

Calibration of Big Bottle RAD H₂O set-up for radon in water using HDPE bottles

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ABSTRACT

Glass bottles are generally employed for water sampling because glass is impervious to radon and is not lost during sample storage. On the other hand, glass is fragile and may break, so 1L High Density PolyEthylene (HDPE) bottles (Thermo Scientific Nalgene) are tested in place of glass vessels employing Big Bottle RAD H₂O device (DurrIDGE Company) coupled with RAD7 monitor. The purpose of this calibration is to quantify radon loss during storage in HDPE bottles, evaluate possible radon uptake by known volume of desiccant (Drierite, granular CaSO₄) and quantify radon interaction with the rubber and plastic parts of the experimental circuit. These processes have been separately investigated, performing proper experiments for the assessment of their influence on resulting radon data using seven series of solutions at known activity concentrations in the range from 27 to 194 Bq/L. Percent radon loss during storage in 1L HDPE bottles has been estimated at 0.0045 min⁻¹. Radon absorption by desiccant, expressed as ‘equivalent’ volume of Drierite is 0.673 ± 0.092 L and is somehow independent, within errors, from *i*) the amount of water already absorbed in Drierite, *ii*) a recirculation time greater than 30 minutes and *iii*) radon concentrations. Radon absorption/desorption from rubber and plastic parts of the experimental device has been assessed as a function of concentration gradient between the inner volume of the circuit and the pores of polymer’s. A final algorithm accounting for the above described physical processes has been developed for long runs (2-3 hours). A simplified calculation method for short measurements (30 minute) is also provided.

Keywords: radon; water radon; diffusion coefficient; radon absorption; RAD7 monitor; HDPE

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1. Introduction

Activity concentration of radon in groundwater is routinely measured using a variety of methods: Lucas cell, liquid scintillation, ionization chambers, gamma and alpha spectrometry of radon gas extracted by aerating the water sample.

Glass bottles are generally employed for sampling the water, especially when the measurement is not performed rapidly, but a delay may follow, because glass is impervious to radon and is not lost during sample storage. On the other hand, glass is fragile and the transport of glass bottles may induce the rupture, especially when springs or wells to sample are located along bumpy country or mountain roads. The need for shatterproof bottles led us to replace the 2.5 L glass bottles provided by Durrige Company Inc. with 1 L HDPE (High Density PolyEthylene) narrow-mouth bottles (Thermo Scientific Nalgene). They are 19.7 x 7.3 cm (height x outer diameter) with a cap size 2.7 x 4.3 cm (height x outer diameter). The mouth size and the screw closure are compatible with the holed cap and aerator used by Durrige in the standard Big Bottle RAD H₂O configuration. The choice of this kind of plastic is also due to its low gas permeability; actually HDPE barrier membranes are employed to prevent radon and vapor intrusion into residential and commercial buildings (Jiranek et al., 2008).

A calibration of Big Bottle RAD H₂O equipment coupled with HDPE bottles was then performed against an activated charcoal collector counted by gamma spectrometry and the radon chamber of INGV (Istituto Nazionale di Geofisica e Vulcanologia)-Roma using standard solutions at different radon activity concentration in order to: 1) account for radon loss during sample storage in HDPE containers; 2) evaluate the radon uptake by known volume of desiccant (granular CaSO₄); 3) quantify radon absorption/desorption by the rubber and plastic parts of the experimental system depending on the concentration gradient between air radon concentration in the closed-loop set up and radon concentration in the polymer's pores; 4) find an algorithm for the assessment of radon concentration based on the measurement provided by RAD7 in 30 minutes.

2. Materials and methods

Seven series of radon-containing solutions were produced and measured using the Big Bottle RAD H₂O device coupled with RAD7 radon detector (Durridge Company). Radon was collected from a radon source, injected in a water enricher and sampled. One or two water samples per series were also extracted for cross-calibration purposes using an activated charcoal collector counted by gamma spectrometry at INGV - Rome Radionuclides Laboratory.

2.1 Big Bottle RAD H₂O equipment

The Big Bottle RAD H₂O is an accessory designed for RAD7 radon detector (Durridge Company) that enables users to measure radon in 2.5 L water samples with high sensitivity. It consists of several components, a 2.5 L glass bottle (standard configuration), an aeration system, a bubble trap, a temperature data logger, vinyl tubings, a Laboratory Dryer and RAD7 (Fig. 1).

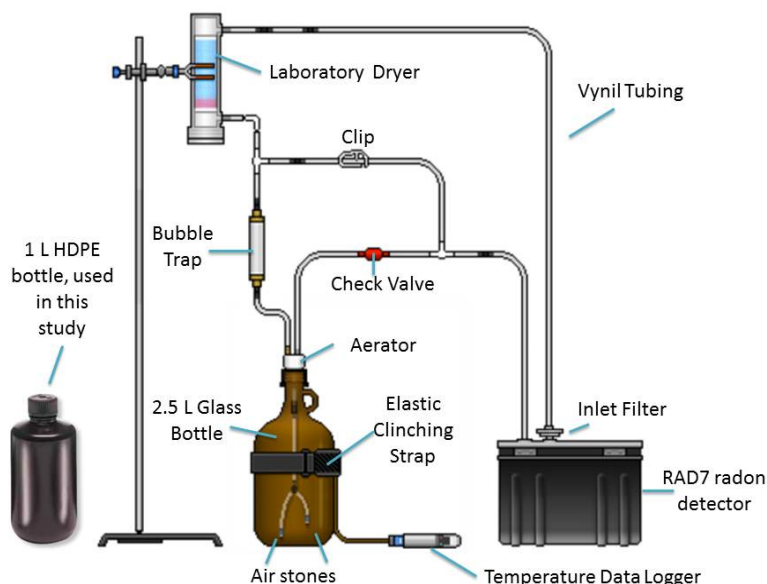


Fig. 1 - Configuration of Big Bottle RAD H₂O (modified from Big Bottle RAD H₂O manual, Durridge Company Inc., available at www.durridge.com). The standard 2.5 L glass bottle is here replaced with 1 L HDPE bottle. Durridge Co. is recently commercializing ‘soda’ bottles made from polyethylene terephthalate (PET, with a suitable adaptor) in place of glass bottles. PET bottles are appropriate to store radon-containing water because gas loss turned out to be even lower than that observed for HDPE and LDPE (Leaney and Herczeg, 2006).

RAD7 is a monitor that uses ^{218}Po , the short-lived daughter of radon (with a 3-minute half-life), without interference from other radiations. The detector is a solid-state, ion-implanted, planar, silicon electrostatic collector that measures the energy of alpha particles converting them directly to an electric signal. During the measurements the built-in pump runs continuously, aerating the sample and delivering radon to RAD7. The clip stays open during the whole run and the radon rich air flows through the closed-loop circuit, reaching an equilibrium with the radon remaining in the water. Eight to twelve 15-minute cycles are set. During the first 15 minutes RAD7 responds to the growth of radon in the measurement chamber, so the first cycle is ignored in the calculation. The radon extraction efficiency is calibrated against the average temperature of the air-water interface monitored about halfway up the bottle using the temperature data logger. Relative humidity detected by a probe placed inside the instrument is kept at about 5 %, because water molecules in the inner chamber influence the electrostatic collection efficiency of the detector.

2.2 Radon Chamber

Radon chamber available at INGV – Rome (Fig. 2, component A) is a 56 L stainless steel container with an alpha sensitive detector fitted across its cap. The detector is made of a scintillating flask (Fig. 2, component F), an Algade™ 120 cm³ pyrex glass bottle coated by polyethylene, optically coupled to a photomultiplier (Fig. 2, component E); electrical pulses are formed by a Tesys™ signal processing module (Mod. SPM 02) (Fig. 2, component D) and sent to a counter: a multichannel analyser (either a Silena™ Mod. Cato or an Ortec™ Multichannel PCI card) sets as multiscaler (Fig. 2, component L). Four air tight connectors (CPC, Colder Products Company) (Fig. 2, components B, C) are available to connect radon sensors in a closed loop; two of them (Fig. 2, component B) are connected to an internal pump to let air circulate through sensors working only for diffusion. In the chamber there is a fan (Fig. 2, component G) to ensure a uniform radon

concentration in the inner volume. The sensitivity of the detector for radon in the radon chamber is is $0.0174 \text{ cpm} / \text{Bq m}^{-3}$.

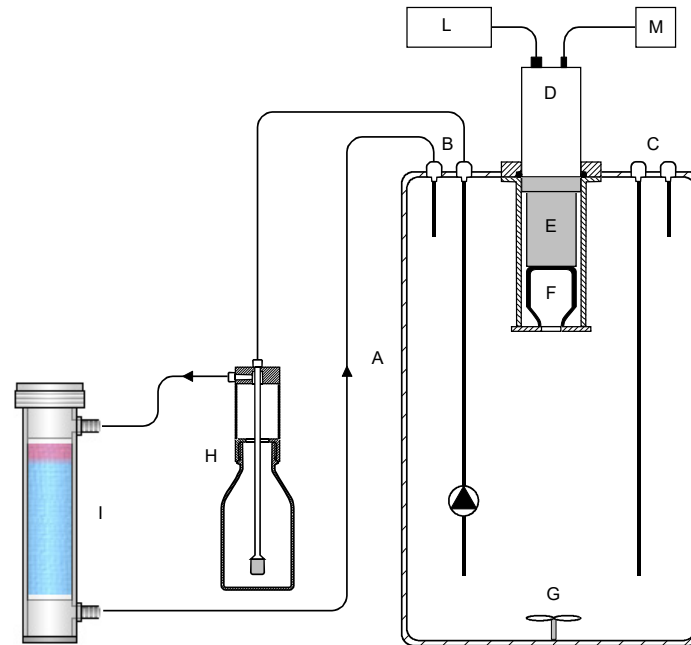


Fig. 2 – Experimental setup circuit for Drierite characterization. A) radon chamber; B) air tight connectors (CPC) to be used when air circulation needs a pump; C) air tight connectors (CPC) when air circulation is guaranteed by an outer device; D) signal processing module; E) photomultiplier; F) scintillating flask; G) fan; H) humidifying device; I) desiccant column (Drierite); L) multiscaler; M) low voltage supply.

2.3 Preparation of radon-containing solution

Seven series of radon-containing solutions were produced. Radon was collected from a radon source, a 300 cm^3 glass bottle containing 100 cm^3 of an acidified ($\text{pH} < 2$) liquid solution of RaCl_2 , activity 3.5 kBq , added with Ba, thus preventing significant radium precipitation. The bottle cap is properly modified to allow the extraction of radon gas by connecting a syringe to a CPC connector and unscrewing a little plastic cap to restore the needed amount of air in the source volume.

Then radon rich air was injected into a water enricher. This device (Fig.3) is made of a plexiglass cylinder, two PVC caps, the lowest being equipped with water proof connectors to i) supply or withdraw water, ii) supply radon gas and iii) supply electricity to a submerged pump. A floating

PVC structure, allowing to choose the amount of water to be enriched in the range 1 – 8 liters, has a cavity in which radon gas is trapped when injected from the lower PVC cap. Water is enriched by its continuous circulation upward where the radon gas is trapped.

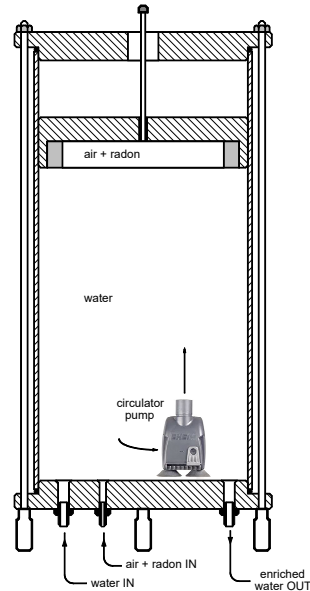


Fig. 3 – Water enricher.

Radon activity concentrations were: 27 ± 1 , 47 ± 2 , 78 ± 2 , 80 ± 3 , 126 ± 4 , 186 ± 5 and 194 ± 5 Bq/L. Three 1 L samples of each solution were sampled and measured using the DurrIDGE experimental apparatus described above, at increasing time from the sampling (1, 24, 48, 72 and 96 hours) to estimate radon loss during storage in HDPE bottles. One or two water samples per series were extracted for cross-calibration purposes using an activated charcoal collector counted by gamma spectrometry at INGV - Rome Radionuclides Laboratory (Galli et al., 1999).

3. Description of physical processes

From the analysis of plots showing radon concentration vs time, various physical processes affecting data, other than decay, obviously, have been identified:

- radon loss during storage in HDPE bottle;

- radon absorption by desiccant;
- radon absorption/desorption from rubber and plastic parts of the measuring system

These processes have been separately investigated, performing proper experiments for the assessment of their influence on the resulting radon concentration value, leading also to corrected 'equivalent' volumes to be used in calculations.

3.1 Radon loss during storage in HDPE bottle

High Density PolyEthylene is a thermoplastic material made from petroleum. Known for its large strength to density ratio, HDPE is commonly used in the production of plastic bottles, corrosion-resistant piping and geomembranes. Its density can range from 0.93 to 0.97 g/cm³.

Percent radon loss during sample storage in 1L HDPE NALGENE bottles (Fig. 4) has been estimated at $(6.5 \pm 0.3) \% d^{-1}$ (linear fit), but Figure 3 also provides an exponential fit of radon data closely corresponding to the linear interpolation. This loss is nearly comparable to that obtained by Leaney and Herczeg (2006) that stored water samples in 2.5 L HDPE Winchester bottle, even if their data indicate for the same storage time a slightly lower radon decrease, probably due to a lower surface/volume ratio.

The observed radon loss is probably due to the combined effect of diffusion through HDPE and of adsorption onto its surface during sample storage (Fernandez et al., 2004). The last process has been considered the basis for radon loss in water samples stored in polyethylene bottles by Saito (1983), whereas others (Arafa et al., 2002; Jiránek et al., 2008; Ashry et al., 2011) working on waterproof radon membrane mainly refer to diffusion. Following this view the estimated radon loss would correspond to a radon diffusion coefficient of $1.85 \cdot 10^{-12} \text{ m}^2/\text{s}$, considering that outer surface area of the bottle is about $5.04 \cdot 10^{-12} \text{ m}^2$ and its thickness about 2 mm. This result is in the order of the values reported in the literature for polyethylene membrane: $4.6 \cdot 10^{-12} \text{ m}^2/\text{s}$ (Arafa et al., 2002;

Ashry et al., 2011) and $5.8 \cdot 10^{-12} \text{ m}^2/\text{s}$ (Jiránek et al., 2008). However the issue of radon adsorption and diffusion is very complex to address and quantify and is beyond the scope of this paper, so we will refer more generally to radon loss during sample storage, as reported also in Leaney and Herczeg (2006).

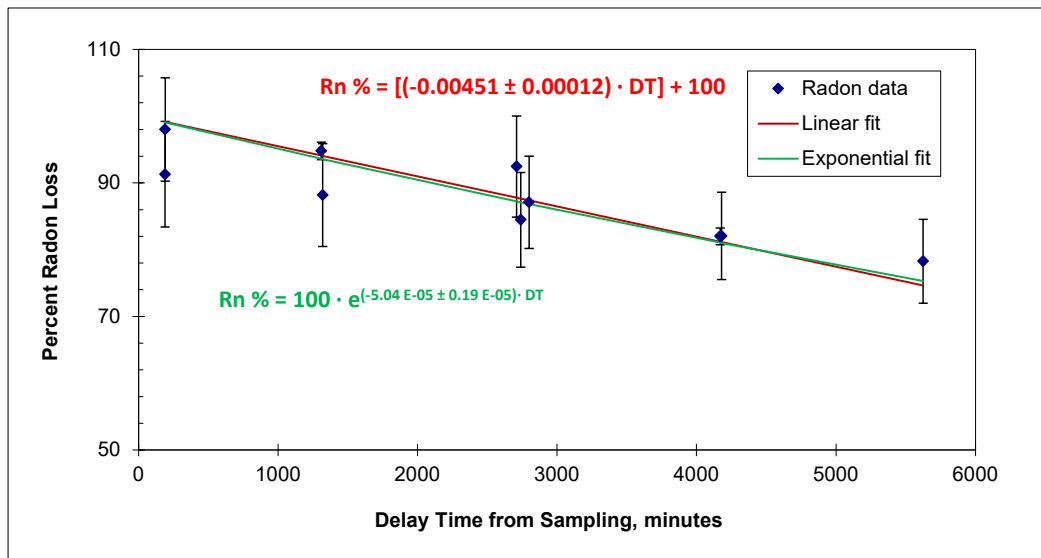


Fig. 4 – Percent radon loss during storage in 1L HDPE bottles manufactured by NALGENE versus delay time from the sampling.

3.2 Radon absorption by desiccant

The sensitivity of an electrostatic chamber used for radon measurements is inversely proportional to the absolute humidity and depends on the electrostatic field which drives charged radon daughters toward the solid state detector. The chamber geometry and the applied voltage define the electrostatic field; in order to strongly reduce variations in the collection of radon daughters on the detector surface, a desiccant (Drierite) is employed in the Big Bottle RAD H₂O instrument. The behavior of the desiccant column (Fig. 2, component I), a cylinder 29 cm high, with an outer diameter of 6.5 cm containing a Drierite volume approximately of 435 cm³, has been investigated by connection in a close loop with a humidifying device (Fig. 2, component H) and a radon

chamber (Fig. 2, component A). As a result the 'equivalent' desiccant volume has been obtained, i.e. a volume which accounts for radon absorption in Drierite.

Experiments have been carried out at various high radon concentrations, flux times and quantities of water already absorbed by Drierite, to assess the 'equivalent' Drierite volume as a function of the above mentioned parameters. Also the behavior of connection tubes in the circuit has been investigated and their volume per linear meter per minute determined to account for the other path through which radon is lost.

A description of a typical experiment follows. After injection of radon in the chamber, its internal fan was switched on to ensure a uniform radon distribution and in a five hour time radon also diffused to the scintillation cell and the secular equilibrium between radon and its daughters was reached. Then the fan was switched off and the experimental apparatus was connected to the radon chamber; the internal pump was switched on and the radon enriched air started fluxing through the humidifying device (a glass bottle employed in a radon degassing unit), then entered the desiccant column and went back to the radon chamber. Each experiment lasted for a time ranging from 30 minutes to 2 hours, then the apparatus was disconnected and the fan switched on again; finally at least 12 hours were waited for establishing a new equilibrium in the radon chamber and for the acquisition of data needed for calculation of the radon concentration in the chamber at the time when the humidifying device and the desiccant column were disconnected.

Concentrations in the chamber before and after each experiment were calculated through exponential fit of available data or by average of concentration values corrected by proper decaying factors (Fig. 5).

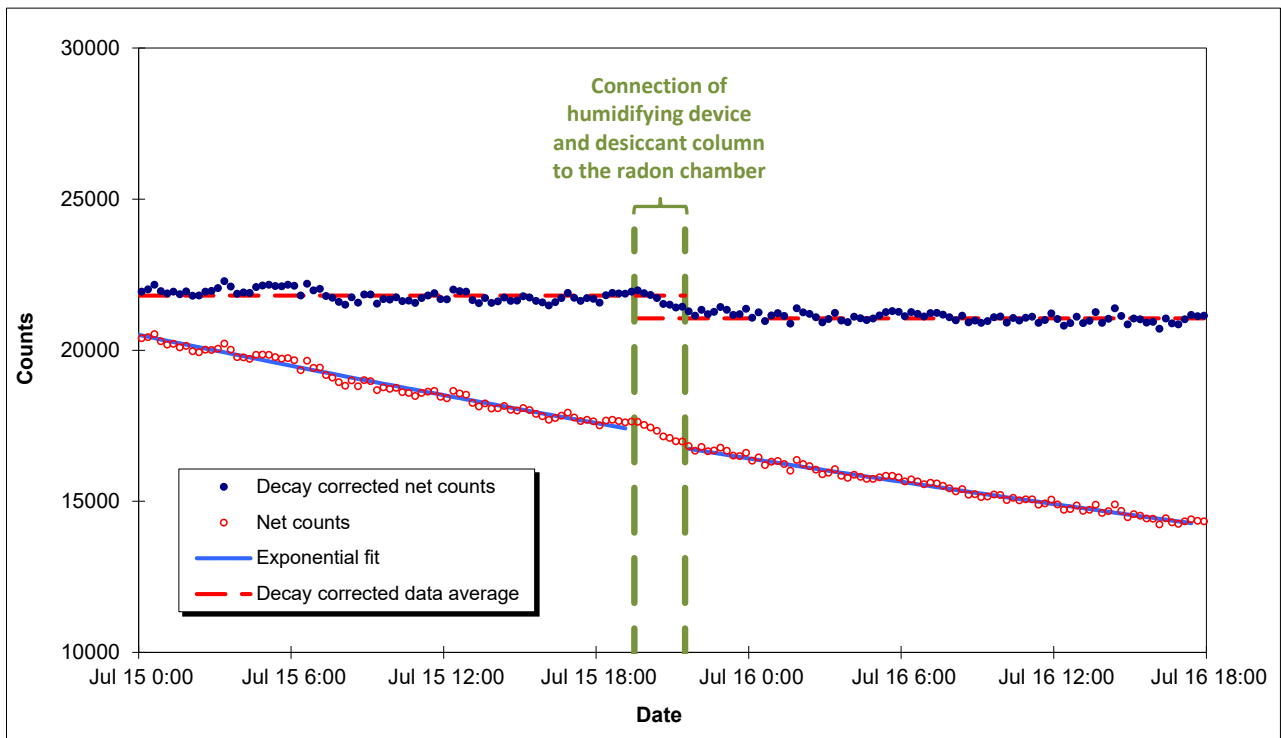


Fig. 5 – Example of data acquired by the multichannel analyzer during the experiment for the determination of the drierite ‘equivalent’ volume.

Given the volume of the radon chamber and that of the humidifying device ($\alpha(T)V_w + V_a$), where $\alpha(T)$ is the solubility of radon in water, V_w is the water volume and V_a is the air volume in the device, on the basis of the free volume in the desiccant column and of the ‘equivalent’ volume of the connection tubing, the ‘equivalent’ Drierite volume was calculated. Data, reported in Table 1, are grouped and eventually repeated to focus on the equivalent volume values as a function of the different investigated parameters. Average equivalent volumes are also reported, showing a limited variability within the errors. The average Drierite equivalent volume obtained without data replication is 0.673 ± 0.092 L.

Table 1 – Effect of fluxing time, water gain and radon concentration on Drierite equivalent volumes. Letters identify different experiments.

		<i>Equivalent volume</i> <i>(1E-03 m³)</i>									
		<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>	<i>G</i>	<i>H</i>	<i>I</i>	<i>Average</i>
<i>Time</i> <i>(min)</i>	30	0.728	0.649	0.848	0.701	0.665	-	-	-	-	0.718 ± 0.079
	60	0.556	0.760	0.623	0.638	0.676	0.825	0.545	0.661	0.552	0.648 ± 0.096
	120	0.634	0.642	0.695	0.566	0.656	0.848	-	-	-	0.674 ± 0.095
<i>Water</i> <i>gain (%)</i>	0 - 4	0.556	0.760	0.623	0.638	0.676	0.825	-	-	-	0.680 ± 0.098
	5 - 35	0.545	0.728	0.649	0.848	0.701	0.634	0.642	0.695	-	0.680 ± 0.088
	36 - 50	0.661	0.665	0.566	0.656	-	-	-	-	-	0.637 ± 0.047
<i>Rn</i> <i>(kBq/m³)</i>	51 - 65	0.552	0.848	-	-	-	-	-	-	-	0.700
	0 - 20	0.638	-	-	-	-	-	-	-	-	0.638
	20 - 40	0.556	0.760	0.623	0.661	0.552	0.656	-	-	-	0.635 ± 0.077
	40 - 60	0.825	0.545	0.649	0.848	0.701	0.696	0.566	0.848	-	0.710 ± 0.121
	60 - 80	0.676	0.728	0.665	0.634	0.642	-	-	-	-	0.669 ± 0.037

3.3 Radon absorption/desorption from rubber and plastic parts of the measuring system

Radon absorption/desorption from rubber and plastic parts of the DurrIDGE measuring system is assessed by analyzing plots of concentration values vs time, as shown in Figs. 6a, 6b and 6c.

Figure 6a shows radon data of a water sample measured after a long purging of the experimental apparatus with radon free air. The decrease of detected activity concentration during the 3 hour run, not justified by the concurring radioactive decay, radon loss during storage in HDPE bottles and continued drierite absorption, is largely due to radon absorption from the plastic and rubber elements within the circuit when the concentration gradients are large. If a second sample of the same solution is measured, after a 30 minute purging (Fig. 6b), the air radon concentration gradients between the closed-loop set up and the polymer's pores is much lower because the pores already contain the gas, resulting in data point aligned along a gently sloping line. This indicates that radon gas is slightly absorbed from plastic or rubber elements. Figure 6c reports another scenario when a low radon solution is degassed and measured using the same experimental set-up, previously

employed for other experiments. In this case the gradient is reversed and the circuit releases the radon amount trapped in the polymer's pores, displaying a radon rising trend during the 2 hour run.

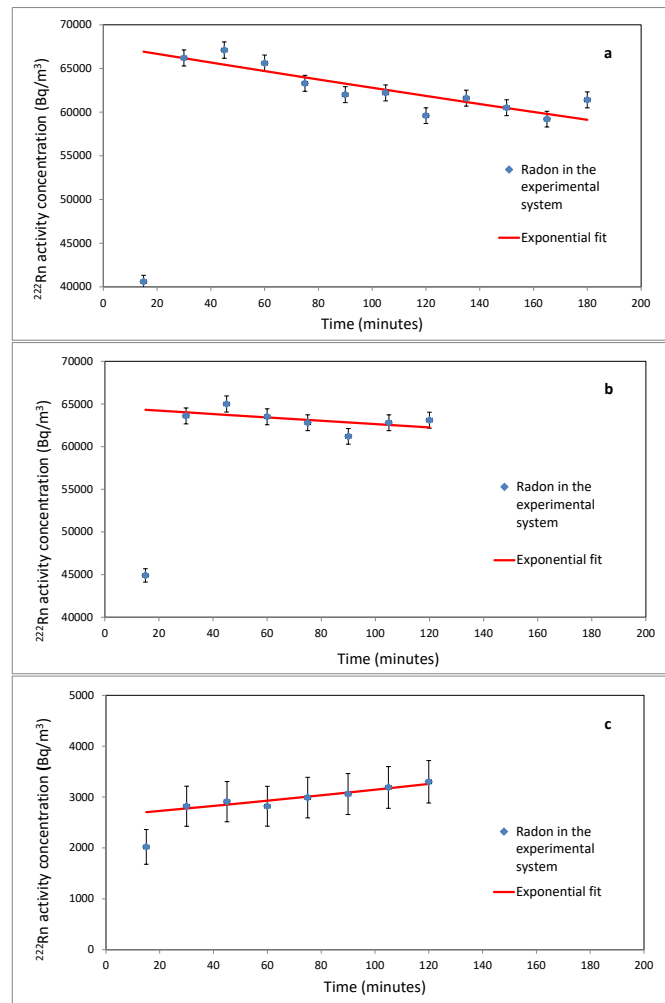


Fig. 6 –Radon activity concentration detected during 2 or 3 hour long measurements using the Big Bottle RAD H₂O device. Radon data decrease (a), stay almost constant (b) or increase (c) during the run as a function of concentration gradients between radon in the closed-loop set-up and in the polymer's pores.

By exponential fit of experimental data acquired from 30 to 120 – 180 minutes, we obtain a time constant (τ) which accounts for the radon decay (τ_{Rn}), loss during storage in HDPE bottle (τ_{HDPE}) and for the absorption/desorption from rubber and plastic parts of the measuring system (τ_{DS}). Being known the constants of radon decay and radon loss during sample storage in HDPE bottles, the absorption/desorption time constant can be calculated, thus a correction to the radon concentration provided by RAD7 instrument for the first 30 minutes can be carried out :

$$\frac{1}{\tau} = \frac{1}{\tau_{Rn}} + \frac{1}{\tau_{HDPE}} + \frac{1}{\tau_{DS}}$$

$$\tau_{DS} = \frac{\tau \cdot \tau_{Rn} \cdot \tau_{HDPE}}{\tau_{Rn} \cdot \tau_{HDPE} - \tau \cdot \tau_{HDPE} - \tau \cdot \tau_{Rn}}$$

4. Calculation model

An equation accounting for the above described physical processes has been developed for the calculation of the dissolved radon concentration using the DurrIDGE Big Bottle RAD H₂O instrument and 1 L HDPE bottles. A comparison with an already characterized method has been performed: for each radon enriched solution to analyse with the DurrIDGE Big Bottle RAD H₂O instrument one or two water samples were extracted at the same time for cross-calibration purposes by using a radon degassing unit in which radon dissolved in water is transferred to an activated charcoal collector, then counted by gamma spectrometry at INGV - Rome Radionuclides Laboratory (Galli et al., 1999). Finally a simplified calculation method is provided for 30 minute measurements.

4.1 Detailed model

Radon concentrations in water were obtained with the DurrIDGE Big Bottle RAD H₂O instrument by applying the following equation, where background concentration in recirculating air is negligible:

$$C_w = C_a FIT30 AF (V_a + \alpha(T)V_w) / (LB DS DF V_w)$$

with

$$V_a = V_{R7} + V_d + V_t + V_b$$

where:

C_w : original radon concentration in the water (Bq/m³)

C_{aFIT30} : radon concentration value at $t = 30$ min of an exponential fit of RAD7 data recorded during each 15 min run (from 30 to 120/180 min) (Bq/m^3)

AF : adjustment of instrument calibration factor (1.038 in this case)

T : temperature of water in bottle ($^{\circ}C$) (see Fig. 1)

$\alpha(T) = 0.105 + 0.405 e^{-0.0502 T}$: equilibrium coefficient from Fritz von Weigel equation (Weigel, 1978)

V_w : volume of water in bottle ($1.02E-03 m^3$)

V_{R7} : internal volume of the RAD7 ($0.768E-03 m^3$)

V_d : equivalent desiccant column volume ($0.673E-03 m^3$)

V_t : volume of tubing & aerator ($0.054E-03 m^3$)

V_b : volume of bubble trap ($0.051E-03 m^3$)

V_a : total volume of air in the system ($1.546E-03 m^3$)

LB: loss factor during sample storage in HDPE bottle as a function of storage time ($= 1 - 0.0045t/100$ or $= e^{-5.04 E-05 t}$, being t (min) the time elapsed between water sampling and the beginning of the run)

DS : absorption/desorption factor from rubber and plastic parts of the measuring system ($= e^{-t/\tau_{DS}}$, being $t = 30$ minutes - see section 3.3)

DF : Decay Factor ($= e^{-t/\tau_{Rn}}$, where t (min) is the time elapsed between water sampling and 30 minutes after the beginning of the run and τ_{Rn} (min) the radon average life)

Radon concentration data from each experiment are reported in Table 2, showing a good match with values obtained with a reference calibrated method. Only in the experiment at 78 Bq/L, with four bottles used instead of three, two values differ more than one standard deviation from the reference

value, maybe due to the insufficient water recirculation responsible for radon dilution by air during the sample collection.

Table 2 – Comparison of radon activity concentration determined using INGV method, Big Bottle detailed method and Big Bottle simplified 30 minute method.

<i>Rn (Bq/L) INGV method</i>	<i>Rn (Bq/L) Big Bottle detailed method</i>	<i>Rn (Bq/L) Big Bottle simplified 30 minute method</i>
194 ± 5	193 ± 10	193 ± 11
	180 ± 10	176 ± 10
186 ± 5	180 ± 9	179 ± 10
	180 ± 10	183 ± 11
	126 ± 7	125 ± 7
126 ± 4	123 ± 7	124 ± 7
	126 ± 7	125 ± 7
	79 ± 4	79 ± 4
80 ± 3	81 ± 4	79 ± 5
	79 ± 4	79 ± 5
	75 ± 4	74 ± 4
	71 ± 4	72 ± 4
78 ± 2	70 ± 4	72 ± 4
	77 ± 4	78 ± 5
	50 ± 3	50 ± 3
47 ± 2	46 ± 2	-
	48 ± 3	48 ± 3
	29 ± 2	30 ± 2
27 ± 1	29 ± 2	28 ± 2
	28 ± 2	28 ± 2

4.2 Simplified model

A simplified calculation method was developed to shorten the time required for the measurement, useful during geochemical surveys and/or for Drierite consumption. A minimum time of 30 min is needed, considering the RAD7 pump flow (~ 0.8 L/min), the volume of the water sample (~ 1 L), the ^{218}Po average life and an acquisition time of 15 minutes, referred to a single cycle. A correction coefficient (S) was introduced, the average of $(C_a \text{ FIT30} / \text{DS}) / C_a \text{ 30}$ ratio obtained from each experiment, being $C_a \text{ 30}$ the radon concentration provided by RAD7 at 30 min. Again, stated the background concentration in recirculating air is negligible:

$$C_w = C_{a\ 30} \text{ AF S } (V_a + \alpha(T)V_w) / (\text{LB DF } V_w)$$

where:

$C_{a\ 30}$: *equilibrium radon concentration in air provided by RAD7 at 30 min, acquisition time set to 15 min*
(Bq/m³)

S: *correction coefficient for simplified method (1.0176 ± 0.0170)*

Again, radon concentration data from each experiment are reported in Tab. 2, showing a good match with values obtained with the detailed Big Bottle and the reference calibrated methods.

5. Conclusions

A simplified method based on the measurement at 30 min to calculate dissolved radon concentration has been developed, based on the RAD7 Big Bottle setup replacing glass with HDPE bottles and considering various physical processes: radon loss during storage in HDPE bottles, absorption/desorption caused by diffusion through Drierite and circuit components.

In particular, the ‘equivalent’ Drierite volume is somehow independent, within errors, from *i*) the amount of water already absorbed in Drierite, *ii*) a recirculation time greater than 30 minutes (minimum time to have a response from the DurrIDGE Big Bottle RAD H₂O instrument) and *iii*) radon concentrations.

The main advantages of this method employed during geochemical surveys are *i*) fragile glass bottles have been replaced by HDPE bottles, *ii*) the time required for each measurement has been shortened and *iii*) the Drierite consumption has been reduced.

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