

## Research Article

# Rapid Determination of Gross Alpha/Beta Activity in Water Based on Reverse Osmosis Membrane Enrichment Pretreatment

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Received 22 September 2021; Revised 24 December 2021; Accepted 27 January 2022; Published 15 February 2022

Academic Editor: Peter Ivanov

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Radioactivity of gross alpha/beta is an index of water quality detection, which can reflect the radioactivity intensity of water. However, the traditional detection method of these parameters, thick source method, has problems of cumbersome and time consumption in sample preparation and cannot realize the rapid detection on-site. Based on this, this paper studies the enrichment method based on reverse osmosis membrane to accurately and quickly determine the gross  $\alpha$  and gross  $\beta$  in water by using the reverse osmosis membrane as the carrier and enriching the radionuclides in water to the high-pressure side of the reverse osmosis membrane to replace the sample preparation process in traditional thick source method, so as to shorten the sample processing time in the detection process and avoid the cumbersome sample preparation process. The reverse osmosis membrane enrichment method for the determination of gross in  $^{241}\text{Am}$  and  $^{40}\text{KCl}$  standard solutions was used to study gross alpha/beta radioactivity, and the results showed that the average recoveries of radioactivity of gross alpha/beta were 95.0% and 93.6%, respectively. At the same time, the results of the thick source method and the reverse osmosis membrane method on the gross alpha/beta of actual water samples in 5 different regions were compared. It showed that the thick source method and the reverse osmosis membrane method had a good consistency in the detection results of total  $\alpha$  and total  $\beta$  radioactivity, and the reverse osmosis membrane method had better stability than the thick source method. The average relative standard deviations (RSD) of the gross alpha and gross beta activity obtained by the thick source method are 11.9% and 7.3%, respectively, while RSD of the gross alpha and gross beta radioactivity obtained by the reverse osmosis membrane method were 6.9% and 4.7%, respectively. The preparation time of single sample was reduced by 75.7%, and the overall detection cycle time was reduced by 68.1%.

## 1. Introduction

Nuclear power plays an important role in the global power supply system. Due to its clean, safe, and efficient characteristics, its share in the energy structure is increasing. Among them, the proportion of nuclear power in Western developed countries is generally high. For example, French nuclear power is the main source of power, accounting for 70.6% of its gross power generation. American nuclear power generation accounts for 19.7% of the country, but the number of nuclear power plants accounts for 21% of the world. At present, China is gradually strengthening the construction of nuclear power units, with a net capacity of more than 47528 MW, ranking third in the world after the

United States and France [1]. However, while vigorously developing nuclear energy, it also faces certain risks. In the event of an accident in a nuclear power plant, nuclear fission products will enter the human body with media such as atmosphere, water, and food [2], and the emitted rays will cause internal radiation damage to the human body [3, 4]. In severe cases, it may lead to various types of cancer [5, 6].

$\alpha$  and  $\beta$  ray intensity is an important parameter to characterize nuclear radiation. By monitoring the gross alpha/beta activity in the water, it can reflect the operating conditions of nuclear power plants, determine whether a nuclear leak has occurred, and ensure the people's water safety. The standard method for the detection of gross alpha/beta activity in water is the thick source method. However,

this method is complicated in operation process, requires reagent consumption, time-consuming, and has poor timeliness, which cannot meet the current demand for nuclear safety monitoring in water. Therefore, improving the detection efficiency of gross alpha/beta radioactivity has become the current research focus of nuclear safety detection in water bodies. Reverse osmosis (RO) is a technology for selective separation of solvents and solutes driven by the pressure difference on both sides of the membrane [7], which has good selection and adsorption characteristics for salt molecules in water. At the same time, reverse osmosis membrane has the characteristics of high separation efficiency, large water flux, simple structure, and low maintenance cost. At present, it has been widely used in seawater desalination [8, 9], industrial wastewater treatment [10, 11], household water purification, and other fields [12]. As early as the 1980s, the reverse osmosis system for the treatment of radioactive wastewater has been put into use [13], and many scholars have studied and proved that reverse osmosis membrane (RO) was an effective method for the treatment of radioactive wastewater [14]. For example, Combernoux et al. used Dow SW-30HR reverse osmosis membrane to detect Cs and Sr in simulated radioactive wastewater [15]. The research results showed that the recovery of Cs and Sr was about 96%. Du Zhihui and others used reverse osmosis membrane to remove salt and nuclide in simulated radioactive wastewater. The results showed that under the influence of operating pressure, raw water pH value, salt content, and other factors, the rejection rate could still be maintained above 97.3%, and the maximum rejection rates of reverse osmosis membrane for nickel, manganese, and cobalt were close to 98.7%, 100%, and 98.8%, respectively [16, 17]. Gu et al. treated simulated boron-containing radioactive wastewater with a two-stage reverse osmosis device. After treatment, the desalination rate and gross boron removal rate remained above 99.50% and 84.30%, respectively. The concentration of  $^{90}\text{Sr}$  in the secondary permeated water was lower than the detection limit, and the concentration of  $^{137}\text{Cs}$  was below  $25 \text{ Bq}\cdot\text{L}^{-1}$  [18]. In conclusion, reverse osmosis technology could effectively remove radionuclides from radioactive wastewater.

Based on this, this paper proposes a water nuclide enrichment technology based on reverse osmosis membrane (Hereinafter referred to as "reverse osmosis membrane method"), which directly extracts radionuclides by intercepting and attaching solutes to the high-pressure side of the membrane, so as to shorten the pretreatment time and realize the rapid detection of gross alpha/beta concentrations in water bodies.

## 2. Materials and Methods

**2.1. Instruments and Reagents.** The customized reverse osmosis membrane TMH10A used in the experiment was purchased from (Toray company, Japan). The experimental instrument included ZK-H-1302 low background  $\alpha/\beta$  measuring instrument (Zhongke Huili company), main detector ZnS plastic scintillator ( $\alpha$  background:  $<0.012/\text{cps}/\text{min}$ ;  $\beta$  background:  $<0.23/\text{cps}/\text{min}$ ), ME204/02 electronic

balance (Mettler, Switzerland), Muffle furnace (Shanghai Lichen company), and DHG-9013A blast drying oven (Shanghai Yiheng company). Residue in water  $\alpha$  standard powder source ( $^{241}\text{Am}$ , specific activity of  $14 \text{ Bq}\cdot\text{g}^{-1}$ , reference date: December 4, 2020) and residue in water  $\beta$  standard powder sources ( $^{40}\text{KCl}$ , specific activity of  $16.1 \text{ Bq}\cdot\text{g}^{-1}$ , reference date: December 4, 2020) were purchased from the China Academy of Metrology.

### 2.2. Sample Preparation and Method

**2.2.1. Detector Efficiency Calibration.** Because the thickness factor is uncertain when the reverse osmosis membrane is used for enrichment, it is necessary to draw the detection efficiency curve for calibration. Spread the  $\alpha$  standard powder ( $^{241}\text{Am}$ :  $14 \text{ Bq}\cdot\text{g}^{-1}$ ) or  $\beta$  standard powder ( $^{40}\text{KCl}$ :  $16.1 \text{ Bq}\cdot\text{g}^{-1}$ ) standard powder sources with masses of 50 mg, 100 mg, 200 mg, 300 mg, and 400 mg as evenly as possible on 5 new reverse osmosis membranes to form 5 groups of standard source membranes with different mass thicknesses. Then, they were respectively placed in the sample tray of the low background gross  $\alpha$  or  $\beta$  measuring instrument to measure the gross  $\alpha$  or  $\beta$  counting rate of the standard source diaphragm. The  $\alpha$  or  $\beta$  detection efficiency of each group of standard source diaphragm was calculated according to formula (1). Finally, the standard efficiency curve (2) of standard source quality and detection efficiency of each standard source diaphragm was obtained by exponential fitting with the least square method.

$$E_i = \frac{1000(R_s - R_0)}{m_i a_s} \times 100\%, \quad (1)$$

where  $E_i$  is the detection efficiency of each group of standard source membranes of different quality, %;  $R_s$  is the count rate of the standard source membrane,  $\text{s}^{-1}$ ;  $R_0$  is the background count rate of the new composite reverse osmosis membrane,  $\text{s}^{-1}$ ;  $m_i$  is the standard powder source quality of each group of standard source diaphragms; and  $a_s$  is the specific activity of standard source,  $\text{Bq}\cdot\text{g}^{-1}$ .

$$\varepsilon = k e^{-am}, \quad (2)$$

where  $\varepsilon$  corresponds to the detection efficiency when the mass of the standard source is  $m$ , %;  $k$  is the detection efficiency when the mass thickness is equal to zero (no self-absorption);  $e$  is the natural constant;  $a$  is the self-absorption coefficient; and  $m$  is the corresponding standard source mass, mg.

**2.2.2. Detection of Standard Samples.** First, the new composite reverse osmosis membrane was weighed and recorded and then placed in the sample tray of the low background gross alpha/beta measuring instrument for background value determination. The volume of 0.1 L, 0.25 L, 0.5 L, 1 L, 1.5 L, 2 L, 2.5 L, and 3 L  $^{241}\text{Am}$  and  $^{40}\text{KCl}$  mixed standard solutions were configured, and the  $\alpha$  activity concentration and  $\beta$  activity concentration in all samples were  $1.4 \text{ Bq}\cdot\text{L}^{-1}$  and  $1.61 \text{ Bq}\cdot\text{L}^{-1}$ , respectively. Subsequently, the composite

reverse osmosis membrane that had completed the measurement of the background value was placed in the cavity, as shown in Figure 1 for filtration and enrichment. The filtration and enrichment mechanism was made of stainless steel, and rubber rings were used at the joints of the movable parts to improve the sealing performance. A hose was connected at the top, the air supply pressure was provided by the screw air compressor to make the raw water pass through the reverse osmosis membrane, and the pressure was set at 1 MPa.

After enrichment, the reverse osmosis membrane with solute attached was placed in a constant temperature drying oven at 105°C for 1 h, cooled to room temperature, weighed again, and placed in the sample tray of the low background gross alpha/beta measuring instrument to determine the sample gross  $\alpha/\beta$  count rate (measurement time was set to 240 min). The overall operation flow of reverse osmosis membrane method is shown in Figure 2.

The calculation formula of water sample volume activity is the following formula:

$$C = \frac{(R_x - R_0) \times M}{m\varepsilon PV}, \quad (3)$$

where  $C$  is the gross water sample  $\alpha$  or gross  $\beta$  radioactive volume activity,  $\text{Bq}\cdot\text{L}^{-1}$ ;  $R_x$  is the counting rate of sample source,  $\text{s}^{-1}$ ;  $R_0$  is the background count rate,  $\text{s}^{-1}$ ;  $M$  is the gross weight of ash residue after sample preparation, mg;  $m$  is the weight of the residue taken to make the sample evenly spread, mg (note: reverse osmosis membrane enrichment method  $M=m$ );  $\varepsilon$  is the detection efficiency, %;  $P$  is the chemical recovery rate of evaporation sample preparation or composite reverse osmosis membrane pair  $\alpha$  and  $\beta$  enrichment recovery, %; and  $V$  is the volume of water sample taken for sample preparation, L.

**2.2.3. Comparative Test of Environmental Water Samples.** 40 L surface water samples were collected from Zhuhai City's drinking water source Youth Reservoir, Shenzhen drinking water source Xili Reservoir, Shenzhen drinking water source Luohu Reservoir, the Yellow River Reach of the first water plant in Lanzhou, and the Dongyangdu River section of Xiangjiang River in Hengyang, respectively. During sampling, avoid disturbing the sediment at the bottom of the reservoir or river section to reduce the impact of solid sedimentation in the water on the sample. Five 2 L supernatant samples were taken from water samples in each region, and gross alpha/beta values were tested by thick source method. It took 48 h to concentrate to 50 mL by electric heating 80°–85°, and it took 24 h to complete the other steps of sample preparation and 72 h to prepare a single sample. In addition, five 2 L supernatants were sampled by reverse osmosis membrane method and the gross  $\alpha/\beta$  values were determined. The preparation process was consistent with the above standard sample testing process. Enrichment process for 17 h (Figure 2), drying for 30 minutes, and the counting rates of gross  $\alpha/\beta$  obtained by the two methods were set at 240 min.

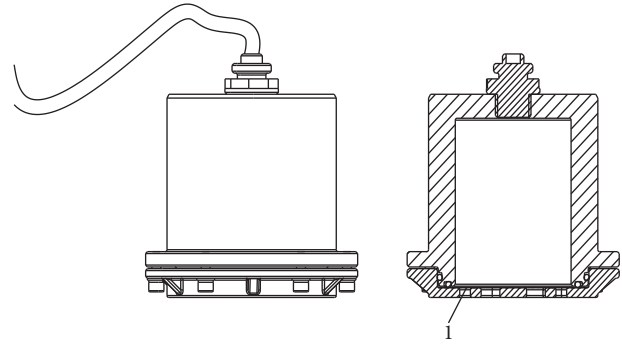


FIGURE 1: Enrichment filter mechanism, wherein marked 1 is the composite reverse osmosis membrane.

### 3. Results and Discussion

**3.1. Analysis of Test Results of Standard Samples.** In order to verify the accuracy of reverse osmosis membrane enrichment method for detecting standard samples prepared in the laboratory, the reverse osmosis membrane enriched with 0.1 L–3 L standard solution was dried and the gross  $\alpha$  and  $\beta$  radioactivity was measured (the specific activity corresponding to the mass of solute is regarded as the real value), as shown in Table 1.

It could be seen from Table 1 that the gross alpha/beta radioactivity increased with the increase of raw water volume, and the gross  $\alpha$  recovery ranged from 87.2 to 102.8%, with an average recovery of 95%. The gross  $\beta$  recovery ranged from 80 to 103.4%, with an average recovery of 93.9%. These results indicated that the recoveries of gross alpha/beta radioactivity measured by reverse osmosis membrane enrichment method were above 90%, and the measurement results were closer to the real values. The recovery rate decreased at 0.1 L, probably because the sample volume was too small, and some of the solution adhered to the inner wall of the cylinder due to the surface tension of the liquid, which led to the reduction of the actual solution through the reverse osmosis membrane. On the other hand, from the perspective of enrichment time, the enrichment time increases almost exponentially with the linear increase of liquid volume at constant pressure, indicating that the TDS (gross dissolved solids) on the high-pressure side of the reverse osmosis membrane increases continuously when the volume of raw water is decreasing, leading to the phenomenon of concentration polarization, which results in a significant increase of enrichment time in the late stage of sample preparation for large volume water samples. The recovery rate of gross  $\alpha$  reached the highest when the volume of raw water was 2 L, 99.1%; the recovery rate of gross  $\beta$  reached the highest when the volume of raw water was 1.5 L, 99%. Therefore, it was not the larger the volume of the test water sample, the higher the accuracy of the test, the gross recovery was greater than 90% at the raw water volume of 0.5 L and the highest recovery at the raw water volume of 2 L. By controlling the raw water volume, it was possible to choose between efficiency and accuracy. Even if the raw water volume was only 0.5 L, the recovery rate was also high,

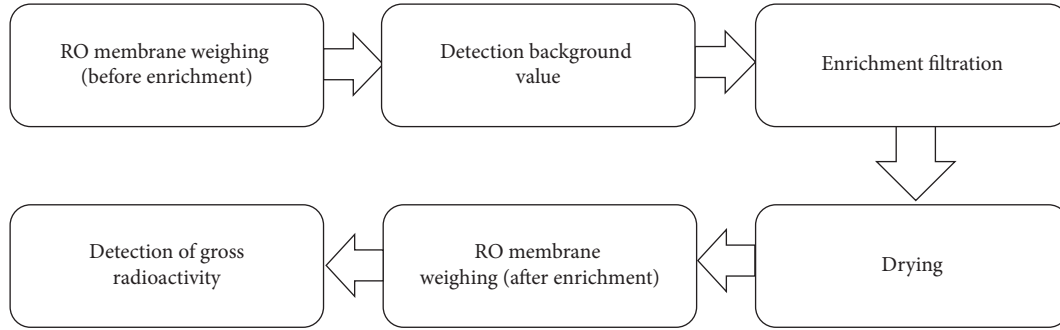


FIGURE 2: Reverse osmosis membrane method overall operation process.

TABLE 1: Results of gross alpha/beta radioactivity in standard solutions by reverse osmosis membrane method.

Volume (L)	Gross $\alpha$				Gross $\beta$				Enrichment time (h)
	Real value (Bq)	Measured value (Bq) ( $k=2$ )	Recovery rate (%)	Relative uncertainty (%)	Real value (Bq)	Measured value (Bq) ( $k=2$ )	Recovery rate (%)	Relative uncertainty (%)	
0.1	0.14	$0.12 \pm 0.038$	87.2	15.7	0.15	$0.12 \pm 0.056$	80.0	23.3	0.5
0.25	0.35	$0.33 \pm 0.064$	94.2	9.7	0.40	$0.35 \pm 0.110$	87.6	15.7	1
0.5	0.72	$0.68 \pm 0.129$	94.4	9.5	0.83	$0.76 \pm 0.170$	92.1	11.2	2
1	1.41	$1.33 \pm 0.231$	94.2	8.7	1.61	$1.50 \pm 0.297$	93.4	9.9	6
1.5	2.10	$1.92 \pm 0.438$	91.5	11.4	2.45	$2.43 \pm 0.257$	99.0	5.3	11
2	2.86	$2.61 \pm 0.376$	99.1	7.2	3.22	$3.33 \pm 0.446$	103.4	6.7	17
2.5	3.48	$3.58 \pm 0.566$	102.8	7.9	4.07	$3.91 \pm 0.563$	96.1	7.2	25
3	4.22	$4.05 \pm 0.680$	96.2	8.4	4.89	$4.64 \pm 0.780$	94.9	8.4	36
Average value	—	—	95.0	9.8	—	—	93.6	11.0	—

but when it was less than 0.5 L, the measurement uncertainty increases gradually. Compared with thick source method, reverse osmosis membrane method had great advantages in sample preparation time.

**3.2. Comparison of Detection Results of Environmental Water Samples.** The supernatant of water samples collected on-site at each water source was divided into 10 parts, of which 5 parts were prepared by thick source method, and the other 5 parts were prepared by reverse osmosis membrane method. Then, the activity detection was completed with the ZK-H-1302 low background  $\alpha/\beta$  measuring instrument, as shown in Figure 3. The measurement uncertainty of total  $\alpha$  in each region in this experiment is shown in Table 2; the measurement uncertainty of total  $\beta$  is shown in Table 3.

The average RSD of gross  $\alpha$  activity concentration was 11.9% and the average RSD of gross  $\beta$  activity concentration was 7.3% for five times of water samples from five areas tested by the thick source method. The average RSD of gross  $\alpha$ -activity concentration was 6.9% and the average RSD of gross  $\beta$ -activity concentration was 4.7% for five times using the reverse osmosis membrane method, which showed that

the stability of the test results of the reverse osmosis membrane method was better than that of the thick source method. Part of the reason was to reduce the error of manual operation, and the other reason was that the enriched reverse osmosis membrane samples can be tested in an extremely flat form. The RMSE of the two methods for detecting the gross  $\alpha$  and  $\beta$  activity concentrations of the water samples in each region were less than 15%. The RMSE for detecting the gross  $\alpha$  activity concentration was 10%, and the RMSE for detecting the gross  $\beta$  activity concentration was 13.9%. Therefore, the detection results of the two methods had good consistency. In addition, the gross  $\alpha$  and  $\beta$  results measured by the reverse osmosis membrane method were slightly higher than those of the thick source method by more than 5%, probably due to the loss of water samples during the heating and concentrating boiling and splashing of the thick source method. Using the reverse osmosis membrane method to measure the gross  $\alpha$  activity concentration of the water samples in the 5 regions, the average measurement uncertainty was 14.7%, and the average measurement uncertainty of the gross  $\beta$  activity concentration was 10.6%, which is equivalent to the measurement result of the thick source method. From the perspective of sample preparation

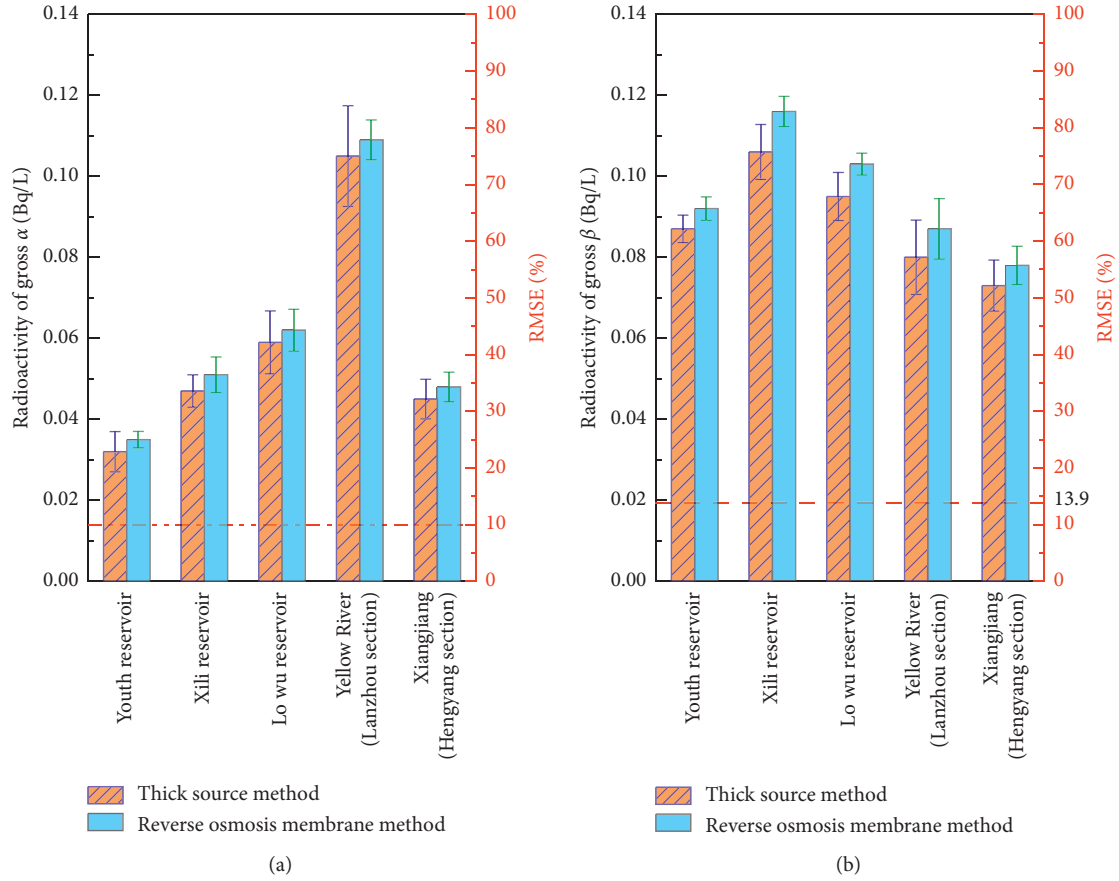


FIGURE 3: (a) The gross  $\alpha$ -activity concentration of 5 water sources measured by thick source method and reverse osmosis membrane method, respectively; (b) the gross  $\alpha$ -activity concentration of 5 water sources measured by thick source method and reverse osmosis membrane method, respectively. The gray bars indicate the relative errors of the two methods. The error bar was SD (standard deviation). The red dotted line is the identification of RMSE (root mean square error).

TABLE 2: Measurement uncertainty of total  $\alpha$  in each region.

Sites	Reverse osmosis membrane method			Thick source method		
	$u_{rel}(C_\alpha)$ (%)	$u(C_\alpha)$ (Bq·L <sup>-1</sup> )	$U(C_\alpha)$ ( $k=2$ ) (Bq·L <sup>-1</sup> )	$u_{rel}(C_\alpha)$ (%)	$u(C_\alpha)$ (Bq·L <sup>-1</sup> )	$U(C_\alpha)$ ( $k=2$ ) (Bq·L <sup>-1</sup> )
1	12.5	0.004	0.035 ± 0.009	14.7	0.005	0.032 ± 0.009
2	14.0	0.007	0.051 ± 0.014	10.6	0.005	0.047 ± 0.010
3	15.6	0.010	0.062 ± 0.019	16.4	0.010	0.059 ± 0.019
4	16.3	0.018	0.109 ± 0.036	19.3	0.020	0.105 ± 0.041
5	15.1	0.007	0.048 ± 0.014	13.5	0.006	0.045 ± 0.012
Average value	14.7			14.9		

$u_{rel}(C_\alpha)$  is the relative uncertainty when measuring the concentration of  $\alpha$  activity;  $u(C_\alpha)$  is the combined uncertainty when measuring the concentration of  $\alpha$  activity;  $U(C_\alpha)$  is the expanded uncertainty when measuring the concentration of  $\alpha$  activity (coverage factor  $k=2$ ).

TABLE 3: Measurement uncertainty of total  $\beta$  in each region.

Sites	Reverse osmosis membrane method			Thick source method		
	$u_{rel}(C_\beta)$ (%)	$u(C_\beta)$ (Bq·L <sup>-1</sup> )	$U(C_\beta)$ ( $k=2$ ) (Bq·L <sup>-1</sup> )	$u_{rel}(C_\beta)$ (%)	$u(C_\beta)$ (Bq·L <sup>-1</sup> )	$U(C_\beta)$ ( $k=2$ ) (Bq·L <sup>-1</sup> )
1	8.8	0.008	0.092 ± 0.016	7.8	0.007	0.087 ± 0.014
2	9.1	0.011	0.116 ± 0.021	12.8	0.014	0.106 ± 0.027
3	8.2	0.008	0.103 ± 0.017	9	0.009	0.095 ± 0.017
4	14.1	0.012	0.087 ± 0.025	14.3	0.011	0.08 ± 0.023
5	12.9	0.010	0.078 ± 0.020	13.4	0.010	0.073 ± 0.020
Average value	10.6			11.5		

$u_{rel}(C_\beta)$  is the relative uncertainty when measuring the concentration of  $\beta$  activity;  $u(C_\beta)$  is the combined uncertainty when measuring the concentration of  $\beta$  activity;  $U(C_\beta)$  is the expanded uncertainty when measuring the concentration of  $\beta$  activity (coverage factor  $k=2$ ).

time, the proposed new method reduced the sample preparation time by 75.7%.

#### 4. Conclusion

This paper proposed a simple and direct way to measure the gross  $\alpha$  and  $\beta$  radioactivity in water by using reverse osmosis membrane as the enrichment carrier. The results showed that the recoveries of gross  $\alpha$  and  $\beta$  radioactivity measured by the reverse osmosis membrane method were above 90% when the volume of raw water was 500 mL–3 L, so it had a good retention effect on the prepared  $^{241}\text{Am}$  and  $^{40}\text{KCl}$  mixed solution. The RMSE of the two methods was 10% for gross  $\alpha$  and 13.9% for gross  $\beta$ , which had good consistency. The overall test results of the reverse osmosis membrane method were higher than those of the thick source method by more than 5%, which might be due to the uncertain loss of the thick source method in the sample preparation process. In addition, when the ambient water samples were all 2 L, compared with the thick source method, the sample preparation time of the reverse osmosis membrane method was shortened by 75.7%, which showed a great advantage in the sample preparation speed and avoided the timeliness problems and instability factors brought by manual operation.

Therefore, the next step is to focus on using the rapid and stable characteristics of the reverse osmosis membrane method to establish an automated system in order to provide timely and stable radiation data of water bodies to provide data support for drinking water safety, environmental water safety, and nuclear safety.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

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