

*Ra-02-RC*

## **RADIUM-226 - EMANATION PROCEDURE**

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### **APPLICATION**

This procedure is specific and may be applied to almost any matrix which can be converted to a homogeneous solution.

Radium-226 in solution can be determined by de-emanating its  $^{222}\text{Rn}$  into an ionization chamber or scintillation cell for measurement. The  $^{222}\text{Rn}$  can be de-emanated by bubbling an inert gas through the solution, either after equilibrium has been established or after any known time period. Two half-lives, 7.65 days for example, give 75% of the maximum buildup.

### **SPECIAL APPARATUS**

1. Radon measuring equipment are described in Radon-222 in Air and Breath Samples, Procedure Rn-01-RC, and in Fisenne and Keller (1985).
2. Radon bubblers - see Specification 7.7.

### **SAMPLE PREPARATION**

1. The sample with barium carrier should be in homogeneous solution in a volume of 15-25 mL. The solution should be acidic with perchloric acid, neutral or in basic EDTA solution. Hydrochloric acid,  $\text{NH}_3$  or other volatile materials must be absent.
2. Detailed preparation procedures for separation of radium from sample matrices are presented in other radium procedures in this Manual.

3. Almost all types of samples require simultaneous analysis of reagent blanks, since radium appears in many chemical compounds. Using distilled or organic reagents reduces possible contamination.

## DETERMINATION

1. Transfer the sample solution to a  $^{222}\text{Rn}$  bubbler.
2. Clamp the bubbler in place to the  $^{222}\text{Rn}$  system.
3. Open the forming gas (85%  $\text{N}_2$ , 15%  $\text{H}_2$ ) tank valve and adjust the line pressure to 70 kPa (10 psi) gauge.
4. Bleed the gas line, the rotometer, and 9.5 mm diameter rubber tubing.
5. Adjust the flow through the rotometer to a rate of  $20 \text{ cm}^3 \text{ min}^{-1}$ .
6. Open both stopcocks on the  $^{222}\text{Rn}$  bubbler.
7. Attach the rubber tubing on the exit of the rotometer to the inlet of the  $^{222}\text{Rn}$  bubbler. Adjust the forming gas flow rate through the bubbler to  $100 \text{ cm}^3 \text{ min}^{-1}$ .
8. Flush the  $^{222}\text{Rn}$  from the solution (de-emanate) for 10 min.
9. Close both stopcocks simultaneously. Record the date and time as the beginning of the  $^{222}\text{Rn}$  build-up period.
10. Shut off the forming gas and remove the rubber tubing from the bubbler.
11. Place the  $^{222}\text{Rn}$  bubbler in a refrigerator for a suitable build-up period, usually 1 week.
12. To emanate  $^{222}\text{Rn}$  into a pulse ionization chamber, repeat Steps 2-4.
13. Attach the rubber tubing from the rotometer to the inlet of the measurement system.

14. Open the valve to the ionization chamber and close the vacuum valves. Partially fill the chamber with forming gas to a pressure of -2.7 (-50 cm of Hg) gauge. Close the chamber valve and system entry valve. Open the vacuum valves and detach the rubber tubing from the measurement system.
15. Attach a small drying tube containing about 5 g of a color indicating drying agent and 4 cm lengths of 9.5 mm diameter rubber tubing at both ends to the outlet of the bubbler and the inlet of the measurement system.
16. Open the system entry valve to evacuate the drying tube for 3 min.
17. Adjust the forming gas flow rate to  $20 \text{ cm}^3 \text{ min}^{-1}$  and attach the tubing outlet of the rotometer to the inlet of the bubbler.
18. Close the vacuum valves and open the ionization chamber valve.
19. Open the outlet stopcock of the bubbler and allow the bubbling to subside. Record the date and time as the end of the  $^{222}\text{Rn}$  build-up period.
20. Carefully open the bubbler inlet stopcock and adjust the forming gas flow through the bubbler to about  $100 \text{ cm}^3 \text{ min}^{-1}$ .
21. When the system gauge indicates that atmospheric pressure has been reached, close both bubbler stopcocks simultaneously. Close the chamber, system entry and forming gas valves. Open the vacuum valves.
22. Remove the bubbler and drying tube from the system. Record the date and time of the end of the emanation as the beginning of the next  $^{222}\text{Rn}$  build-up period.
23. Turn on the ionization chamber's high voltage. A red LED will light when the chamber is at or above atmospheric pressure.
24. Measure the sample for at least 17 h.

### Notes for Proper Operation:

1. If the Teflon stopcocks of the bubbler are snug fitting, it is not necessary to grease them. If they do leak, however, a silicone stopcock grease may be used.
2. The glass-to-glass ground joint at the top of the bubbler should also be lightly greased with silicone.
3. During the de-emanation, the gas bubbling should be started slowly to prevent popping of the bubbler top or stopcocks.
4. A better transfer of  $^{222}\text{Rn}$  from acid or neutral solutions is obtained if the bubbler is first chilled in a refrigerator. This apparently reduces the bubble size and improves the transfer efficiency. For samples dissolved in alkaline EDTA solution, a drop of octyl alcohol is added to reduce foaming.

### STANDARDIZATION

The chambers are standardized by de-emanating aliquots of a National Institute of Standards and Technology SRM  $^{226}\text{Ra}$  solution contained in  $^{222}\text{Rn}$  bubblers.

### DATA PROCESSING AND ANALYSES

1. For routine work, discard the first 5 h of counting data (equilibrium for  $^{222}\text{Rn}$  and progeny).
2. Determine the total count over the remaining counting period.
3. Calculate the net counts per hour per sample by determining the total sample counts per hour and subtracting the background counts per hour.
4. Divide the net counts per hour by the chamber standardization value of net counts per hour per Bq of  $^{222}\text{Rn}$ .
5. Using the midpoint of the counting interval as the time of counting, extrapolate the value obtained to the time noted for the end of  $^{222}\text{Rn}$  buildup. Figure 1 may be used for this calculation. This correction is only a few percent. The decay during the counting period is small and is not corrected for.

6. Correct the  $^{222}\text{Rn}$  measurement at the end of buildup for the fractional buildup. The figure in Procedure Rn-01-RC may be used to obtain the build-up factor.
7. Subtract the value obtained by carrying a blank determination through the full procedure.
8. Samples are measured twice and the mean and standard deviation of the duplicate emanation results are reported.
9. A computational data sheet is attached for handling routine calculations on a step-by-step basis.

LOWER LIMIT OF DETECTION (LLD)\*

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Counter Efficiency	(%)	57.5
Counter Background	(cps)	0.0028
Yield	(%)	90
Blank	(cps)	0.0012
LLD (400 min)	(mBq)	3.3
LLD (1000 min)	(mBq)	1.7

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\*Reagent blank must be analyzed with each set of samples.

## REFERENCE

Fisenne, I. M. and H. W. Keller  
"The EML Pulse Ionization Chamber Systems for the Measurement of Radon-222"  
USDOE Report EML-437, March (1985)

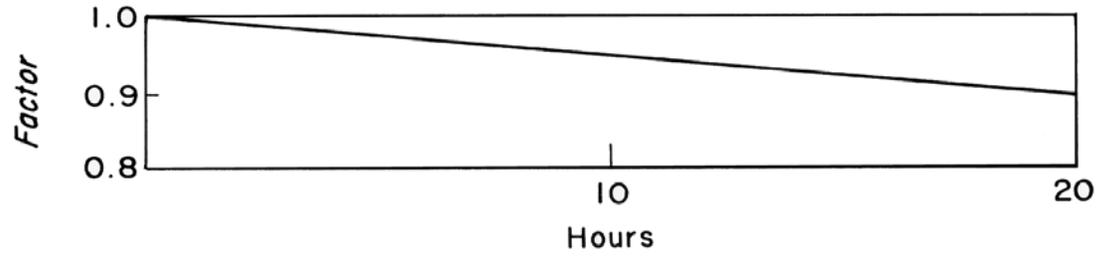


Figure 1. Correction factor from midpoint to the counting interval to the start of radon measurement interval.

Date \_\_\_\_\_

Sample Number

		Operation						
1	Chamber number	-						
	BACKGROUND							
2	Total count	-						
3	Count interval (h)	-						
4	Background (cph)	2/3						
	STANDARDIZATION							
5	Chamber factor cph Bq <sup>-1</sup>	-						
	SAMPLE COUNT							
6	Total count	-						
7	Count interval (h)	-						
8	Sample at count time (cph)	6/7						
	CORRECTIONS							
9	Time for midpoint of count	-						
10	Time for end of buildup	-						
11	Difference (days)	9-10						
12	Factor (from graph)	-						
13	Sample at end of buildup	8/12						

