid calcium chloride, solid aluminium sulfate, solid ferric sulfate, solid aluminium chloride, 20% hydrogen peroxide solution, nitric acid, hydrochloric acid, concentrated ammonia water, sodium hydroxide and sodium carbonate and strontium nitrate.

2.2 Experimental Methods

Soil sample pretreatment: Accurately weigh about 10 g of soil sample and place it in a crucible. Send it into a muffle furnace and heat it continuously at 550℃ to decompose organic matter. Take it out every two hours, cool it to room temperature and weigh it. When the soil weight no longer changes, it indicates that the organic matter has been completely decomposed. Use an electric hot plate for digestion. Transfer the ashened soil to a beaker. In one of the spiked groups, a standard solution containing 100 µg Sr was added, and then the soil was digested at 90 ℃ on an electric hot plate in a certain proportion to 8 M HNO_3 solution, and the insoluble matter was filtered and separated. After cooling, the supernatant was filtered through a filter paper into a centrifuge tube, and the beaker and filter paper were washed with clean water until the color of the filter paper turned white and the sample was completely washed. Mix the filtrate thoroughly, weigh the total mass, and take a small portion to dilute and set aside.

Removal of Ca by precipitation method Take the previously diluted solution and add 6 M NaOH. Heat at 70 ℃ and stir for 30 minutes. Cool and centrifuge to separate $Fe(OH)_3$ precipitate. Then add Na_2CO_3 and adjust pH with 6 M NaOH to precipitate Ca. Centrifuge to take the supernatant and repeat three times. Then add excess Na_2CO_3 to form $SrCO_3$ precipitate. After centrifugation, dissolve with 8 M HNO_3.

Sr resin separation: Load approximately 0.6 g of the pre-soaked Sr resin into the separation column. Ensure that the column is packed wetly to avoid any air bubbles. The dimensions of the resin column is 0.5 ×5cm. Adjust the liquid flow rate in the column to no more than 0.6 mL/min. Pre-elute the remaining Sr with 5 mL of 0.05 M HNO_3 and pre-equilibrate with 5 mL of 8 M HNO_3. Load the target solution onto the column, wash the column with 30 mL of 8 M HNO_3, elute continuously with 5 ml of 0.05 M HNO_3, and collect the eluate. Evaporate the eluent to near-dry, make up to volume with 2 mL 3% HNO_3, and use ICP-OES to measure the Sr and other interfering element loading curves, and compare them with the stable Sr content in the spiked group solution to obtain the Sr recovery rate in the separation and purification process.

2.3 Results and Discussion

The column curves of different interfering elements Zr, Ca, Fe, Y measured by ICP-OES after the soil sample solution passed through the Sr resin column are shown in the Figure 1. It can be seen that the content of Ca element is the highest among the soil matrix elements, and the adsorption capacity of other interfering elements on the Sr resin is very small, which meets our requirements for specific extraction of Sr.

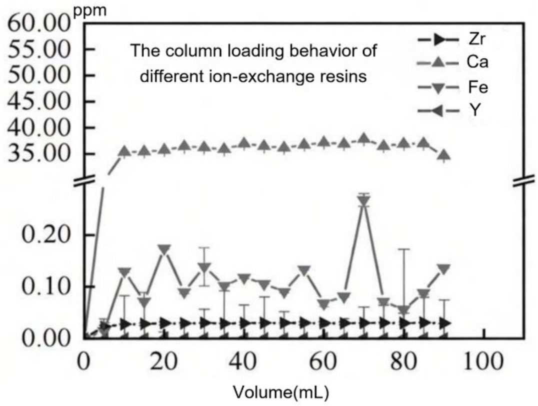

Fig. 1. The column loading behavior of different ion-exchange resins

Meanwhile, the effects of whether Ca was removed from the soil sample dissolution solution by precipitation on the Sr column penetration curve are shown in the Figure 2 and Figure 3.

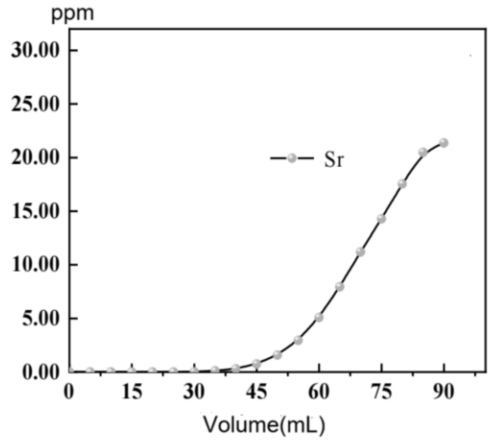

Fig. 2. The breakthrough curve of Sr on the column without using co-precipitation method to remove Ca

When Sr appears in the effluent, it indicates that the resin column is penetrated. When the concentration of Sr in the effluent remains unchanged, it indicates that the resin column is completely penetrated. At this point, the adsorption capacity of the resin for Sr can be calculated. By comparing the two figures, it can be seen that if no additional Ca is removed, the adsorption capacity of the resin for Sr is only 8.5 mg/L, but after the soil sample solution is treated by co-precipitation to remove Ca, the adsorption capacity of the resin for Sr can reach 25 mg/L.This is because the content of the matrix element Ca in the soil sample is several orders of magnitude higher than that of Sr. A large amount of Ca will competitively adsorb on the active sites of the resin, causing the resin column to be blocked. Therefore, the removal of Ca by co-precipitation can significantly improve the extraction effect and reduce the consumption of resin at the same time.

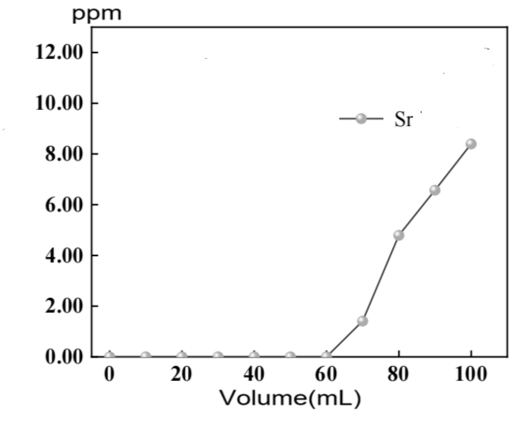

Fig.3. The breakthrough curve of Sr on the column when using co-precipitation method to remove Ca

The decontamination factors of the main interfering ions before and after separation and purification of the sample measured by ICP-OES are shown in the Table 2. Most interfering elements are reduced below the detection limit, and other major interfering ions also have a significant decontamination effect, and no other interfering is observed to be introduced.

Table 2. Decontamination Factors for Separation Processes.

Impurity ions	Original concentration	Elution concentration	Decontamination factors
Sc	500 ppb	1.62 ppb	309
Ti	560 ppm	<IDL	$>10^6$
V	16 ppm	0.42 ppb	3.8×10^3
Cr	11 ppb	<IDL	$>10^4$
Mn	95 ppm	<IDL	$>10^5$
Fe	4100 ppm	31.6 ppb	1.30×10^5
Ni	80 ppb	<IDL	$>10^5$
Ge	180 ppb	0.69 ppb	261
Se	5 ppb	0.39 ppb	13
Y	3 ppm	<IDL	$>10^4$
Zr	19 ppm	8.82 ppb	2150

0.5 mL was taken from the 100 mL sample solution before and after chemical separation and purification, diluted 20 times, and the total amount of Sr was measured before and after. Six groups of 5 g soil samples were treated with the co-precipitation method combined with Sr resin for separation. The results are shown in Figure 4. The chemical treatment recovery rate of the samples was 88 ± 3%, which met the requirements for separation and purification of ^{90}Sr in soil samples before analysis.

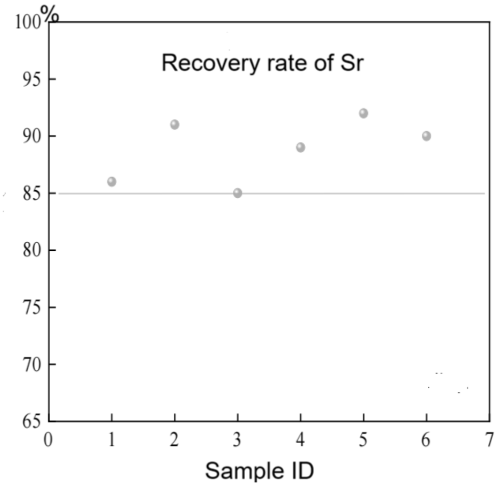

Fig.4. The Parallel experimental results of Sr recovery rate

3 Establishment of an ICP-MS method for measuring ^{90}Sr

The method of determining ^{90}Sr using ICP-MS not only provides higher sample throughput but also saves more time and is more suitable for application in rapid analysis fields such as continuous monitoring of nuclear facilities and nuclear emergency response. However, the interference of isobar with ^{90}Zr, the tailed peak of stable isotopes with ^{88}Sr, and the influence of molecular ion interference such as $^{74}Ge^{16}O^+$ have raised the detection limit of ICP-MS measurements, which is not conducive to the detection and analysis of low levels of ^{90}Sr. Analysis was conducted using CRC-ICP-MS with triple quadrupole mass filters (Agilent 8800) to explore methods for rapid and sensitive analysis of ^{90}Sr.

3.1. ICP-MS Instrument Parameter Settings

Using the 8800 series quadrupole ICP-MS produced by Agilent Technologies, Inc..It is equipped with a collision reaction cell (CRC) in front of the quadrupole. Through gas chemical reactions, the CRC significantly reduces the mass spectrometry interference ions, while another quadrupole in front of the CRC achieves two mass selections, further reducing the interfering substances entering the CRC, ensuring the controllability of the reaction in the CRC and avoiding new interferences caused by mass transfer. After the CRC treatment, The ions to be tested and the overlapping interfering ions are effectively separated. The product ions coming out of the CRC are then selected by the second quadrupole (Q2) and enter the detector for measurement.Table 3 shows some of the parameter values of the ICP-MS instrument in operation, which are fine-tuned for the focusing, extraction, and measurement process of the Sr ion beam for ^{90}Sr measurement in ICP-MS/MS series mode.

Table 3. Recommended operating parameters for Agilent 8800 ICP-MS.

Parameters	Recommended Value	Parameters	Recommended Value
Extraction lens 1	-10 V	Q1 entrance	-10 V
Extraction lens 2	-160 V	Q1 pre-filter deflection voltage	-10 V
Omega bias voltage	-70 V	Q1 deflection voltage	0 V
Omega lens voltage	2 V	Post-filter deflection voltage	-10 V
Collision cell entrance	-50 V	Q1 exit	-5 V
Octopole RF	170 W	Collision cell focus	0 V
Collision cell exit	-70 V	Octopole deflection voltage	-10 V
Deflect	-3 V	Energy discrimination	-5 V
Plate Bia	-50 V	Scan line slope factor	0.4
Cleaning time	2 min	Scan line gain factor	0.9
Measurement stabilization time	30 s	Waiting time compensation	2 min

3.2 Experimental Section

During the study, due to the optimization of plasma conditions, interference with ^{90}Zr, which is currently reported to have the greatest impact on measurements, has been reduced to a minimum. The focus on the removal of the collision reaction in the experiment was on the removal of GeO, and NH_3 reaction gas was attempted because of the high reactivity of Ge with NH_3. For safety reasons, ammonia was often used in a mixture of 10% He gas, with the optimal ammonia flow rate kept constant, and the flow rate of He gas was changed starting from 0.5 mL/min. Adjust the range from 5% to 100%, increasing by 0.5 mL/min each time, then keep the He gas flow rate constant, adjust the NH_3 gas flow rate to the corresponding value, and compare the effects of these three adjustments on the 10 ppb Sr, Zr, GeO signal values to obtain the optimal flow rate of NH_3. And the effects of different NH_3-He ratios and He flow rates on the measurement results.

Finally, prepare standard solutions containing different concentrations of simulated soil matrix, form calibration curves, and calculate the detection limits of the method according to the Formula 1.

$$IDL = 3SD_{blk} \times STD_{conc}/(STD_x - BLK_x) \quad (1)$$

IDL refers to the instrument detection limit; SD_{blk} represents the standard deviation of multiple blank sample measurement signal values; STD_{conc} refers to the concentration of the standard solution; STD_x refers to the signal value of the standard solution; BLK_x refers to the blank signal.

3.3 Results and Discussion

The influence of NH_3-He gas element sensitivity observed in the experiment is shown in the Figure 5 and Figure 6. As the flow rate of NH_3-He gas gradually increased, both GeO^+ and ^{88}Sr interferences were effectively reduced. The formation of interferences detected at the maximum flow rate was 0, indicating that both interferences were reduced below the instrument detection limit at this time and could not form a count in the measurement. The signal intensity of the 0.1 ppt ^{90}Sr standard solution is 10.67 cps. The tailed peak of the 500 ppm stable Sr and the $^{88}SrH_2^+$ product do not generate counts at m/z = 90. The requirement of effectively reducing mass spectrometric interference in the ICP-MS/MS measurement of ^{90}Sr has been achieved. At the same time, in the collision reaction gas experiment, the major interference of ^{90}Sr in surface soil - molecular ion interference, especially the oxide GeO with the highest formation efficiency, has been significantly reduced. The established measurement method has high sensitivity to Sr and a low interference formation rate, and has a good detection effect on low-level ^{90}Sr.

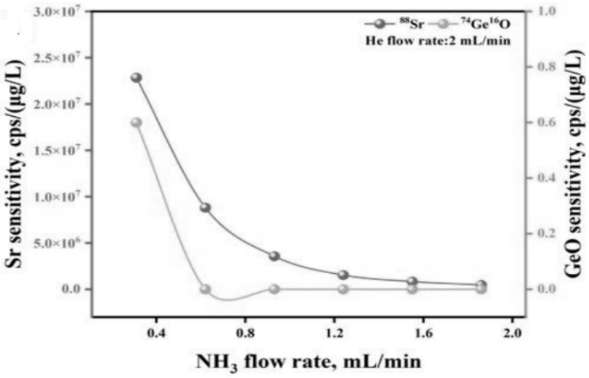

Fig. 5. The influence of NH_3 gas flow rate on the sensitivity of Sr and Ge

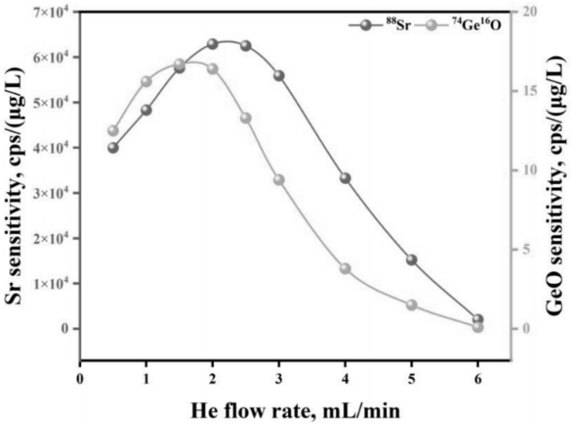

Fig. 6. The influence of He gas flow rate on the sensitivity of Sr and Ge

The optimal parameters for measuring ^{90}Sr in the cold plasma NH_3-He mode were determined as RF power 650 W, sampling depth 13.0 mm, carrier gas flow rate 0.70 L/min, compensation gas flow rate 0.50 L/min, and NH_3-He reaction gas of 1 mL/min NH_3 and 2 mL/min He. Compared with traditional radiometric measurement techniques, this method significantly increased the sample processing volume, shortened the processing time, and the total measurement time for each sample was only 1 minute. Compared with traditional ICP-MS, the ability to remove interference is greatly enhanced.

4 Conclusions and prospect

Aiming at the goal of rapid analysis of ^{90}Sr in environmental soil samples, a complete set of processes was determined, including dry ashing and acid leaching pretreatment, chemical separation process combined with $Ca(OH)_2$ co-precipitation and Sr resin extraction chromatography, and analysis process of CRC-ICP-MS, to achieve accurate analysis of ^{90}Sr above 4.3 mBq/g level in environmental soil samples. Compared with the analysis cycle of more than 3-5 days required by traditional radiometric measurement methods, this process takes approximately 10 hours, significantly reduces the analysis cycle, greatly improves the detection efficiency of ^{90}Sr in environmental soil samples, uses less chemicals, and ensures the stability of the analysis process with an Sr recovery rate of 88%. It is a rapid and accurate analysis method It can meet the demand for rapid analysis of ^{90}Sr in environmental soil samples under nuclear emergency response conditions.

Although this paper has established a rapid method for analyzing ^{90}Sr in environmental soil samples and achieved certain progress, there are still many directions that are worth further exploration and improvement. In view of the current ICP-MS/MS method being affected by matrix effects and requiring relatively strict sample pretreatment, it is possible to consider developing new online enrichment technologies, directly coupling the separation and enrichment process with the detection system, and exploring the tandem application of multi-functional columns to achieve the separation of multiple radionuclides in a single separation process, thereby realizing the automated processing and analysis of environmental samples.

In terms of method validation and application expansion, only laboratory simulation conditions and small-scale sample processing have been carried out so far. More systematic validation work on real environmental samples and large-scale sample processing needs to be conducted. At the same time, application databases should be established for different regions and different sample characteristics, and more detailed parameter optimization should be carried out for different samples in a targeted manner. Additionally, the method can be considered for extension to the analysis of other environmental media such as water, biological samples, and aerosols, to improve the rapid analysis method of ^{90}Sr under different environmental conditions.

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