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Metrology infrastructure for radon metrology at the environmental level

Annette Röttger[1], Stefan Röttger[1], Daniel Rábago[2], Luis Quindós[2], Katarzyna Woloszczuk[3], Maciej Norenberg[3], Ileana Radulescu[4], Aurelian Luca[4]

[1] *Physikalisch-Technische Bundesanstalt, Bundesallee 100, 38116 Braunschweig, Germany*
[2] *University of Cantabria (UC), Spain,*
[3] *Central Laboratory for Radiological Protection (CLOR), Poland*
[4] *"Horia Hulubei" National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Romania*

ABSTRACT
Since 2020 a large consortium has been engaged in the project EMPIR 19ENV01 traceRadon to develop the missing traceability chains to improve the sensor networks in climate observation and radiation protection.
This paper presents results in the areas of: Novel ^{226}Ra standard sources with continuous controlled ^{222}Rn emanation rate, radon chambers aimed to create a reference radon atmosphere and a reference field for radon flux monitoring. The major challenge lies in the low activity concentrations of radon in outdoor air from 1 Bq·m^{-3} to 100 Bq·m^{-3}, where below 100 Bq·m^{-3} there is currently no metrological traceability at all. Thus, measured values of different instruments operated at different locations cannot be compared with respect to their results. Whin this paper, new infrastructure is presented, capable of filling this gap in traceability. The achieved results make new calibration services, far beyond the state of art, possible.

Section: RESEARCH PAPER

Keywords: radon calibration; radon flux calibration; traceRadon; radiation protection; climate observation

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Corresponding author: Annette Röttger, e-mail: annette.roettger@ptb.de

1. INTRODUCTION

Radon gas is the largest source of public exposure to naturally occurring radioactivity. Radon activity concentration maps, based on atmospheric measurements, as well as radon flux maps can help Member States to comply with the EU Council Directive 2013/59/Euratom and, particularly, with the identification of Radon Priority Areas. Radon can also be used as a tracer to improve Atmospheric Transport Models and to indirectly estimate greenhouse gas (GHG) fluxes. This is important for supporting successfully GHG mitigation strategies. One approach to estimate GHG fluxes on the local to the regional scale is the so-called Radon Tracer Method (RTM), which is based on the night-time correlation between atmospheric concentrations of radon and GHG measured at a given station together with information on the radon flux data within the station's footprint. Thus, atmospheric monitoring networks are interested or are already measuring atmospheric radon activity concentrations using different techniques but a metrological chain to ensure the traceability of all these measurements has been missing till traceRadon [1] started. An overview on the project is available in [2], details on the metrology needs can be found in [3].

This paper presents in section 2 new activity standards which are available to provide new reference atmospheres. The creation of theses atmospheres is a complex task as explained in section 3. To bring all capacities together a reference field for the radon flux from soil to atmosphere is needed. This infrastructure, the so-called exhalation bed facility is presented in section 4. The paper closes with a summary and an outlook.

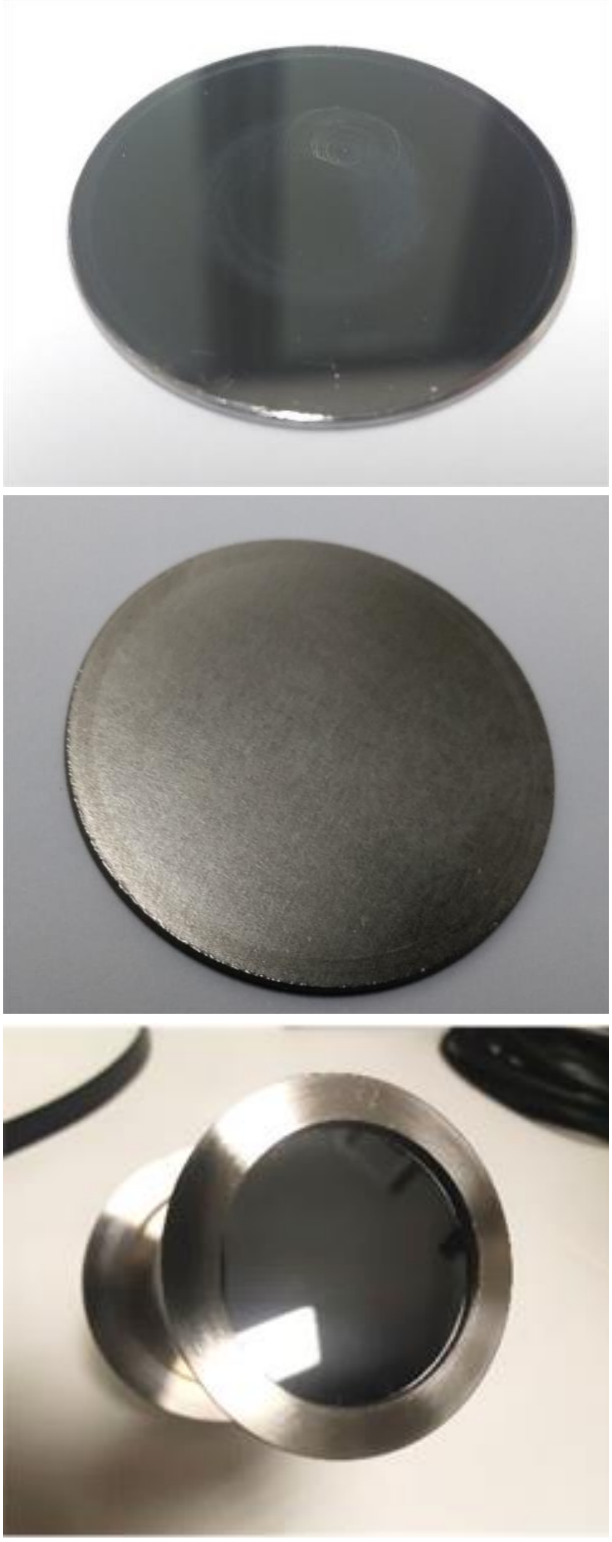

Figure 1. New kind of activity standard sources for ^{222}Rn based on the principle of emanation of ^{222}Rn from electrodeposition, implantation or physical vapor deposition of ^{226}Ra activity from standards, produced at PTB. From top to bottom: (1) Electro-deposited ^{226}Ra activity standard: Deposition at 30 V < U < 200 V (easy to produce, but still dependant of environmental parameters during utilisation); (2) Implanted ^{226}Ra activity standard: Implantation of ^{226}Ra into W / Al after mass separation at the RISIKO mass separator at the university of Mainz with about 30 keV (ideal metrological source, but difficult and expensive to produce); (3) PIPS ^{226}Ra activity standard: 450 mm², 300 µm with about 160 Bq $^{226}RaCl_2$ layer from thermal, physical vapor deposition (new kind of source/detector combination with online emanation traceability).

2. NEW ACTIVITY STANDARDS

The metrology of radon (^{222}Rn is considered here) with respect to the representation of the unit of activity concentration in Bq m^{-3} can be fundamentally divided into two different approaches:

One way to quantify radon absolutely is the method of Picolo [4], in which gaseous radon from the decay of radium (^{226}Ra) is frozen out at a cold point in vacuum. The alpha particles emitted by the solid radon are measured at a defined solid angle so that the unit of activity can be traced back to the base units of seconds and meters. The radon is then returned to the gas phase and can subsequently be used to produce atmospheres of known activity concentration.

Other methods are based on liquid scintillation counting [5] or $4\pi\gamma$ method [6]. The activity concentration of an atmosphere produced in this way decreases exponentially with a half-life of 3.8 days, limiting the counting statistics and hence the achievable uncertainty in the calibration of test samples. Thus, this approach is not suitable for representing reference atmospheres with activity concentrations comparable to those in outdoor air (< 100 $Bq \cdot m^{-3}$).

The use of this activity concentration for calibration purposes requires that the released activity of radon can be quantified traceable to the SI system.

Since the mechanism causing the release of radon consists of a combination of two processes, on the one hand the recoil of the nuclei resulting from alpha decay and on the other hand the diffusion processes, the amount of radon released usually correlates with environmental parameters, such as relative humidity and temperature, since these influence the effective diffusion coefficient. To investigate these dependencies, novel sources have been developed at PTB with the metrological possibility of continuous determination of the emanation [8]. Thus, it can be shown that at low penetration depth of the ion-implanted ^{226}Ra, there is no measurable dependence of the emanation on the ambient humidity and only a small dependence on the temperature [9]. The absolute determination of the activity of ^{226}Ra is done for this type of sources by α-spectrometry under a defined solid angle, while the released fraction of ^{222}Rn is quantified by γ-spectrometric comparison with radon-dense sources of the same design. On this basis, several primary ^{222}Rn activity standards have been fabricated that are suitable for representing the unit activity in the activity concentration range < 100 $Bq \cdot m^{-3}$ in large-volume climate chambers. This is because typical sizes of walk-in climate chambers are 10 m³ to 30 m³.

Figure 1 shows three different activity standards in an overview: At the top a source based on electro-deposition of ^{226}Ra on stainless steel backing, in the middle mass separated ion-implanted ^{226}Ra onto a tungsten backing (tungsten or aluminium are both possible as backing using laser resonance ionization at the RISIKO 30 kV mass separator of Johannes Gutenberg-Universität Mainz) and on the bottom thermal physical vapor deposition of $^{226}RaCl_2$ onto 450 mm² ion-implanted silicon detector.

A completely new approach in traceability and thus a milestone in the metrological development is represented by the new device at the bottom of Figure 1: It accomplishes several challenges, such as high temporal resolutions and at the same time is able to generate high counting statistics. It also needs to meet specific requirements, like to be able to achieve low uncertainties at low activities, for which the highest possible realizable detection efficiency is needed.

The innovative approach was to coat commercially available silicon detectors with a thin layer of ^{226}Ra: Integrated ^{226}Ra Source/Detector (IRSD). After vapor deposition, ^{226}Ra is located on the surface of the silicon detector, so that about 50% of the radon produced is released. Both, the alpha particles of the ^{226}Ra and those of the remaining radon can thus be detected spectrometrically under ambient pressure with a counting yield of about 50%, separately from each other and continuously. Since the total amount of radon is a conservation quantity, the continuously recorded alpha spectra allow the amount of released radon to be determined. This type of measurement represents a mathematical inversion and requires special analytical procedures. Based on the Kalman filter for state estimation in linear dynamic systems, novel algorithms have been designed and implemented, which can be used to calculate the time course of the released activity of radon and the associated uncertainty from the continuously acquired measurement data. Using the described method, even the release of one radon atom per second (about 2 $\mu Bq \cdot s^{-1}$) can still be determined within a few hours integration time with an uncertainty of around 2 % (k = 1) on average. In the future, the IRSD will be used to calibrate radon monitors continuously, automatically and with feedback from radon chambers but even in the field and under changing climatic conditions.

3. NEW REFERENCE ATMOSPHERES FOR CALIBRATION OF RADON MONITORS

The importance of radon exposure to the population and workers was noticed in 2013 by the European Union when the Basic Safety Standards Directive (BSS) was published in the Council Directive 2013/59/Euratom. A significant part of this Directive refers to the issues related to the radon hazard in dwellings and workplaces. The EU member states were required to establish national radon action plans addressing long-term risks from radon exposures in dwellings and workplaces. Hence, member states needed to establish national reference levels for indoor radon concentrations, which should not be higher than 300 $Bq \cdot m^{-3}$. This created new challenges for the radon dosimetry and calibrations because the previous limit used in certain EU member states had been much higher at 1000 $Bq \cdot m^{-3}$. Among others arose the need for harmonization of existing radon measurement procedures and for creating new ones, ensuring the traceable calibration of radon measurement instruments at low radon activity concentrations with low uncertainties. These aspects could have been solved over the years, inter alia, due to research projects, like for instance MetroRADON, www.metroradon.eu.

Radon is not only addressed as the largest natural source of public exposure to ionizing radiation but also as a useful tracer for understanding atmospheric processes and estimating greenhouse gas emissions [10]. That is why reliable measurements of low-level radon activity concentrations, such as those found in the environment (< 20 $Bq \cdot m^{-3}$), are important both for organizations responsible for radiation protection and climate research. Despite the enormous changes in radon metrology that have occurred in recent years, the activity concentrations below 100 $Bq \cdot m^{-3}$ have not been subject to metrological research so far. This poses new challenges, which are the development of traceable methods and robust technology for measurements of environmental low-level radon activity concentrations and radon fluxes. Both are important to derive information from greenhouse gas fluxes in the environment and therefore important for reduction strategy planning.

Properly defined closed volume is necessary to create the reference radon atmosphere. In calibration laboratories two types of radon chambers are used: The first type is a large container (typically the volume of such chambers is greater than 10 m$^{3)}$. Often designed as a walk-in chamber with an air-lock, allowing entry and exit with the minimum disturbance in the radon atmosphere. The second type of radon chambers are small containers only for the equipment under test. The volumes are usually less than 1 m^3. The large radon chambers, due to their large volume, allow to perform intercalibrations of active radon monitors or calibration of devices measuring potential alpha energy concentration defined as:

"*The concentration of short-lived radon-222 or radon-220 progeny in air in terms of the alpha energy emitted during complete decay from radon-222 progeny to lead-210 or from radon-220 progeny to lead-208 of any mixture of short-lived radon-222 or radon-220 in a unit volume of air. The SI unit for potential alpha energy concentration is J m^{-3}*", given in ICRP 115 [11]

The smaller radon chambers are usually used to expose passive detectors in reference radon activity concentration. Radon chambers with a volume of about 1 m3 represent an interesting option for calibration laboratories, allowing a successful approach to the calibration of both active and passive radon monitors.

A Radon chamber is not only a "radon container", but also a system that ensures the maintenance of radon concentration inside and appropriate environmental conditions. The most frequently monitored parameters are temperature, relative humidity, atmospheric pressure, the ambient aerosol concentration, size distribution of radioactive aerosols, the radon decay products concentration and fractionalization, equilibrium factor of radium-radon gamma-ray dose or dose rate. The System for Test Atmospheres with Radon (STAR), also called "Radon Chamber", has four inseparable parts: the equipment for containing the atmosphere, the equipment for producing the atmosphere, the reference atmosphere thus created, and the equipment and methods for monitoring the atmosphere [12]. Devices used to characterise the atmosphere shall be traceable to a primary standard.

Table 1 shows radon chambers presented in this article. They are compared according to their basic features. In the table the chambers are represented by the name of the institute and country which operating them: Central Laboratory for Radiological Protection (CLOR), Poland, "Horia Hulubei" National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Romania and University of Cantabria (UC), Spain. All these chambers fulfil the requirements of a STAR. One essential part of the system is the reference radon monitor, used to give the radon concentration inside. In the three cases exposed, the radon monitors have been calibrated at the Bundesamt für Strahlenschutz (BfS), accredited by the national accreditation body for the Federal Republic of Germany DAkkS, according to the ISO standard 17025, which is traceable to the national standards of the National Metrology Institute (PTB) [13]. To validate the two types of low-level ^{222}Rn emanation sources developed within the traceRadon project, comparison measurements will be performed in low-level radon calibration chambers. Based on a protocol, these sources are going to be measured also at IFIN-HH radon chamber.

The Ionizing Radiation Metrology Laboratory (LMRI, IFIN-HH, Romania) has developed this facility in the framework of

Table 1. Radon chambers features of CLOR, IFIN-HH and UC from left to right. All chambers provide services like calibration of active monitors, and exposure of passive detectors.

		CLOR, Poland		IFIN-HH, Romania	UC, Spain
V (m^3)		12	0,47	1	1
Radon concentration range ($Bq \cdot m^{-3}$)		100 to 50 000	100 to 50 000	100 to 10 000	250 to 11 000
Calibration and Measurement Capability (CMC) (k = 2)		7%		5%	5%
Accreditation		According to ISO 17025		Designation according to ISO 17025	According to ISO 17025
Reference devices		AlphaGUARD		AlphaGUARD DF2000	AlphaGUARD Atmos12
Traceability		To BfS Reference Chamber			
Environmental conditions	Adjustable	Temperature, Humidity, Ambient aerosol concentration	-	-	Temperature
	Monitored	Temperature, Humidity, Pressure, Size distribution of radioactive aerosols	Temperature, Humidity, Pressure	Temperature, Humidity, Pressure	Temperature, Humidity, Pressure

national and European joint research projects [14]. The chamber is cylindrical in shape, with a volume of 1 m^3 and has accessories used to produce radon reference atmospheres They ensure international metrological traceability and equivalence of radon activity measurements, while performing reliable measurements of radon activity concentration. An AlphaGUARD DF2000 (Saphymo GmbH) radon monitor traceable to BfS is used to take measurements of radon activity concentration inside the chamber. Recent testing and improvement of the radon chamber tightness and traceability capacity have been done using the reference instruments and standard radon gas sources, thus obtaining designation by the Romanian Nuclear Authority, National Commission for Nuclear Activities Control (CNCAN) as Calibration Laboratory for instruments measuring radon activity concentration in air, according to the ISO standard 17025. The laboratory is already performing calibrations of active radon measurement instruments belonging to various Romanian users.

Two radon chambers are located in the Central Laboratory for Radiological Protection (CLOR), which is the only laboratory in Poland that has accreditation (in accordance to ISO/IEC 17025) in the field of calibration of radon activity concentration and potential alpha energy concentration monitors. One of them is the walk-in radon chamber with a volume of 12.3 m^3. It is an air-tight climatized room made of 100 mm PUR sandwich elements, covered outside with zinc-coated steel and plastic and inside with stainless steel. The chamber is built with an air-lock, allowing entry and exit with minimum disturbance to environmental conditions or radon atmosphere. The chamber has the option of setting the temperature and relative humidity within a wide range. It has also several input ports that allow the entry of radon and aerosols, the collection of air samples and the connection of instruments outside. This climatic radon calibration chamber fulfills the requirements of the international standard IEC 61577-4.

The second one is a small cylindrical shaped, tight container with a volume of 0.47 m^3. This newly developed radon chamber is planned to be used for creating low-level reference radon atmospheres using new low-activity sources developed within the traceRadon project. So far, to generate the reference radon atmospheres, two certified dry flow-through radon sources by Pylon Electronic Development Co. have been used. Their activities are 137.3 kBq and 502.5 kBq.

The radon chamber of the University of Cantabria (UC) Spain is the only Spanish facility that provides calibration of radon monitors and exposure of passive radon detectors, according to ISO/IEC 17025 accreditation. It is a cubic shaped, stainless steel container with a wall thickness of 3.25 mm and an internal volume of 1 m^3. The access to the chamber is provided by lifting the top lid of the chamber. It also allows access through three circular holes of 80 mm diameter, commonly used to insert and remove passive detectors without disturbing the radon inside during the process. Radon measurement equipment can also be inserted inside at different heights. To homogenize the internal concentration, a fan is built in, working non-stop during the exposures. The radon sources used to generate the reference atmosphere are certified sources model Pylon RN-1025 (Pylon Electronics Inc.) and sources powdered with high ^{226}Ra content and encapsulated in a PVC jar which provides a constant radon diffusion rate. The radon monitors with traceability to international standards are the Atmos12 (Gammadata Instruments AB) and the AlphaGUARD (Saphymo GmbH). These devices are connected to a computer located outside the chamber to know the concentration in real time. The radon concentration inside the chamber can be controlled and modified by using an open-air circuit that includes a pump, extracting air from inside the chamber.

4. REFERENCE FIELDS FOR THE CALIBRATION OF RADON FLUX MONITORS

The exhalation bed facility provided by the Laboratory of Environmental Radioactivity, University of Cantabria (LaRUC), acts as a standard flux source of radon activity across a defined area per time.

Thus, its aim is to be used for calibrating continuous radon flux systems, that can be applied as a transfer standard for test validation of existing radon flux monitors by comparison campaigns in field or on the same exhalation bed under laboratory conditions. The exhalation bed facility consists of two exhalation beds, one with a high radon exhalation rate and another with a low radon exhalation rate (see Figure 2).

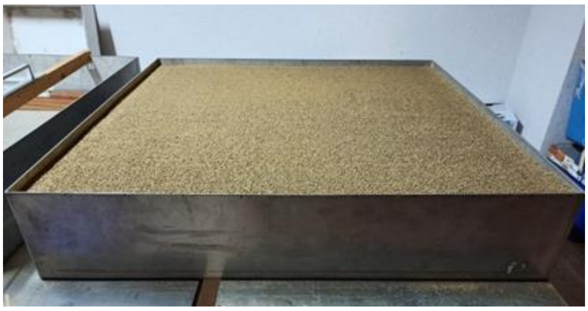

Figure 2. Picture of the Exhalation Beds: Brown, to the bottom: high radon flux, grey, to the top: low radon flux.

Each exhalation bed has an effective surface of 1 m^3 and is made up of five stainless steel welded plates shaping a box, with the upper part open. This configuration avoids the leakages through the plates and forces radon to escape to the top surface. The materials for the exhalation beds were selected according to their radioactive content and properties, such as soil texture, structure, dry bulk density, porosity, etc. The soil for the high exhalation bed was collected from the former Spanish uranium mine, located in Saelices el Chico (Salamanca, Spain) and managed by the Spanish National Uranium Company ENUSA. The low exhalation bed soil, however, was taken from the Fos-Bucraa mine (western Sahara). The materials were dried, sieved and homogenized before putting them into each container.

The radon flux reference value of each exhalation bed has been approached by two ways.

Firstly, the theoretical one that is obtained from the resolution of the diffusion equation [15]:

$$E = \varepsilon \cdot C_{\mathrm{Ra}} \cdot \rho \cdot \lambda \cdot z \,, \tag{1}$$

where ε is the soil emanation factor, C_{Ra} is the radium 226 concentration, ρ is the dry bulk density, z is the soil thickness and λ the radon decay constant. The equation (1) is an approximation when the soil thickness is much smaller than the radon diffusion length. Such assumption was checked from the relationship between the diffusion length L and the porosity p and moisture content m [16].

The second approach is the experimental one, i.e. monitoring the radon increasing in a hermetically closed accumulation chamber [17]. The soil features and the exhalation rate obtained using both approaches are shown in Table 2.

Moreover, during the intercomparison campaigns in the framework of the traceRadon project two different areas were tested that could be a reference site to check radon flux systems under environmental conditions [18]. The high radon flux field is in the grounds of a former uranium mine managed by the Spanish Uranium Company (ENUSA), located in Saelices el Chico (Salamanca, Spain).

The average soil radium concentration in this area is (814 ± 65) $\mathrm{Bq \cdot kg^{-1}}$ (k = 2). The reference radon flux value obtained during the intercomparison campaign was 2364 $\mathrm{Bq \cdot m^{-2}\ h^{-1}}$ with a standard deviation of 1172 $\mathrm{Bq \cdot m^{-2}\ h^{-1}}$. The low radon flux field, located in Esles de Cayón (Cantabria, Spain), contains an average radium concentration of (29 ± 3) $\mathrm{Bq \cdot kg^{-1}}$ (k = 2) in soil. In the low area, the reference value obtained was 50 $\mathrm{Bq \cdot m^{-2} \cdot h^{-1}}$ with a standard deviation of 15 $\mathrm{Bq \cdot m^{-2} \cdot h^{-1}}$. Moreover, it was a good agreement between the consensus experimentally obtained values and the output of the model proposed by [19].

The radon flux systems that can be tested in both scenarios, exhalation bed facility and field sites, should have defined protocols, both for installation and for measurement and analysis. In case of the exhalation bed, it is possible to open and close the accumulation chamber manually, if necessary, however in field tests it is preferable to have an automatic system in order to check the correlation with environmental parameters.

5. SUMMARY AND OUTLOOK

Naturally occurring radon is the cause for most of the population's exposure to ionizing radiation. At the same time, radon is a highly efficient tracer for understanding atmospheric processes, for evaluating the accuracy of chemical transport models, and for providing integrated emission estimates of greenhouse gases. A metrological system for measuring

Table 2. Results of the parameters influencing the calculation of radon exhalation rate in each “Exhalation Bed” for theoretical approach. Uncertainties are expressed with the coverage factor (k = 1).

		Exhalation Bed	
Parameter	**Symbol (unit)**	**High**	**Low**
Moisture content	m	0.013	0.05
Porosity	p	0.36	0.61
Emanation factor	ε	0.18 ± 0.03	0.53 ± 0.02
Radium concentration	C_{Ra} (Bq/kg)	19130 ± 350	214 ± 8
Bulk density	d (kg/m³)	1645 ± 2	893 ± 0.6
Radon decay constant	λ ($\mathrm{h^{-1}}$)	(7.5575 ± 0.0004) ·10^{-3}	
Thickness	z (m)	0.165 ± 0.005	0.130 ± 0.005
Diffusion Length	L (m)	1.29	1.55
Exhalation Rate (theoretical)	E_{theo} ($\mathrm{Bq\ m^{-2}\ h^{-1}}$)	6900 ± 1000	100 ± 6
Exhalation Rate (experimental)	E_{exp} ($\mathrm{Bq\ m^{-2}\ h^{-1}}$)	6320 ± 240	94 ± 4

atmospheric radon activity concentrations as well as the radon flux from the soil is therefore needed for atmospheric, climate, and radiation research.

The activities developed and performed within the traceRadon project went even further to increase the metrological capabilities of low atmospheric radon concentrations calibration and monitoring in the range below 100 Bq m^{-3}. This has included up to now:

- the development of new primary ^{222}Rn activity standards suitable for representing the unit activity in the range below 100 Bq m^{-3} in the established walk-in radon chambers.
- the first successful calibrations in the new reference atmospheres radon monitors using the new developed activity standards.
- the development of laboratory exhalation beds tests for the calibration of radon flux monitors.
- intercomparison campaigns in two types of fields, low and high radon exhalation rates.

The results obtained in the above-mentioned activities feed into the traceRadon project with respect to radionuclide metrology and radiation protection at the environmental level. Knowledge of such joint efforts can offer a solid background to providing more accurate and traceable results for these measurement methods, being even more challenging due to the outdoor environment.

Climate change and radiological protection both affect humankind and the environment worldwide. For the planet to combat both climate change and radiation exposure, measurements must be supported by reliable metrology. By addressing a topic (i.e. the measurement of low levels of radon in the environment) that supports both climate observation and global radiological protection, this project simultaneously supports the long-term economic, social and environmental work of ICOS, the Integrated Pollution Prevention and Control (IPPC) Directive 2008/1/EC, the IAEA, Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) and WHO.

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