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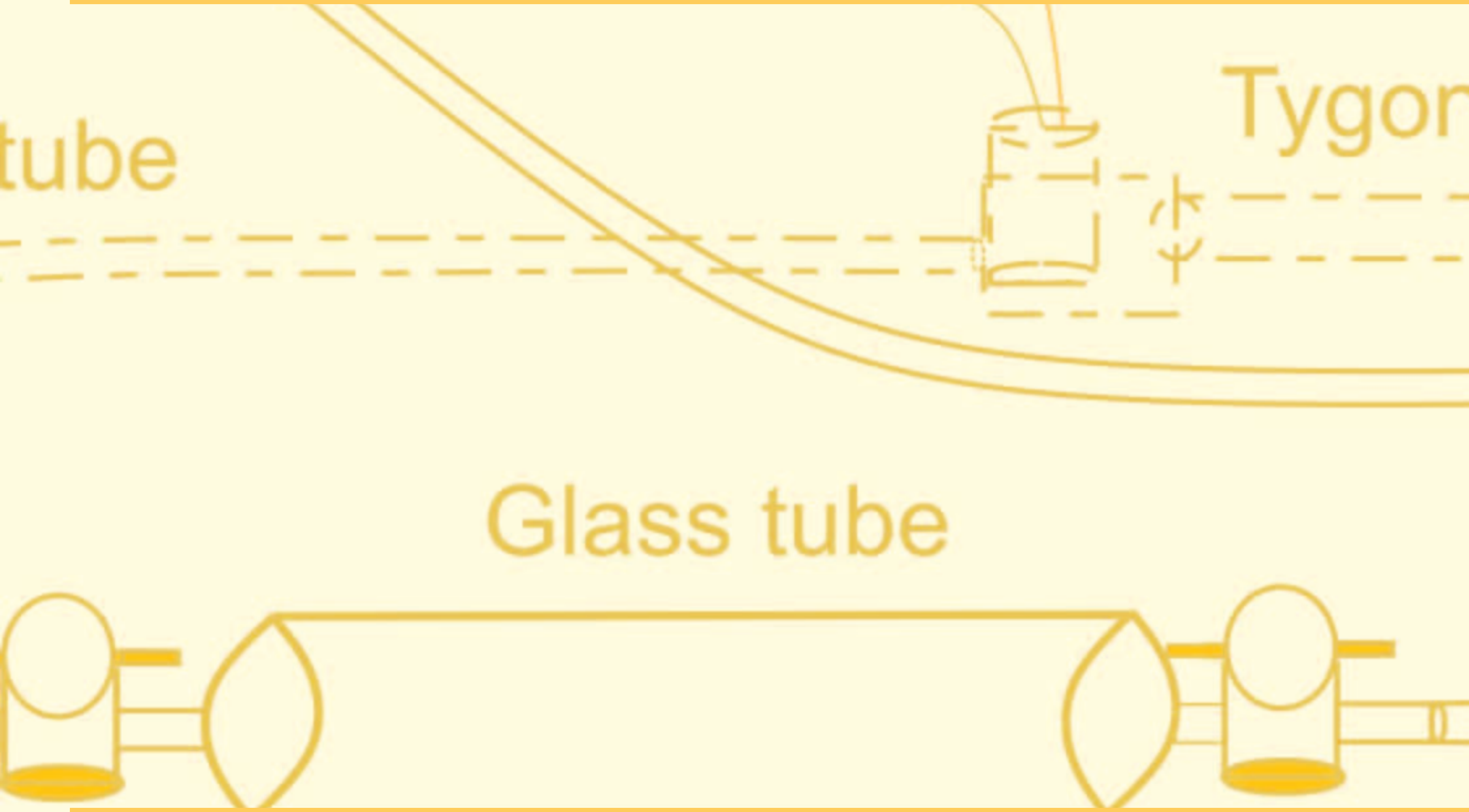

RAPPORTI TECNICI INGV

A procedure to use the RAD7 detector for measuring ^{222}Rn in soil gases exceeding instrumental limits: an application to chemically aggressive fumaroles of the Campi Flegrei area

473

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A procedure to use the RAD7 detector for measuring ^{222}Rn in soil gases exceeding instrumental limits: an application to chemically aggressive fumaroles of the Campi Flegrei area

Raffaella Silvia Iovine[1,*], Rosario Avino[1], Carmine Minopoli[1], Emilio Cuoco[1], Stefano Caliro[1], Gianfranco Galli[2], Monica Piochi[1]

[1]INGV | Istituto Nazionale di Geofisica e Vulcanologia, Sezione di Napoli - Osservatorio Vesuviano
[2]INGV | Istituto Nazionale di Geofisica e Vulcanologia, Sezione Roma 1

**Corresponding author*

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Abstract

This report proposes a proper-designed method for the RAD7 detector to measure ^{222}Rn in gases discharged from sulfurous fumaroles, tested at temperatures between 94°C and 162°C.
RAD7 is one of the most widely used commercial instruments for ^{222}Rn measurements, either dissolved in water or fluxing from soils, offering a reliable comparison of data acquired in different laboratories being already calibrated by the manufacturer. However, RAD7 cannot be used for measurements in hot and acidic gas soils.
The proper-designed method involves sampling of the gas in-situ in Tedlar® bags, transferring the gas into glass tubes and, finally, measuring the gas via a closed loop using the RAD7 after the removal of H_2S by proper traps.
This new method has been tested for measuring radon in the hostile environments of Solfatara and Pisciarelli in the Campi Flegrei volcano (Italy), providing the instrumental base for creating timely Rn content datasets.

Riassunto

La presente relazione propone un metodo opportunamente progettato per misurare il ^{222}Rn nei gas emessi dalle fumarole sulfuree, a temperature comprese tra 94°C e 162°C con il RAD7.
Il RAD7 è uno degli strumenti commerciali più utilizzati per le misure di ^{222}Rn dissolto nelle acque ed emesso dai suoli, in grado di offrire un confronto attendibile fra i dati acquisiti in diversi laboratori essendo già calibrato dalla casa madre. Il RAD7 però non può essere utilizzato per misurazioni del gas emesso in condizioni di alta temperatura e di estrema acidità.
Il metodo progettato prevede il campionamento del gas tal quale in sacche di Tedlar®, il suo trasferimento in tubi di vetro e, infine, la misurazione attraverso un circuito chiuso utilizzando il RAD7 dopo la rimozione dell'H_2S mediante apposite trappole. Questo metodo è stato testato per la misurazione del radon negli ambienti ostili di Solfatara e Pisciarelli nel vulcano dei Campi Flegrei, in Italia, ponendo le basi per la raccolta di datasets temporali del Rn.

Keywords Solfatara and Pisciarelli; Radon; Alpha detector | *Rilevatori alfa*

Introduction

^{222}Rn, an alpha-emitting radioactive noble gas with a half-life of 3.8 days, derives from the alpha decay of ^{226}Ra in the ^{238}U-^{206}Pb chain. Being generated in rocks and soils of the Earth's crust, the emanated fraction can move through the pore spaces and dissolve into underground and surface waters or reaching the atmosphere [Cecil and Green, 2000; Nazaroff, 1992]. Worldwide labeled as an ideal tracer of fluid transport at active faults and hydrothermal systems due to its characteristics, i.e. short-half life, inertness, high abundance (2–3 orders of magnitude) in groundwater than in surface water, sensitivity to sudden changes in subsurface conditions etc. [Sukanya et al., 2022 and references therein], radon has a wide range of applications in different fields of the geosciences, from seismology to volcanology to environmental tracking [Avino et al., 1999; Barbosa et al., 2015; Galli et al., 2019; Ghosh et al., 2009; Piersanti et al., 2015; Sabbarese et al., 2020; Tedesco et al., 1988].

In hydrothermal and geothermal areas, such as the Campi Flegrei volcano in the Campania region (Southern Italy), the study of Rn can be useful to evaluate preferential degassing pathways (i.e. fractures and faults), fluid ascent dynamics and hydrological circulation. At the fumarolic sulfureous field of Campi Flegrei (namely Solfatara and Pisciarelli) possible variations in Rn concentration, coupled with other gases commonly monitored like CO_2 and H_2S [Cardellini et al., 2017; Chiodini et al., 2010], may be of particular interest to track the hydrothermal dynamics and to evaluate the role of a fluid carrier having a sufficient efficiency, such as CO_2, to transport Rn towards the surface.
However, the high H_2S levels and temperatures existing at Solfatara and Pisciarelli sites do not allow direct measurements of Rn, because reaction of H_2S with oxygen will give sulfuric acid, and the acidic environment can damage the instrument (RAD7). This gave rise to develop a technique for sampling and measuring radon in fumarolic gases in critical areas.
In this report we present the developed instrumental setup to allow Rn measurements, and the first results obtained by analysing the ^{222}Rn concentrations in two fumarolic gases from Solfatara (Bocca Grande and Bocca Nuova) and one from Pisciarelli, at the Campi Flegrei volcano, testing a proper-designed method that involves sampling of the gas in-situ in Tedlar® bags, and in the laboratory transfer it into glass tubes and, finally, measures via a closed loop by using the RAD7 after the removal of H_2S by proper traps.
The activity has been performed in the frame of the "Pianeta Dinamico" project-Task V2 (2021) funded to Monica Piochi of the Istituto Nazionale di Geofisica e Vulcanologia (INGV) in Naples, Osservatorio Vesuviano, in order to employ Rn as geochemical tracer of the hydrothermal dynamics at the Campi Flegrei volcano. The project enabled the purchase of two RAD7® Radon-in-air detectors (Durridge Co) and related accessories for measuring radon in waters and soils.
A collaboration between the INGV Radionuclides Lab in Rome and the Geochemical Fluid Lab in Naples was established through the project participants to achieve the best performance and outcomes, with the support of the IRON (Italian Radon mOnitoring Network; https://www.ingv.it/en/monitoring-and-infrastructure/monitoring-networks/ingv-and-its-networks/iron) research group.

1. RAD7: the instrument

RAD7 (Figure 1a) is a silicon alpha detector that counts the pulses produced by the decay of ^{222}Rn short lived daughters, $^{218}Po^+$ and $^{214}Po^+$. The instruments can be connected to different accessories, e.g. Big Bottle RAD H2O and DRYSTIK for water analyses or soils gas probes for measurements in soils. It has several advantages compared to traditional alpha scintillation method, the most important, being its rapidity when used in *sniff mode* (only ^{218}Po is analyzed). Traditional methods, instead, require the transfer of samples to a scintillation cell and a long pre-measurement waiting time of ~ 3 h, needed for the radioactive equilibrium be established also with ^{214}Po, since it cannot be discriminated from ^{218}Po [Broecker, 1965; Key et al., 1979; Mathieu et al., 1988].
RAD7 can measure Rn along an open circuit or in a closed loop. The first method is used in situ for Rn measurements in soils, pumping the gas from the soil into the detection volume and expelling it after the measurement. The closed loop is generally used, in laboratory, for the dissolved Rn, connecting RAD7 to different accessories. RAD7 radon monitor uses an automatic pump, which drives the gas sample into RAD7 detection chamber (volume ~ 750 cc) and a solid-state detector that converts alpha radiation directly to an electrical signal, allowing to immediately distinguishing radon from thoron by the energy of the emitted alpha particles. Air is drawn to the RAD7 inlet first, through a small drying tube filled with drierite desiccant, and then the air from the outlet is recirculated into the RAD7 detection chamber.

Prior to start a measurement run, the instrument is purged (Test- Purge command) to remove i) the residual radon (and thoron) from the previous measurement and ii) the atmospheric moisture of the detection volume; through the "User Setup" menu the most frequently used parameters: *cycle*, *recycle*, and *mode* are configured. *Cycle* time represents how frequently the RAD7 takes a reading (the acquisition time); the *recycle* parameter refers to the total number of cycles within a complete run; *Sniff* mode is used in order to track rapid changes in radon concentration levels, since only ^{218}Po alpha decays (ignoring the subsequent and long-lived progeny) are counted. As a matter of fact, the 3 min ^{218}Po half -life allows to reach the radioactive equilibrium in around 15 - 20 min. For measures in fumaroles, we used 5-min cycles, 6 or 7 recycles and Sniff mode. After every cycle RAD7 automatically prints out a summary, showing the mean of ^{222}Rn (printed raw data; Figure 1b). At the end of each run, RAD7 prints out a summary, showing the average radon readings from all the recycles, a bar chart of the total readings, and a cumulative spectrum, which is stored for later analysis. After the run, RAD7 is connected via a serial USB adapter to a PC and the run data are downloaded by Capture, the RAD7 software tool produced by Durridge Co (values must be recalculated for radon dissolved in water samples). A typical cumulative spectrum obtained from the software is presented in Figure 1c.

For laboratory analysis, decay correction is applied for the time lag between sample collection and measurement, considering only Rn concentrations inferred from ^{218}Po after the radioactive equilibrium is reached (i.e. recycles 4-5-6 and sometimes 7, also for statistical reasons regarding the average value).

The equipment was set up by performing numerous tests for the measurement of Rn concentration in waters and soils. In addition, the two RAD7s used in our study were characterized at Radionuclide Laboratory of the INGV – Sezione Roma 1, following the procedure described in Galli et al. [2019], which involves a combined analysis of the response of all the sensors (silicon detector, thermometer and hygrometer) within the instrument.

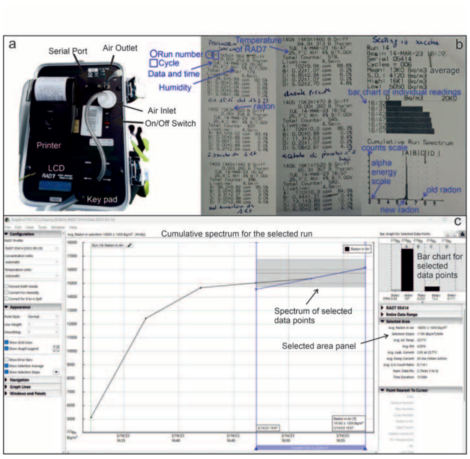

Figure 1a RAD7 instrument. Note the inlet and outlet valves for gas; **1b** example of printouts with explanation of the significant parameters; **1c** example of Capture graph with radon content in air gas at each recycle. The dashed area is related to the last three stable measurements.

2. Procedure adopted for sampling and measuring gas from fumaroles

2.1 Rationale

The RAD7 manual reports the possibility to perform soil gas measurements by taking the gas in a Tedlar® bag of 5 liters, using the Sniff mode linking the bag to the inlet, and circulating the gas in the instrument toward the outlet. Based on this indication, and considering the possibility to do measurement in a closed loop, we established a method to filter H_2S species and measure the fumarole gases in the lab. Figure 2 reports the essential phases, summarized as follows:

- at fumaroles sites, sampling the gas in Tedlar® bag of 1 and 3 liters in order to have the possibility to repeat the measurements two or three times to verify the accuracy of the data.
- In laboratory, preparation of H_2S traps by filling silicone tubes with lead acetate powder and closing at both ends with hydrophilic cotton.
- Transferring fumarole gas from Tedlar® bag into a glass tube following three steps: a) glass tube under vacuum; b) gas from the sampling bag to the glass tube; c) distributing gas into the entire circuit.
- Connection of the glass tube to the RAD7 in a closed loop for analysis after RAD7 calibration.
- Rn printouts obtained from RAD7 and corrected for the time lag between sampling and measurement.
- Comparison between RAD7 and charcoal measurements to compare the results obtained through two different methodologies.

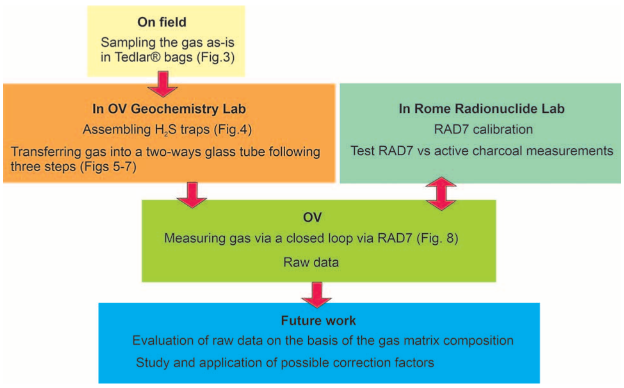

Figure 2 Steps in the proper-designed method for Rn measurement in gas fumaroles.

2.2 Sampling method

Gas is sampled according to the conventional method suggested by Giggenbach [1975]. A stainless tube (Figure 3) is completely dug into the fumarole, where temperature is close or above 100°C, in order to keep a satisfactory gas flow without cooling. Gas is led to a condenser through insulated tubes that carry the gas to a sampler at a proper distance from the vent, at a temperature as close as possible to the source one. Gas is cooled down through a condenser, flown by a refrigerant liquid, and sampled in the air-tight Tedlar® bag (1-3 liter), composed of a single fitting of inert polypropylene that combines the hose/valve and the septum holder. The purpose of the condenser is to obtain a fast and quantitative separation of non-condensable gases from the aqueous phase. To allow the sample container to be filled at a pressure above that of the atmosphere, a syringe is used (Figure 3).
Samples are labelled with sample location, date and time to later correct for the decay during the time interval between sampling and analysis at the Osservatorio Vesuviano.
The three main fumaroles at the Campi Flegrei, i.e., Bocca Nuova and Bocca Grande within the Solfatara crater, and Pisciarelli on its NE slope, have been sampled repeatedly: 11/2021, 12/2022, 03/2023, 04/2023, 05/2023, 06/2023 and 07/2023, and some analysed were repeated in duplicate or triplicate.

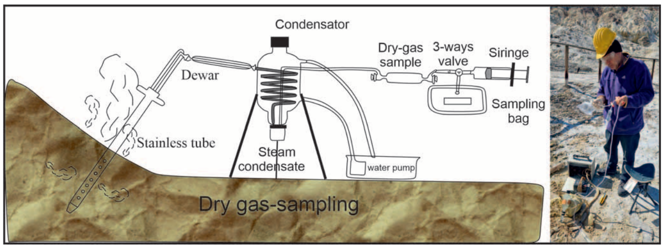

Figure 3 Scheme for sampling gas from fumaroles; on the right, operations at the Solfatara crater.

Temperatures of the sampled gases were about 144°C at Bocca Nuova, 162°C at Bocca Grande, and 94.3°C at Pisciarelli. Water vapour was the dominant gas, accounting to more than 85%; there were no other condensable phases. Among the incondensable species, CO_2 was at 98-99%, H_2S less than 2% and CO, N_2 and CH_4 were present in minor abundances.

2.3 Assembling H_2S traps

Removal of hydrogen sulfide (H_2S) from the sampling bag is crucial, because this compound can cause corrosion and damages to instruments even at low concentrations. H_2S traps are prepared operating under a laminar flow hood; they consist of silicone tubes filled with lead acetate ($Pb(CH_3COO)_2*3H_2O$) powder (hereafter ACETATE TRAP in Figures 4 - 8) and closed at both ends with hydrophilic cotton (Figure 4). The lead acetate is hydrophilic and was pulverized by an agate mortar immediately before its introduction in the silicone tube. Two or three consecutive traps were used to prevent H_2S entering the RAD7.

Figure 4 H_2S traps, made of lead acetate powder filled into silicone tubes. Note the colour of the traps: darkish on the right one due to the interaction with H_2S from the incoming gas, and whitish on the left one after complete hydrogen sulphide removal.

2.4 Transferring gas from sampling bag to glass tube

2.4.1 1st step: glass tube under vacuum

The fumarole gas must be transferred from the Tedlar® bag into a glass tube equipped with two valves, needed for handling the closed measurement circuit. The gas container tube is made of borosilicate glass and has the volume of the Tedlar® bag sampler (1 or 1.2 liters). The transfer of gas from the Tedlar® bag is possible after creating vacuum in the glass tube. One of the H_2S traps is connected to the outlet of the glass tube. The glass tube and the lead acetate are attached to a battery supplied diaphragm pump (hereafter pump) via two 3-way valves. The 3-way valve after the lead acetate trap (hereafter A) is also connected to RAD7. The other 3-way valve (hereafter B), between the glass tube and the pump, is linked to the vacuum rotary pump through a silicon tube, fitted with a filter array (Figure 5).

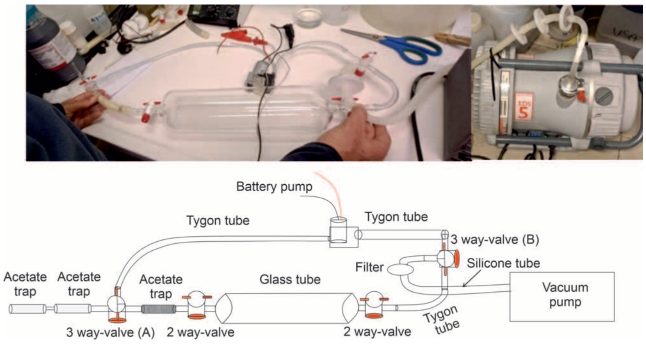

Figure 5 1st step: configuration for the glass tube to be under vacuum. On the right the vacuum rotary pump needed to create vacuum in the glass tube.

2.4.2 2st step: gas from the sampling bag to the glass tube

By closing the A valve towards the last two acetate traps (Figure 5) and turning on the vacuum rotary pump for about 1 minute, the glass tube will be free of air; then, by attaching the Tedlar® bag sampled at the fumarole and opening the bag valve, gas will flow from the bag to the glass tube (Figure 6).

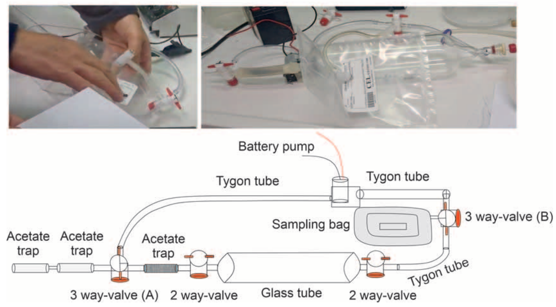

Figure 6 2nd step: gas transfer from the Tedlar® bag to the glass tube. Note the A valve closed towards the external way.

2.4.3 3rd step: distributing gas into the entire circuit

Closing the way at the valve toward the Tedlar® bag, it is possible to remove the bag. The A and B valves are now closed towards the external circuit and it is now possible to run the pump for about 5 minutes. Within a few minutes, the pump will keep the gas circulating through the entire circuit and H_2S will be trapped by the lead acetate (Figure 4), which will turn dark. At this point by closing all valves (configuration in Figure 7), the glass tube is ready to be connected to the RAD7 line for analysis.

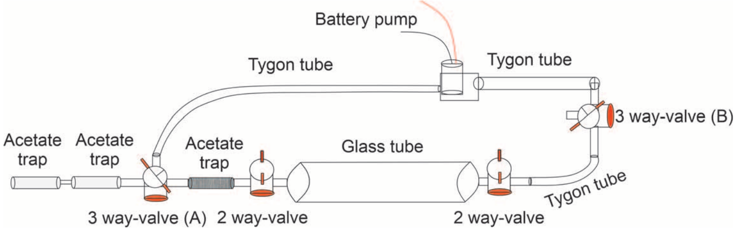

Figure 7 3rd step: scheme showing the configuration before measurement at RAD7. Note the A and B valves closed.

2.5 RAD7: ^{222}Rn measurement

RAD7 setup consists of 6/7 recycles, SNIFF protocol (only ^{218}Po alpha decays are counted) and pump in AUTO mode. The closed loop between the RAD7 and the glass tube is reported in the scheme of Figure 8. The line across the pump is excluded. So, the A and B valves are connected to the RAD7: B at the outlet and A at the inlet preceded by a dust filter and a small drying tube to prevent any dust and humidity from reaching the instrument, respectively. Note that two additional lead acetate traps are connected as a precaution between the A valve and the dust filter.
Measurement will start upon the A, B and 2-way valves opening along the main circuit (i.e., excluding the pump path); the RAD7 pump will keep the gas circulating. In any case, measurements must be stopped at the complete darkness of the acetate trap before the dust filter, if eventually it will occur.
Rn printouts are obtained from RAD7. We therein adopt the criteria established for measurements in soil gas: the 1° recycle is not considered; the average value of recycles 2 and 3 is used to obtain the Rn-220 (thoron) concentration, the average value of recycles 4 – 5 – 6 (and 7) is used to obtain the Rn-222 (radon) concentration. Due to the low concentration and fast-decay, the sampled gas has virtually no Rn-220.

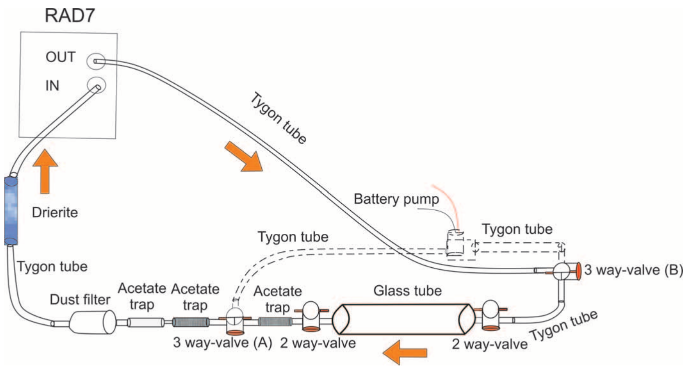

Figure 8 Configuration for connecting the glass tube to RAD7 to measure radon gas sampled from fumaroles. Arrows indicate the direction of gas flow. The dashed part is excluded from the flow path.

Results: ^{222}Rn raw concentrations

The raw Rn concentration corresponds to the above average value of recycles 4 – 5 – 6 (and 7) corrected for the time lag between sampling and measurements (Table 1).
^{222}Rn raw concentrations of fumaroles sampled over time vary from 2026 Bq/m^3 (Bocca Grande, sampled in November 2021) to 25077 Bq/m^3 (Pisciarelli, sampled in December 2022).

Locality	Sampling date and time	^{222}Rn (Bq/m^3)	2σ (Bq/m^3)
Bocca Nuova	02/11/2021	4127	774
Bocca Nuova	01/12/2022	4479	846
Bocca Nuova	13/03/2023	5699	927
Bocca Nuova	03/04/2023	3900	730
Bocca Nuova	03/04/2023	4125	745
Bocca Nuova	04/05/2023	4131	856
Bocca Nuova	04/05/2023	4299	816

Bocca Nuova	05/06/2023	2975	645
Bocca Nuova	05/06/2023	2192	613
Bocca Nuova	10/07/2023	2133	575
Bocca Nuova	10/07/2023	4095	748
Bocca Nuova	10/07/2023	3241	656
Bocca Grande	02/11/2021	2026	667
Bocca Grande	02/11/2021	3703	788
Bocca Grande	02/11/2021	3564	783
Bocca Grande	01/12/2022	4147	806
Bocca Grande	01/12/2022	4701	837
Bocca Grande	13/03/2023	3074	666
Bocca Grande	03/04/2023	3872	716
Bocca Grande	04/05/2023	4430	834
Bocca Grande	05/06/2023	2688	701
Bocca Grande	05/06/2023	2030	571
Bocca Grande	06/07/2023	2879	645
Bocca Grande	06/07/2023	2451	621
Bocca Grande	06/07/2023	2997	631
Pisciarelli	02/11/2021	17080	1650
Pisciarelli	01/12/2022	23987	1740
Pisciarelli	01/12/2022	25077	1800
Pisciarelli	13/03/2023	18683	1520
Pisciarelli	04/05/2023	20948	1640
Pisciarelli	05/06/2023	16270	1310
Pisciarelli	11/07/2023	15688	1480
Pisciarelli	11/07/2023	18249	1510

Table 1 ^{222}Rn raw concentrations of fumarolic gases sampled at discharge areas of Solfatara crater (Bocca Nuova and Bocca Grande) and slope (Pisciarelli), at the Campi Flegrei volcano in Italy.

Discussion

Raw Rn concentrations must be corrected by considering i) the whole volume of the loop (i.e. the described experimental line plus RAD7 measurement cell) to account for dilution, ii) the specific calibration for each instrument employed, and iii) the possible effect of the specific sampled gas matrix as well. For example a high CO_2 amount was indicated to decrease the response of the RAD7 silicon detector [Lane-Smith and Sims, 2013].
Anyway, the reported raw data allow us to gain information on relative variations occurred through time at the Campi Flegrei fumaroles.
Since the analysed gases have the same matrix, essentially made of CO_2, the other species (N_2, CO, He, CH_4) that may affect the RAD7 response are at a very low contents; therefore the raw data are useful indeed to evaluate differences among fumaroles and variations through time without further adjustments.
The results show a slight increase in 2022 compared to 2021 and 2023 (Table 1; Figure 9). As seen from Figure 9, at Pisciarelli there are greater variations than in the Solfatara area (i.e., Bocca Nuova and Bocca Grande), where concentrations remain quite constant during the analysed period. Also, Pisciarelli is much richer in radon respect to Solfatara crater.
Identify radon source is challenging matter, because radon contents in soils are dependent on

several factors, making difficult to assess the cause of its temporal and spatial changes. In particular, the Rn contents was found to be related to: i) atmospheric conditions, ii) temperature, humidity and permeability of specific site, iii) content of uranium or radium parents [e.g., Barbosa et al., 2015; Nazaroff, 1992; Sukanya et al., 2022]. Moreover, at active volcanoes, the radon contents in soils can also derive from the own dynamics of hydrothermal and magmatic system. Because no seasonality effects are recognized on the collected data, we suggest that observed changes in Rn content in the Solfatara and Pisciarelli fluids could be related to endogenous rather than exogenous (e.g., temperature fluctuation or raining) processes. If further data will support our advice, radon measurements can integrate other geochemical parameters to improve the knowledge and the monitoring of the Campi Flegrei volcanic system.

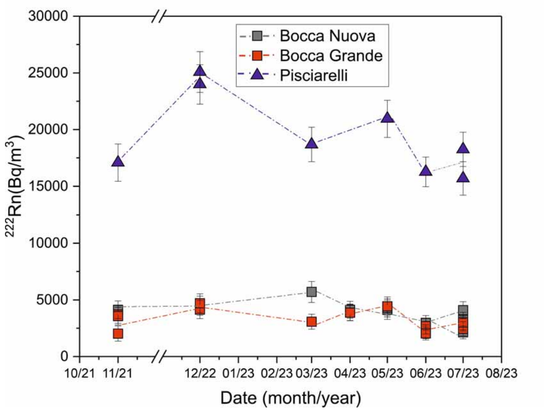

Figure 9 ^{222}Rn concentrations in gas fumaroles of the Campi Flegrei volcano through different time.

Conclusions

RAD7 has been used at the INGV of Naples for fumarolic emissions. Preliminary results demonstrate that the methodology utilized in this work enables the analysis of Rn concentrations even in H_2S-bearing gases, discharged from the fumaroles of the Campi Flegrei volcano and, most importantly, without instrumental issues. Fumaroles may be sampled and analysed over time according to the methodology descripted in this work, suitable for volcanic and environmental monitoring purposes. Future work will be focused on testing if lead acetate traps absorb radon, evaluating the possible analytical interference of major gases (i.e., CO_2) on the effective radon content, and finding proper correction factors for raw data.

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Coordinamento editoriale
Francesca DI STEFANO
Istituto Nazionale di Geofisica e Vulcanologia

Progetto grafico
Barbara ANGIONI
Istituto Nazionale di Geofisica e Vulcanologia

Impaginazione
Barbara ANGIONI
Patrizia PANTANI
Massimiliano CASCONE
Istituto Nazionale di Geofisica e Vulcanologia

Istituto Nazionale di Geofisica e Vulcanologia
Via di Vigna Murata, 605
00143 Roma
tel. +39 06518601

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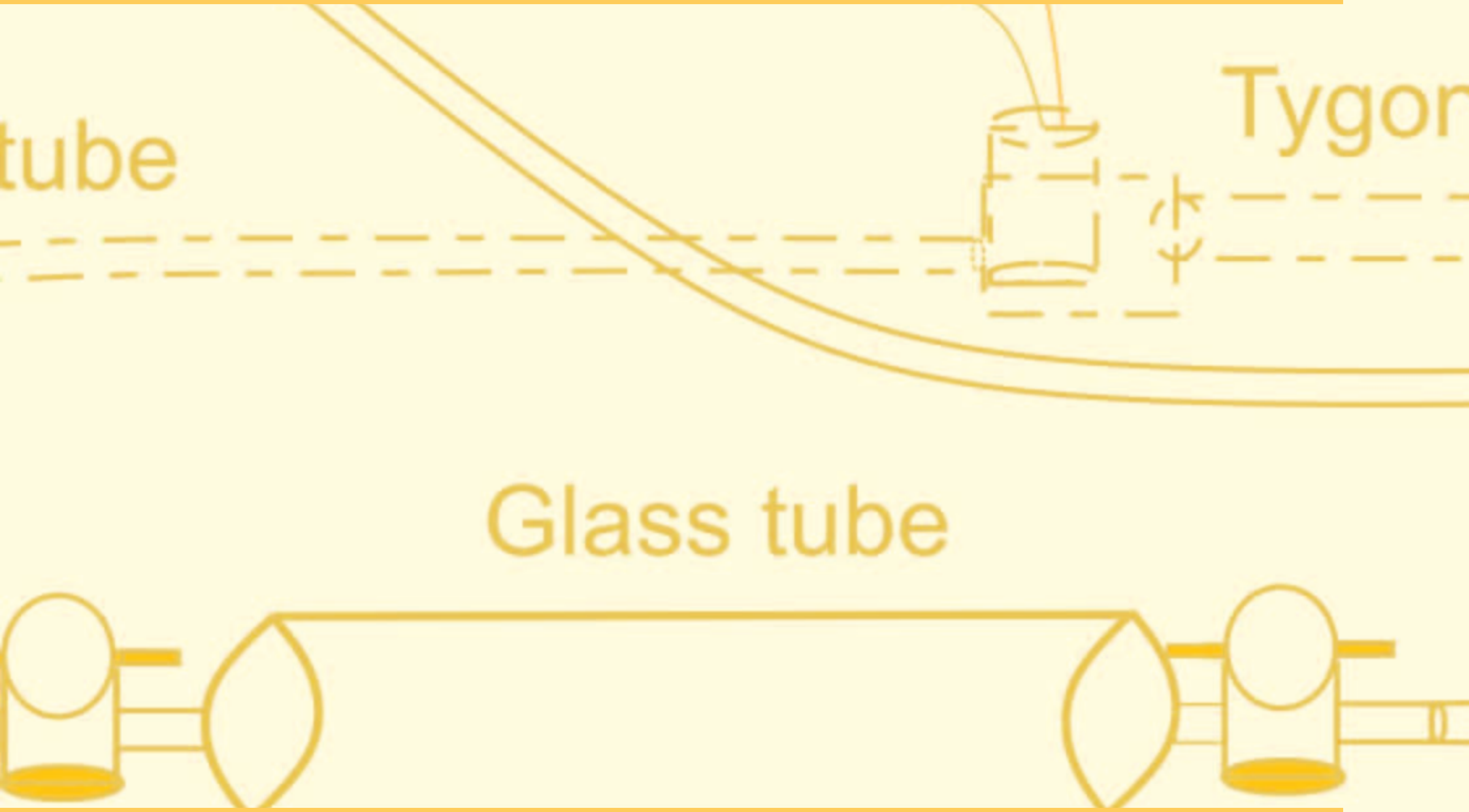

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