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Measuring ^{222}Rn in aquatic environment via Pulsed Ionization Chamber Radon Detector

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Abstract

Radon (Rn) is a naturally occurring radioactive inert gas in nature, and ^{222}Rn has been routinely used as a powerful tracer in various aquatic environmental research on timescales of hours to days, such as submarine groundwater discharge. Here we developed a new approach to measure ^{222}Rn in discrete water samples with a wide range of ^{222}Rn concentrations using a Pulsed Ionization Chamber (PIC) Radon Detector. The sensitivity of the new PIC system is evaluated at 6.06 counts per minute for 1 Bq/L when a 500 mL water sample volume is used. A robust logarithmic correlation between sample volumes, ranging from 250 mL to 5 000 mL, and system sensitivity obtained in this study strongly suggests that this approach is suitable for measuring radon concentration levels in various natural waters. Compared to the currently available methods for measuring radon in grab samples, the PIC system is cheaper, easier to operate and does not require extra accessories (e.g., drying tubes etc.) to maintain stable measurements throughout the counting procedure.

Key words: ^{222}Rn, radon measurement, Pulsed Ionization Chamber Radon Detector, radon in discrete water samples, submarine groundwater discharge

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1 Introduction

Radon (Rn) is a naturally occurring radioactive inert gas in nature. Inhalation or intake of radon will cause harm to human body. Radon accumulation in groundwater in high levels is an essential concern and it requires comprehensive monitoring in the environment (Zhao, 1993). According to published literature, the concentration range of ^{222}Rn in natural waters is significant (between 10^{-5} Bq/L and 20 Bq/L) with groundwater's levels 3–4 orders of magnitude higher than that of typical surface water (Broecker et al., 1967; Burnett et al., 2001; Cook et al., 2003; D'Alessandro and Vita, 2003; Guo et al., 2020). On the other hand, ^{222}Rn, with a half-life of 3.82 d, has been widely used as a robust tracer for assessing natural processes on time scales of 20 d, such as submarine groundwater discharge (SGD) and air-sea gas exchange fluxes, to mention a few (Baskaran, 2016; Cable et al., 1996; Lambert and Burnett, 2003; Savatier and Rocha, 2021; Seo and Kim, 2021; Yu et al., 2020; Zhang et al., 2016).

Currently, the most utilized instruments used to evaluate ^{222}Rn in natural waters include radon line emanation counter, RAD7 radon analyzer, and Pulsed Ionization Chamber (PIC) Radon Detector, etc. The "radon line emanation" technique was widely used in the 70s and 80s, 20th century. The background of the system is relatively low and the counting efficiency is high (theoretically to be 300%). This allowed using it for radon investigations in the open ocean since the GEOSECS era (Key et al., 1979). However, this system requires liquid nitrogen for concentrating the radon gas and high-purity helium as the carrier gas, which would often limit its on-site application. At the beginning of this century, a new automated radon-in-air analyzer called RAD7 was assembled with grab water samples and has been widely used currently to test the radon activities in discrete groundwater samples (Kim et al., 2001). The same radon detector can also be equipped with proper accessories and adopted to measure radon activities continuously in large variety of aquatic environments such as rivers and streams, estuaries, bays and other coastal settings (Baskaran, 2016; Burnett et al., 2001; Schmidt et al, 2008). However, the RAD7 has some limitations, one being maintaining relatively low humidity (RH%) inside the chamber (<10%) to ensure higher test efficiency (Kim et al., 2001). This could be often limiting when processing large num-

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ber of samples. Another disadvantage is the relatively high price of the RAD7 unit, especially for developing countries. The later had limited groundwater explorations using radon and a tracer in these parts of the world.

Recently, a new cheaper and more robust method for measuring radon in water using a PIC was proposed (Seo and Kim, 2021). The PIC technique offers high detection efficiency and sensitivity to radon variations. Since PIC counts electrons produced by air ionization, detection efficiency will not be affected by humidity (Gavrilyuk et al., 2015; Seo and Kim, 2021).

In this work, we further investigate the performance of the PIC technique under large range of ^{222}Rn activities that are observed in natural groundwater and seawater. This piece of knowledge will highly benefit not only the groundwater research community, but also government institutions who monitor radon activities in natural waters with radiological health protection purposes.

2 Method

2.1 *Instrument and principle*

The principle of detection of radon in gas phase has been described elsewhere (http://www.radonftlab.com). Briefly, a PIC Radon Detector is comprised a stainless chamber and a counting probe installed beneath the center of the top cover, which are negatively charged and positively charged respectively. Alpha (α) particles produced by radon radioactive decay ionize air in the ionization chamber to form electron ion pairs, which are gathered and counted by the PIC counter. The pulse electrical signal is converted into digital signal which is proportionally correlated with radon concentration (Seo and Kim, 2021).

More specifically, the experimental device in this study includes (1) an ionization chamber (200 mL), (2) air pump, and (3) the sample bottle (Fig. 1). If the inlet and outlet of the PIC counter are closed, the system will be sealed as a closed loop. When testing samples, a micro filtration membrane (0.45 μm) is placed in the upper stream of the PIC counter to prevent dust entering the chamber. We obtained the data using a self-designed software. Details about the software could be found in Li et al. (2022).

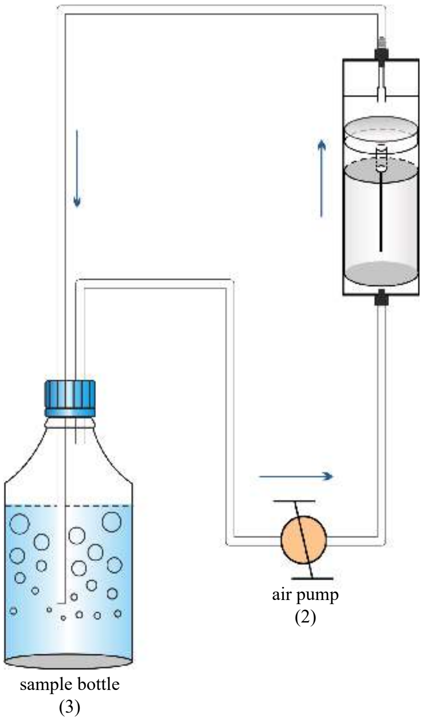

Fig. 1. Schematic diagram of experimental set-up by using Pulsed Ionization Chamber (PIC) counter to measure ^{222}Rn in water samples.

Theoretically, PIC Radon Detector measures the activities of all radon isotopes, including ^{222}Rn, ^{220}Rn and ^{219}Rn. However, the natural abundance of ^{219}Rn is very low because of its very short half-life of 3.96 s, so contribution from ^{219}Rn counting should be negligible to the total counts. The half-life of ^{220}Rn is 55.6 s, and it can decay completely in 5 min when unsupported. The half-life of ^{222}Rn is 3.82 d, which is longer than that of the other two radon isotopes. Hence, the activity of ^{222}Rn can be distinguished based on the difference of half-lives among the three radon nuclides.

2.2 *Preparation of standard and blank samples*

To evaluate the overall sensitivity of the system, describing in Fig. 1, we prepared several standard solutions with known amount of radon in the water. We used a standard solution of ^{226}Ra with traceable activity concentration 1 000 Bq/L. Since the half-life of ^{226}Ra ($T_{1/2}$=1 600 a) is much longer than the half-life of ^{222}Rn, the activity of the parent equals its daughter after five times of daughter's half-life according to the principle of secular equilibrium. Therefore, the concentration of dissolved ^{222}Rn in the standard solution equals the concentration of ^{226}Ra.

For these standards, we used natural seawaters which was filtered through Mn-fibers to remove naturally dissolved ^{226}Ra (Moore, 1976). A known amount of ^{226}Ra standard solution was then added to the radium free seawater to produce a series of standard samples, with volumes of 250 mL, 500 mL and 5 L, based on the size of the bottles. For the bottle with volume of 500 mL, six sub-samples were prepared with activity gradient of 0.04–0.8 Bq to evaluate the counting efficiency of the method. The pH of the standard seawater sample was adjusted to about 8 with ammonia solution. Before testing, water samples were purged with high-purity helium gas to remove any residual dissolved radon in the prepared solutions. One blank sample was also prepared following the same procedure but without the radium spike added. All the samples were then sealed for 7–10 d to allow the radon ingrowth to levels that would be detectable by the system.

2.3 *Protocol of water sample analysis*

Before each sample (or standard) test, the PIC counter was purged with helium gas to remove residual radon in the instrument. The bottles filled with water sample were then connected in the loop as shown in Fig. 1. We let the air pump turned on and run for 2 h to ensure radon was degassed completely from water and homogenously distributed in the system. The water samples were then removed, and the PIC counter was sealed for 5 min to let ^{220}Rn in the enclosed volume completely decay. The PIC counter was then reset and measured the activity of ^{222}Rn continuously for 2–3 h with 10 min data reporting interval. An experiment was considered completed when total counts were higher than 1 000, resulting in a counting error of <3%. If the sample activity is relatively low, the counting time was extended accordingly.

2.4 *Instrument background test*

To assess the detection limit of the system, the instrument background was tested. Immediately before this test, the system was purged with high-purity helium gas for about 1 min to remove the residual air in the PIC Radon Detector. The PIC was then sealed by quickly closing the inlet and outlet valves of the chamber (Fig. 1). The background of the instrument was then counted continuously for 24 h at an interval of 10 min to get a good understand of instrument background, which standard deviation could be used to calculate the detection limit.

2.5 *Aeration time test*

The required time for complete degassing of radon from water (i.e., the sparging times) is an important parameter when evaluating the efficiency of the method. We tested four different aeration times, 1 h, 1.5 h, 2 h and 3 h. Count rates were recorded while pump was optional in a certain sparging times condition. Counting statistics were obtained after 400 min counting time at an interval of 10 min.

2.6 *Sample collection and measurement*

To test the system, on a monthly basis form October 2021 to December 2021, we collected a total of 7 shallow groundwater samples (250 mL) and 2 seawater samples (500 mL) from the tidal flat of the west bank of Jiaozhou Bay, China. To compare the performance of our new system duplicates of groundwater samples was simultaneously measured by both PIC and RAD7-H_2O. The PIC method was developed by this study with details described in Section 2.4. The RAD7 method is combined with its H_2O accessory, with more details reported by Lee and Kim (2006).

To verify the test precision of the PIC method, a standard sample with volume of 500 mL and activity of 0.4 Bq/L were prepared in the laboratory based on the method described in Section 2.2. The sample was repeatedly measured for eleven times and the method precision was evaluated based on the standard deviation.

3 Results

3.1 *Instrument background*

Within 24 h counting, the instrument background value range of 0–0.08 cpm (counts per minute) as shown in Fig. 2, with an average of (0.04±0.02) cpm (n=143). The detection limit of the instrument is considered as three times of standard deviation, so the detection limit of the PIC method established in this study is 0.07 cpm.

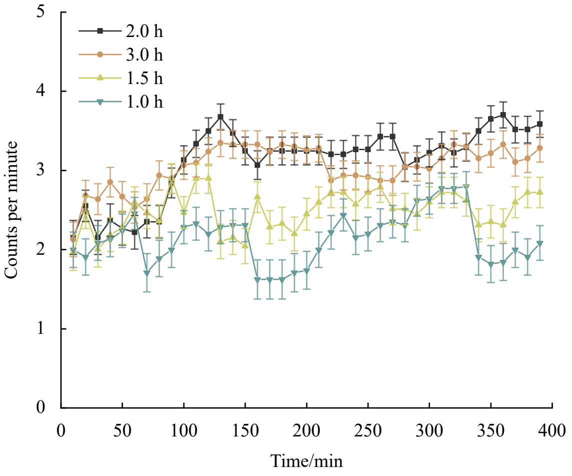

Fig. 3. Comparison of different sparging times experiment results.

3.2 *Sparging times*

The results based on different sparging times are shown in Fig. 3. The results show that radon degassing benefits from longer sparging times up to 2 h. We found that there was no statistical difference between sparging times of 2 h and 3 h. By averaging the count rates of the platform period, the average value was (2.1±0.3) cpm (n=39) when the sparging time was 1 h, (2.5±0.2) cpm (n=39) for a sparging time of 1.5 h, (3.1±0.2) cpm (n=39) and (3.0±0.2) cpm (n=39) when the sparging times were 2 h and 3 h, respectively. When we compared the data from the three experiments lower degassing efficiency and greater variation were found when the sparging time was shorter. Longer than 2 h sparging times did not contribute to significant radon degassing. Based on these results, we suggest that 2 h is an optimum sparging time for this method.

3.3 *Method efficiency and precision*

The result from counting the blank sample, (0.04±0.03) cpm, indicated that it was consistent with the instrument background value ((0.04±0.02) cpm), i.e., the blank sample had no radium and radon.

The results from analyzing the system efficiency using the prepared standard samples with a volume of 500 mL are presented in Fig. 4. Each standard was measured twice. Based to the slope of the linear fitting line, the efficiency of this particular PIC and sample of 500 mL water sample is 6.06 cpm for 1 Bq/L. We recommend that users conduct similar experiments when de-

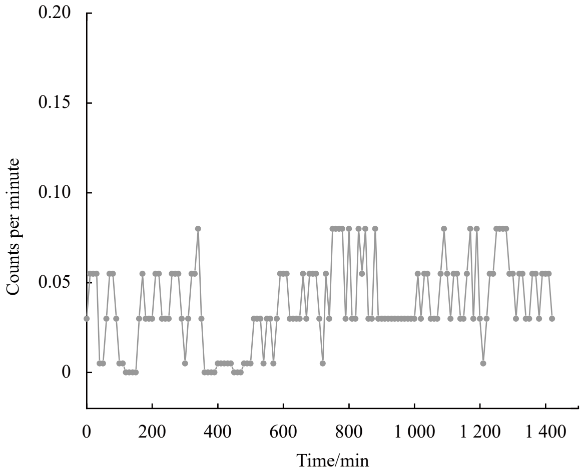

Fig. 2. Background value of Pulsed Ionization Chamber.

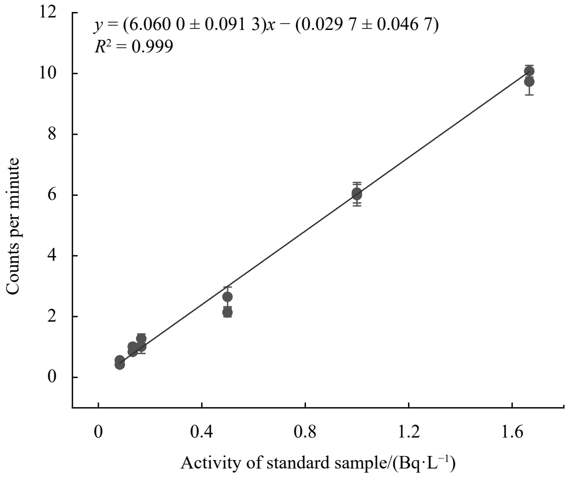

Fig. 4. The counting rate (cpm) vs. activities of standard samples.

termine the efficiency of their own equipment.

To evaluate the relationship between the overall system efficiency with sample volumes, the same PIC system was used to test the efficiency of sample volumes of 250 mL, 500 mL and 5 L samples. Each sample was measured twice, and results are shown in Fig. 5. It can be seen from the figure that with the increase of sample bottle volume, the test efficiency of the instrument also increases following a good logarithmic relationship (R^2=0.994).

In order to evaluate the test precision of the method, with a sample total activity of 0.2 Bq of ^{226}Ra was tested 11 times, and the results are shown in Fig. 6. The average activity of the 11 tests is (2.1±0.1) cpm, and the relative standard deviation is 6%. The test precision of the method is 6%, which is feasible for determining ^{222}Rn activity in aquatic environment.

3.4 *Natural waters sample measurements*

For groundwater samples and surface seawater samples collected from the Jiaozhou Bay, the activity of ^{222}Rn in the parallel samples was tested with the PIC Radon Detector and a RAD7. The test results are shown in Table 1. Because ^{222}Rn concentration in groundwater is high, bottle volume size of 250 mL was used for groundwater sample analysis. Results of the PIC on the two parallel sample ^{222}Rn in groundwater were consistent with the RAD7 result but with lower uncertainty. In another occasion, two months later, we collected a second set of groundwater samples, and the test results of PIC were consistent with those of RAD7.

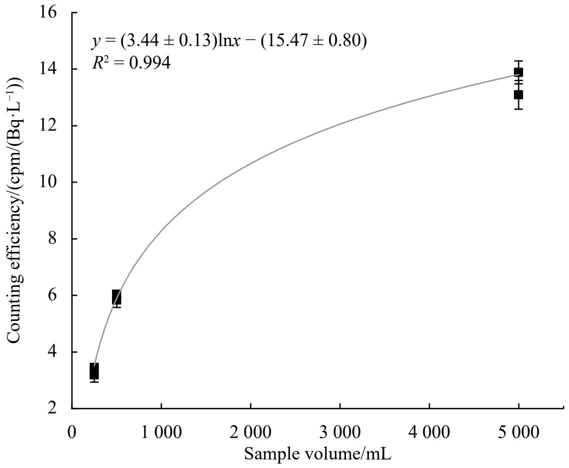

Fig. 5. Relationship between Pulsed Ionization Chamber counting efficiency and sample volume.

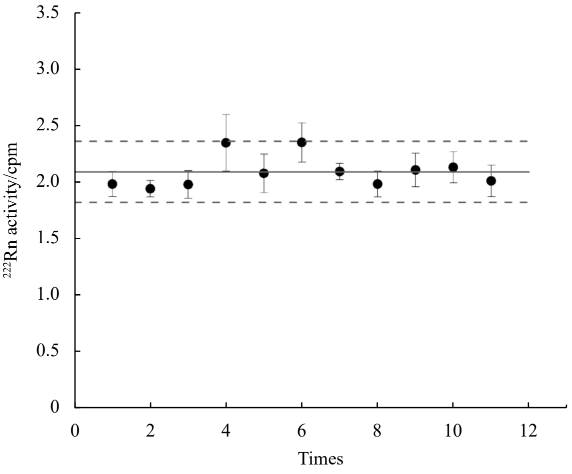

Fig. 6. Eleven tests of the same lab made water sample with water bottle volume of 500 mL and ^{226}Ra content of 0.2 Bq. The solid line represents the average value of 11 tests, and dotted lines represent two standard deviations (2σ).

Table 1. Comparison of ^{222}Rn measured by Pulsed Ionization Chamber Radon Detector and RAD7 in groundwater and surface seawater samples collected in the Jiaozhou Bay

Sample	Date (YYYY.MM.DD)	Sample bottle volume/mL	PIC/(Bq·L^{-1})	RAD7/(Bq·L^{-1})
Groundwater	2021.10.20	250	8.3 ± 0.6	8.5 ± 1.2
	2021.10.20	250	8.6 ± 0.8	8.5 ± 1.2
	2021.11.17	250	2.3 ± 0.2	1.7 ± 0.6
	2021.12.19	250	3.1 ± 0.2	2.7 ± 1.0
Surface seawater	2021.11.17	500	0.30 ± 0.03	0.3 ± 0.1

For surface seawater, considering low ^{222}Rn concentration and sufficient water volume for collection, water volume of 500 mL was used for sample collection and analysis. The ^{222}Rn concentration in the seawater sample collected in November 2021 measured by PIC and RAD7, and the result matched well with each other.

4 Discussion

We found that the detection limit of PIC Radon Detector used in this study is 0.07 cpm. According to the detection limit of the instrument and the efficiency value of the instrument under different volumes, when the sample volume is 250 mL, the detection limit of the measured aquatic ^{222}Rn concentration would be 0.021 Bq/L, which is feasible for almost all groundwater samples. In the case of sample volume of 500 mL, the detection limit of the instrument is 0.012 Bq/L, which could be used for groundwater ^{226}Ra measurement after secular equilibrium. If the sample volume increased to 5 L, the detection limit of the instrument would decrease to 0.005 2 Bq/L, which will be valid for most of the river, estuary and coastal sea settings. Therefore, when the PIC method applied to the actual sample in different ^{222}Rn concentration scenarios, the appropriate sample volume can be selected according to the approximate activity of the sample. Radon activity in groundwater is relatively high, ranging from a few to hundreds of Becquerel per litre. For example, radon concentration in groundwater of the lower Huanghe River ranges from 0.14–11.67 Bq/L (Zhang et al., 2018). In Tuticorin District of Tamil Nadu, the activity of radon concentration in groundwater in India ranges from 0.070–40.7 Bq/L (Singaraja et al., 2016). When testing such groundwater with high radon activity, the system with 250 mL sample volume can be selected for testing. Estuarine areas or inshore sea areas with low radon activity, such as the coastal areas of Vizhinjam, Thiruvananthapuram and Kerala, have a radon activity of (0.012 0±0.001 7) Bq/L (Jacob et al, 2009). The radon activity in the bottom water of the Changjiang River Estuary is 0.008 2–0.15 Bq/L (Guo et al., 2020). In the Yellow-Bohai Sea, the activity of surface water is 0.005 5–0.077 Bq/L, and that of bottom water is 0.005 7–0.062 Bq/L (Wang et al., 2021). A sample volume of 500 mL or 5 L can be selected for testing according to the radon activity in natural water. If the radon activity in water is very low, larger sample volumes can be selected for testing.

Drinking water is a fundamental resource for human life. ^{222}Rn is the most dangerous radon isotope due to its relatively long half-life and more quantity of radioactive decay products. Moreover, ^{222}Rn has long-lived decay products ^{210}Pb (22.3 a) and ^{210}Po (138.4 d) possessing long elimination periods in human body (Cantaluppi et al., 2021; Kelleher et al., 2017). The relationship between different volume efficiency of the PIC Radon Detector can be used to monitor ^{222}Rn in drinking water, which is of great significance for us to test the activities of radioactive radon in drinking water, avoid human exposure to radiation due to inhalation or intake of excessive radioactive substances, and protect people's health.

5 Conclusions

In this study, the PIC Radon Detector used has a low background value, and the radon in the gas path reaches equilibrium after 2 h of aeration. Through efficiency correction, the efficiency of the test system with water volume of 500 mL is 6.06 cpm for 1 Bq/L. The efficiency is related to the volume of the system volume, the counting efficiency of PIC and other factors. There will be differences in the test efficiency between different systems. Therefore, each set of the test system needs to be calibrated before testing samples.

Good logarithmic correlations were found between sample volume and efficiency. Water volume of 250 mL would be sufficient for analysis of most of groundwater samples. If the sample volume increased to 5 L, the PIC method will be valid for most of the river, estuary and coastal sea settings. According to the comparison analysis, there is no significant difference between PIC Radon Detector and RAD7 in sample test. Considering not affected by humidity and easy to operate, PIC Radon Detector is recommended to be used as the analysis instrument of ^{222}Rn in aquatic settings.

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