

RADON-THORON CALIBRATION ACTIVITIES QA/QC PROGRAMME



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PREFACE

- **The National Radiation Protection Institute (NRPI)** is a non-profit organization established by the decision of the chairman of the State Office for Nuclear Safety (SONS) on May 26, 1995, which became effective on July 1, 1995. The Institute was based mainly on the former Centre for Radiation Hygiene of the National Institute of Public Health in Prague *transferred under the authority of SONS* , and it continues its *tradition of many years*.
- **Institute research** is focused on protection against ionizing radiation, including the provision of infrastructure and research in areas of:
 - Security research
 - Radiation Monitoring Network research
 - Research on exposures to artificial sources of ionizing radiation
 - Research on medical exposures
 - Research on exposures to natural radiation sources.

PREFACE

The NRPI - Department of Natural Exposure – Radon/Thoron Lab.

- organizing inter-comparisons (since 2010)
- participates in R&D Projects
- participate in a special radon diagnostics in a house under the scope of a National radon action plane

MAIN GOAL

- To help relevant institutions and itself in their QA/QC process coming out from accreditation requirements. (issued Reports to participants and publications RPD Vol. 145, No.2-3, 2011, RPD Vol.160 No. 1-3, 2014, RPD Vol.164, No.4, 2015)
- Financial benefit

TARGET QUANTITIES OF INTEREST

- Radon / thoron gas concentration in air including their mix field
- Short- lived radon/thoron decay products in air (EEC)
- Unattached and attached part of EEC
(focused on all the types of measuring instruments i.e. spot, passive integral, and continuous monitors):
- Air exchange rate in buildings (continuously, passive integrals).

THE NRPI - CERTIFICATE OF ACCREDITATION




NÁRODNÍ AKREDITAČNÍ ORGÁN

EA MLA Signatory
Český institut pro akreditaci, o.p.s.
Olšanská 54/3, 130 00 Praha 3

issues

according to section 16 of Act No. 22/1997 Coll., on technical requirements for products, as amended

CERTIFICATE OF ACCREDITATION

No. 135 / 2015

Státní ústav radiační ochrany, v.v.i.
with registered office Bartoškova 1450/28, 140 00 Praha 4, Company Registration No. 86652052

to the Testing Laboratory No. 1479
SÚRO Testing Laboratories

Scope of accreditation:

Measurement of the content of radionuclides in products, raw materials, building and waste materials, food chain samples, water, human body, biological materials and other components of the environment; determination of dosimetric quantities and radon activity for radiation protection purposes to the extent as specified in the appendix to this Certificate.

This Certificate of Accreditation is a proof of Accreditation issued on the basis of assessment of fulfillment of the accreditation criteria in accordance with

ČSN EN ISO/IEC 17025:2005

In its activities performed within the scope and for the period of validity of this Certificate, the Body is entitled to refer to this Certificate, provided that the accreditation is not suspended and the Body meets the specified accreditation requirements in accordance with the relevant regulations applicable to the activity of an accredited Conformity Assessment Body.

This Certificate of Accreditation replaces, to the full extent, Certificate No.: 360/2014 of 9 June 2014, or any administrative acts building upon it.

The Certificate of Accreditation is valid until: **9 June 2019**

Prague: 23 February 2015





Jiří Růžička
Director
Czech Accreditation Institute
Public Service Company



The Appendix is an integral part of
Certificate of Accreditation No. 135/2015 of 23/02/2015

Accredited entity according to ČSN EN ISO/IEC 17025:2005:

Státní ústav radiační ochrany, v.v.i.
SÚRO Testing Laboratories
Bartoškova 1450/28, 140 00 Praha 4

Ordinal number ¹⁾	Test procedure/method name	Test procedure/method identification	Tested object
2.	Determination of attenuation properties of materials by ionometric method in Isovolt Titan x-ray beams	SOP 09 (ČSN EN 61331-1)	Protective materials
3.	Determination of kerma in air and kerma rate in air by ionometric method in Isovolt Titan x-ray beams and OG-8 irradiator beams	SOP 10 (IAEA TRS No. 457 IAEA TRS No. 469)	Ionizing radiation field

5. Dosimetry Department

Tests:

Ordinal number ¹⁾	Test procedure/method name	Test procedure/method identification	Tested object
1.	Determination of personal doses from external exposure using TLD Harshaw 6600 system	M1	External radiation exposure of persons
2.	Determination of ambient dose equivalent and directional dose equivalent using TLD Harshaw 6600 system	M2	Ionizing radiation field

6. Department for Radon and NORM

Tests:

Ordinal number ¹⁾	Test procedure/method name	Test procedure/method identification	Tested object
1. *)	Determination of radon activity concentration time records using continual monitors	M12 (SÚJB Recommendation Methods of measurement and evaluation of natural radionuclides in buildings, building sites, building materials and water)	Indoor air of buildings and structures



THE NRPI METROLOGICAL (QA) CONCEPT

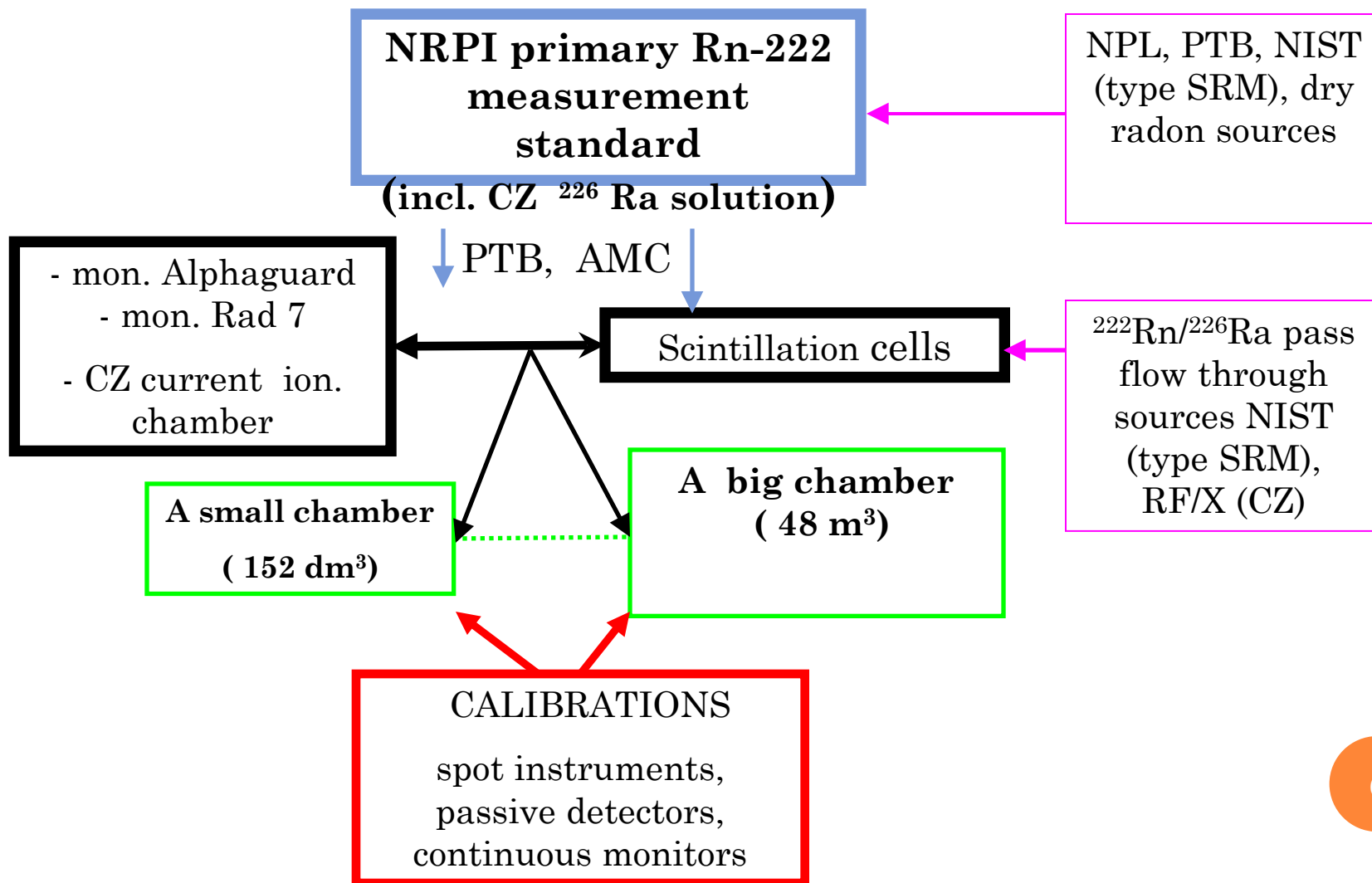
I. Is based on existence :

- A) Relevant measurement standard for each mentioned quantity of interest.
- B) Used comparing instrument for each quantity of interest allowing transfer of relevant quantity from the standard to another instruments and an independent control of self metrology in renowned Labs.
- C) Two crucial tools allowing both routine calibrations and applied research (The NRPI is a scientific institution - not pure metrological):
i.e. a big radon chamber and a small thoron/ radon chamber.

II. Is focused primarily on:

Indoor ambient conditions i.e. p, T, RH, aerosol concentration and size distribution, air exchange rate, surface of walls etc. (available in the big chamber).

Traceability scheme for radon gas measuring instruments



The NRPI radon gas measurement standards



Fig. 1 Based on pulsing ion. chambers.

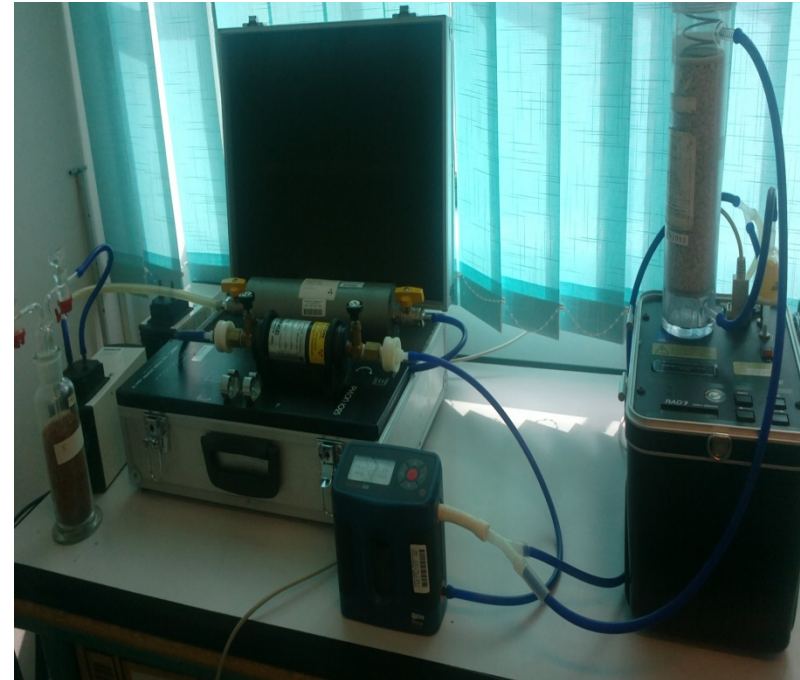


Fig.2 Based on defined $^{222}\text{Rn}/^{226}\text{Ra}$ source production P and air flow rate f . $a_v = P/f$

In the both cases is overall uncertainty of transferred quantity radon concentration to the NRPI reference comparing instruments about 3% ($K=1$).

The NRPI 201 reference current ion. chamber and used radon sources and calibrators.



Fig.3 Ion chamber in current mode



Fig. 4 $^{222}\text{Rn}/^{226}\text{Ra}$ radon sources and calibrators

NOTE: Another used emanation sources $^{222}\text{Rn}/^{226}\text{Ra}$ type Pylon A-2000 and Czech made (CMI Prague) were not illustrated.

The NRPI precise air flow calibrators and relevant stable pumps and samplers.

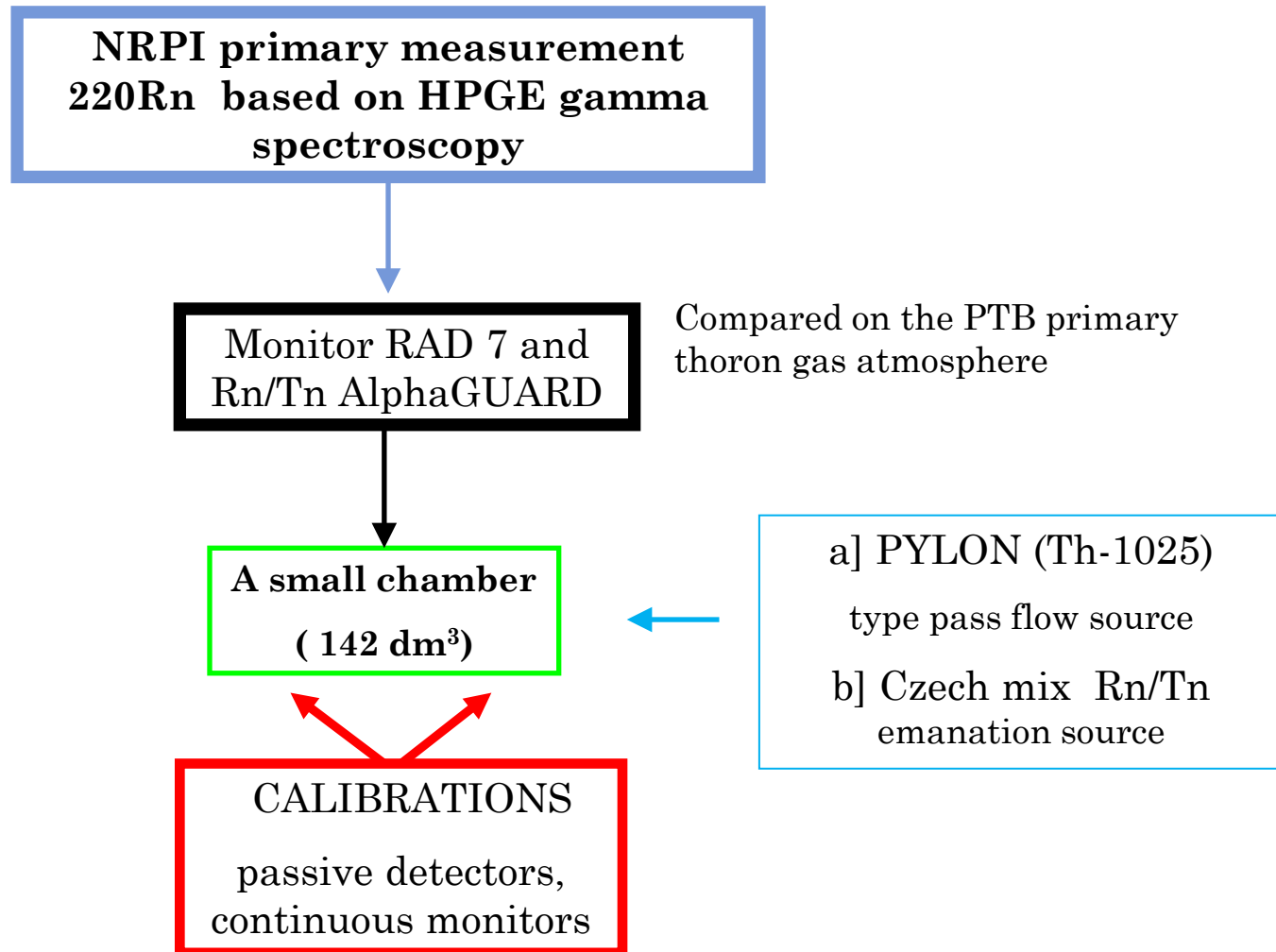


Fig. 5 Overview of used flow meter calibrators and pumps working in a wide rage.

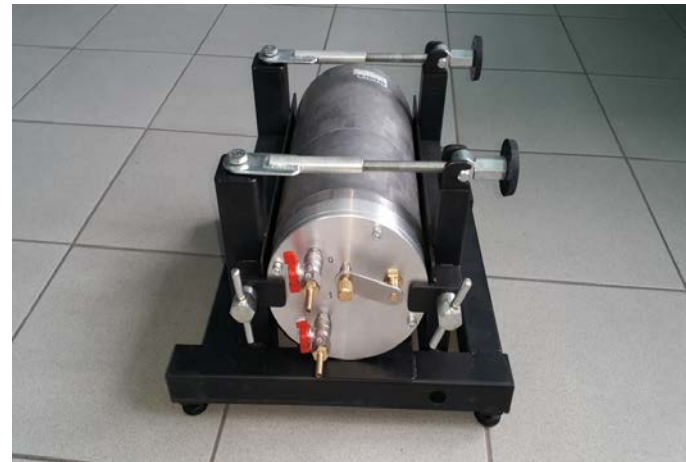
Dynamic range: 5 ml/min - 30 000 ml/min

Overall flow rate measurement uncertainty (1,5 – 2)% for K=1.

Traceability scheme for thoron gas measuring instruments



THE NRPI - THORON GAS MEASUREMENT STANDARD



$$\text{Thoron concentration} = P ({}^{220}\text{Rn})/f = A({}^{228}\text{Th})/f \times E \times K(\text{losses}) \lambda_{\text{Tn}}$$

I.

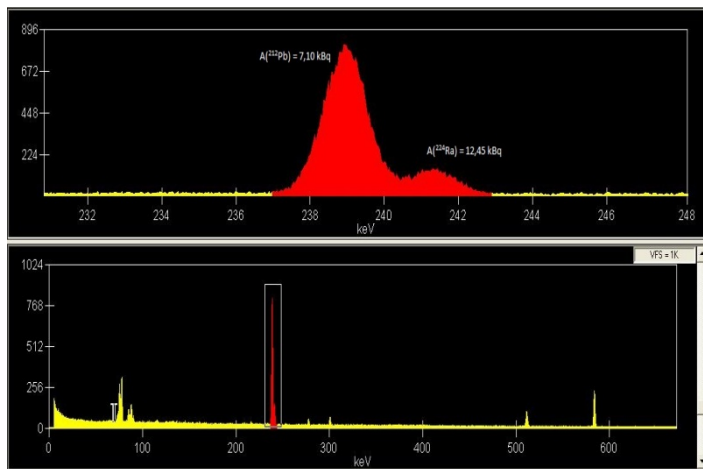
A (228Th) via HPGE by means of ${}^{224}\text{Ra}$
E as ratio of ${}^{212}\text{Pb}/{}^{224}\text{Ra}$

Gammas from ${}^{224}\text{Ra}$ (3.66 d 4)

E_γ (keV) I_γ (%) Decay mode

E [keV] **Nuclid**
238,632 **Pb-212**
240,986 **Ra-224**

240.986 6 4.10 5 α
 292.70 10 0.0062 7 α
 404.2 2 0.0022 5 α
 422.04 10 0.0030 5 α
 645.50 10 0.0054 9 α



 ***** P E A K A N A L Y S I S R E P O R T *****

Detector Name: DET01
 Sample Title: Sample title.
 Peak Analysis Performed on: 18.9.2018 17:23:22
 Peak Analysis From Channel: 114
 Peak Analysis To Channel: 16384

Peak No.	ROI start	ROI end	Peak centroid	Energy (keV)	FWHM (keV)	Net Peak Area	Net Area Uncert.	Continuum Counts
M 1	5777-	5928	5827.11	238.91	1.40	2.80E+004	172.91	1.49E+003
m 2	5777-	5928	5884.69	241.28	1.40	4.65E+003	77.90	1.27E+003
3	6739-	6809	6772.71	277.71	0.99	1.28E+003	55.94	7.63E+002
4	7290-	7363	7326.85	300.44	0.92	1.63E+003	59.68	7.78E+002

Declared total uncertainty for thoron gas < 10% (K=1)

The NRPI thoron gas calibration facility

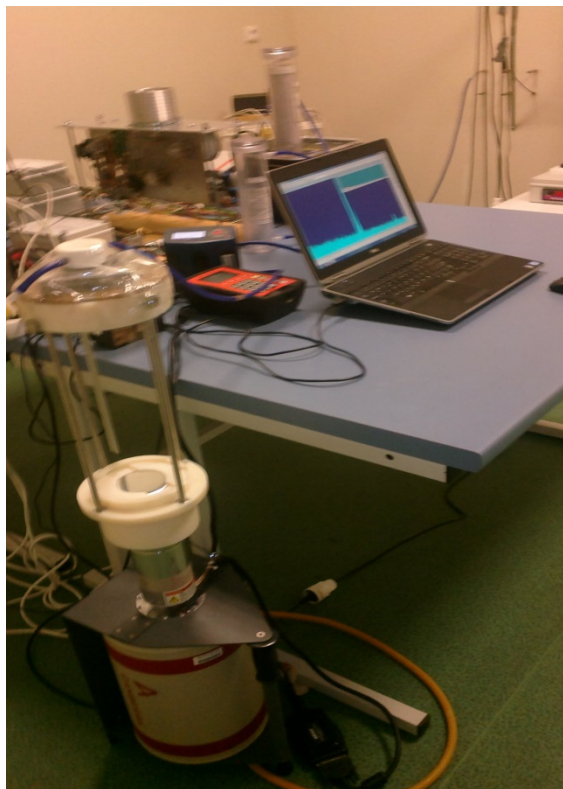


Fig.6 Thoron calibrations via HPGe gamma spectroscopy



Fig. 7 NRPI – NIRS (JPN) comparing instruments

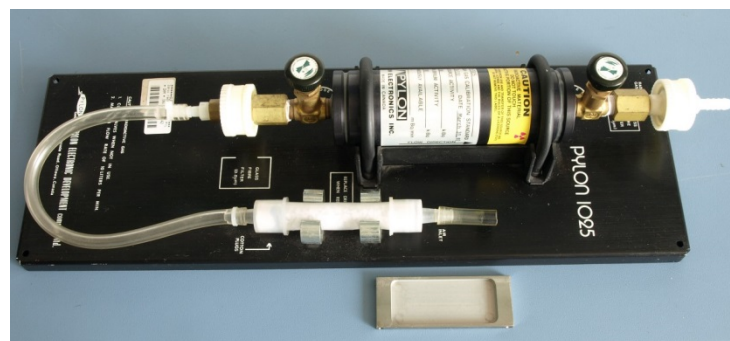


Fig.8 Used Tn sources in routine

The NRPI radon/thoron chamber (150dm³)

Comprises:

- Stainless steel vessel
- Internal 3 fans,
- Internal removable rack
- Bushings – monitoring, VN, sampling and filling
- Internal RH/T sensor
- Thoron homogeneity is assured and tested via Electret ion chambers

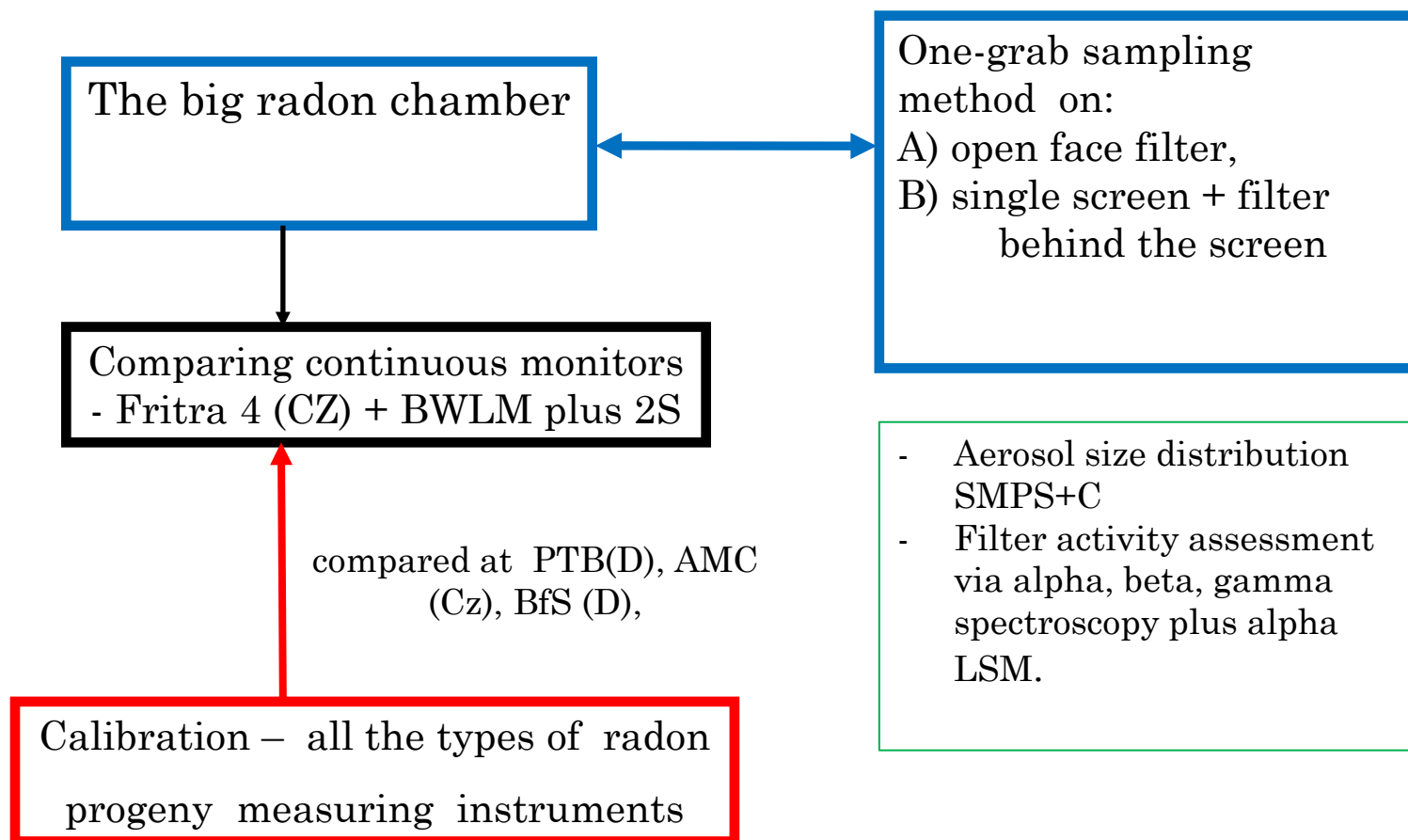


Fig.10 US and CZ Electrets



Fig.9 Thoron/radon chamber

Traceability scheme for short-lived $^{222}\text{Rn}/^{220}\text{Rn}$ decay products measuring instruments including fp



NRPI reference instruments and methods for measurement EEC and its unattached fraction f_p



Fig. 11 Alfa spectroscopy for assessment of filter activity of $^{222}\text{Rn}/^{220}\text{Rn}$ progeny with aerosol spectrometer DMA + CPC Grimm (D)



Fig. 12 Sampling filter and screen
Cut off = 4nm

NRPI big radon chamber

The chamber works in a dynamic regime and allows measurement, defined changes and keep on stable following quantities:

T, RH, air exchange rate (ACH), aerosol size distribution based on caranauba wax and NaCl generation, radon and its short / lived DP/ thoron and its DP.

radon gas , EECr \approx order ($10^1 - 10^4$) Bq/m³

RH - (5 – 95)%, T – indoor dynamic range

T - (10 - 45)°C

F - (2 – 90)%, fp (2 – 90)%

Z – up to $N \times 10^4$ p/cm³ GM = (0,04- 5)μm , GSD = 1,15

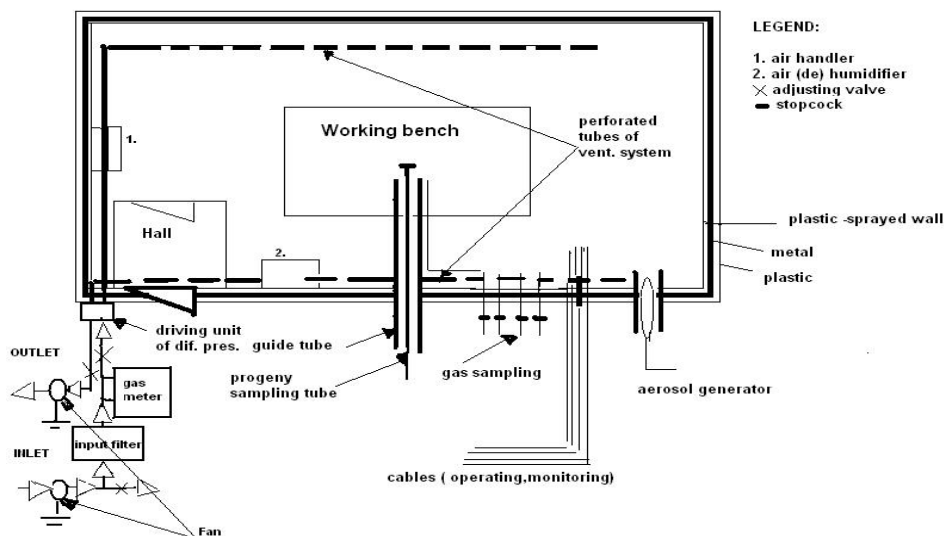
TOPAS (liquid), solid (0,1-0,3) μm, ACH - (0,05 – 2)h-1



Fig.13 The chamber interior

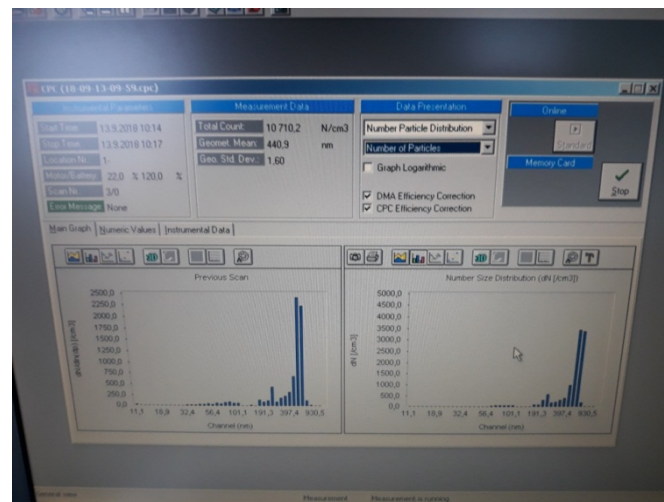
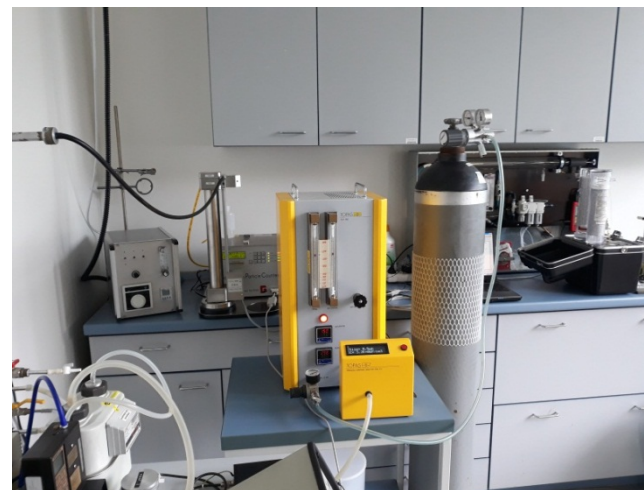


Fig.14 Ventilation system



AEROSOL GENERATORS AND AEROSOL SIZE DISTRIBUTION

DEHS	0.1 ... 5 μ m (8 μ m)
Stearic Acid	0.1 ... 6 μ m (12 μ m)
Carnauba Wax (solid)	0.1 ... 3 μ m (5 μ m)
Other Tested Materials	Emery 3004, Engine Oil 15W40, Paraffines
Geom. Stand. Dev.	<1.15
Number Concentration	10 ⁶ Particle/cm ³
Total Flowrate	200 ... 250l/h
Saturator Temperature	Up to 400°C
Reheater Temperature	Up to 400°C
Carrier Gas	Nitrogen 250l/h@5bar
Power Supply	110...240VAC 50...60Hz
Dimensions	550 × 300 × 250mm
Weight	17kg (19kg)



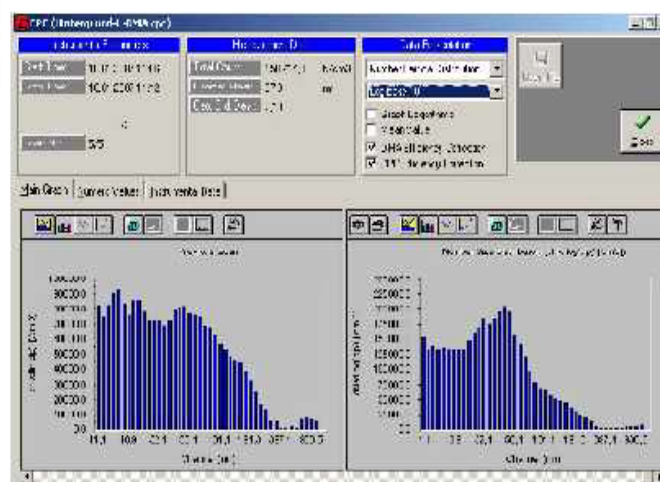
Measured ASD with SMPS + C
(Grimm)



Single Nano Particle Counters Series 5.400

SMPS+C = CPC + DMA + Software

By adding a DMA the user can upgrade the CPC to a high resolution Scanning Mobility Particle Sizer (SMPS+C) that records size distributions between 5 and 1100 nm.



Outdoor aerosol size distribution and indoor activity size distribution

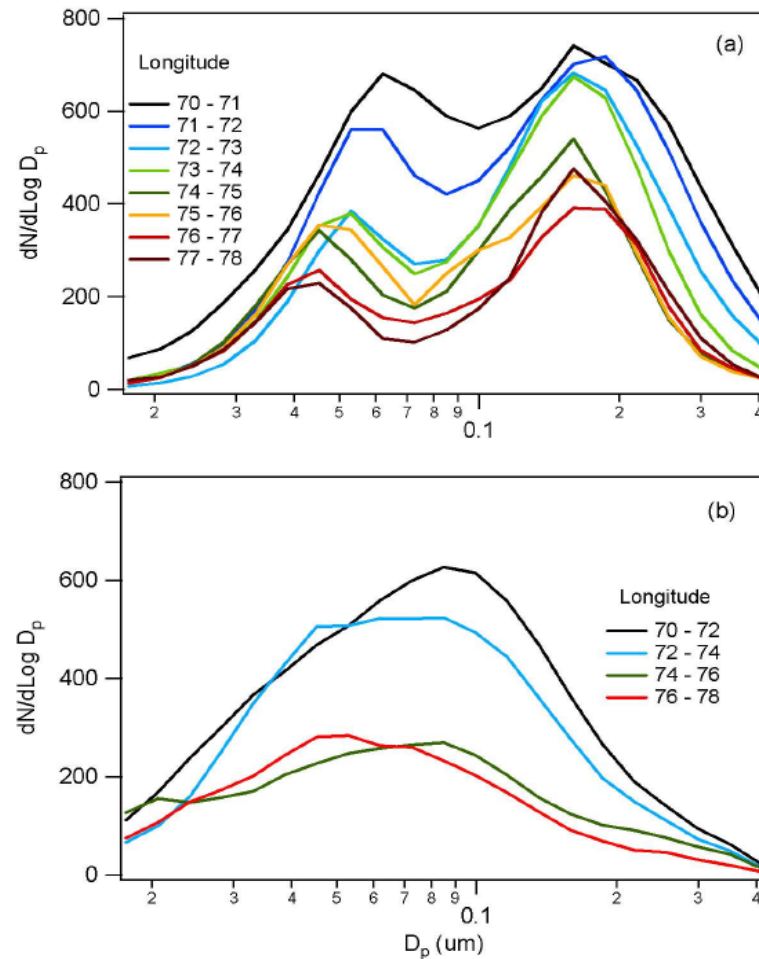
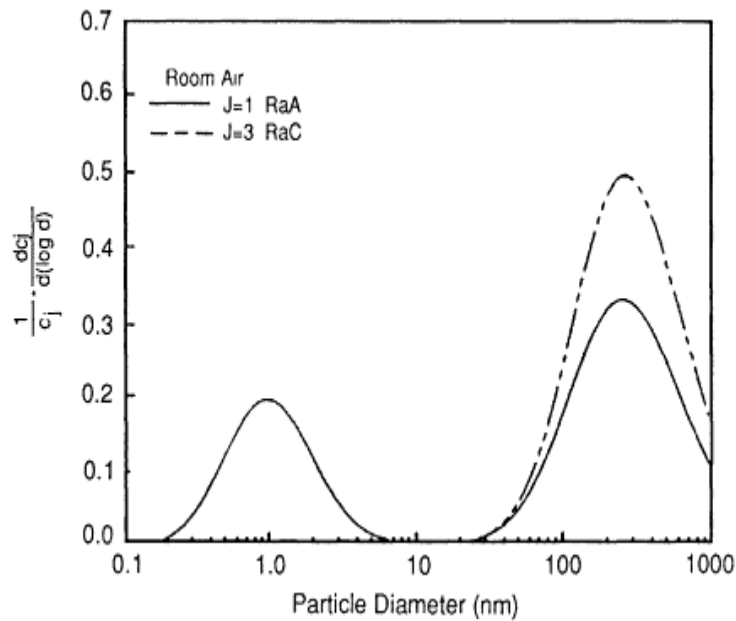


Fig. 8. Aerosol size distribution measured with SMPS (a) in the MBL below 800 m and (b) above cloud layer.

An alternative methods used for assessment of f_p

- Approximations according to Prof. Pörstendorfer (J- P. room model)
via measured ratio of unattached (F)/attached(A) activity of ^{218}Po as follows:

$$X = \lambda_1 (a_1^{iF} / a_1^{iA}) - (\lambda_1 + \text{ACH} + q^A)$$

$$q^F = \lambda_1 \{ [a_0^i / (a_1^{iA} + a_1^{iF})] - 1 \} - (\lambda_1 + \text{ACH} + X)$$

- Calculation of the attachment rate X by means of measured aerosol size distribution $Z(d)$ and known attachment coefficient $\beta(d)$ as follows:

$$X(Z) = \int_0^\infty \beta(d)Z(d)dd$$

- Algebraic inversion of the J- P. room model (Dr. J. Thomas)
- Calculation of ACH via the tracer gas method .

Comparing instrument Fritra 4 for $^{222}\text{Rn}/^{220}\text{Rn}$ progeny including their unattached and attached fractions

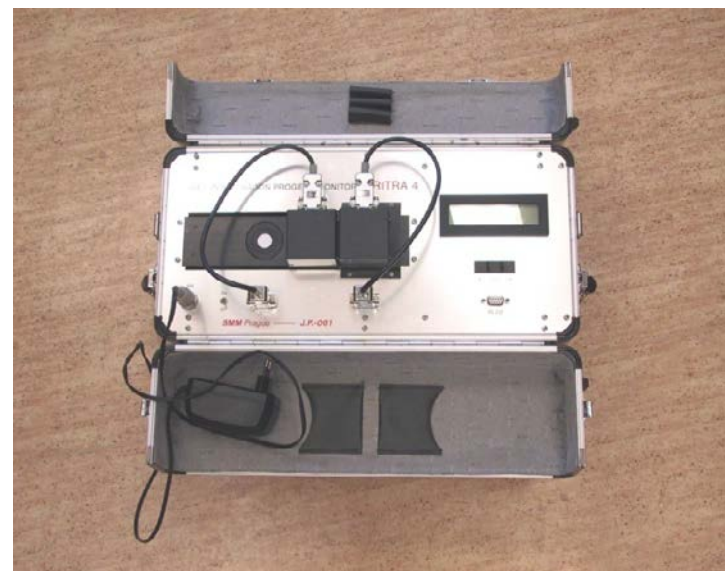
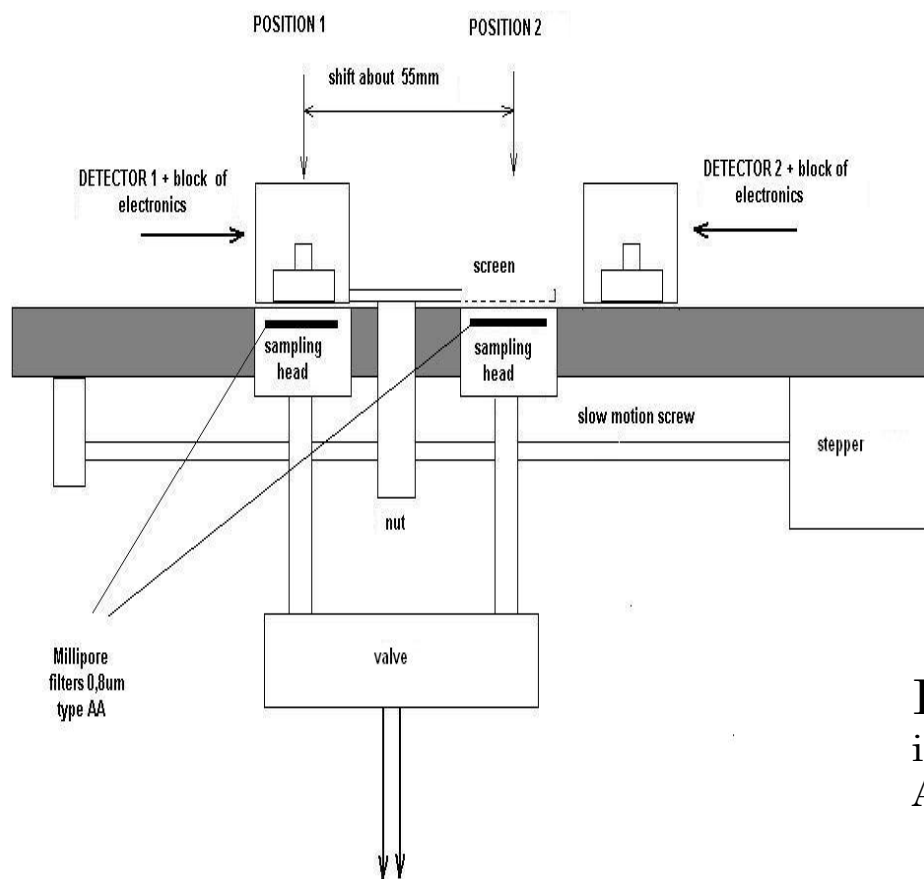
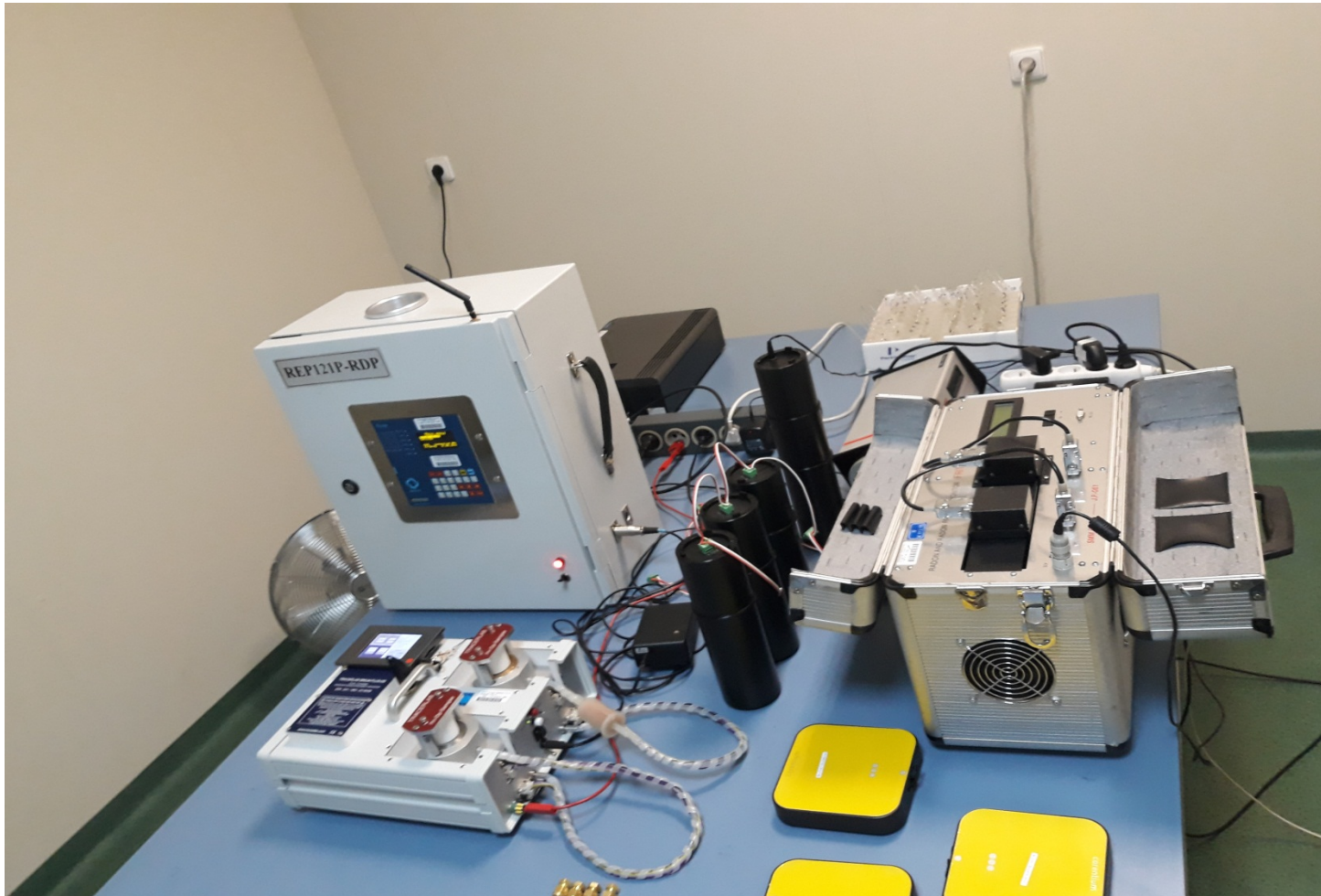


Fig. 15 Monitor Fritra 4 (CZ) tested also in the PTB (D), HPA (UK), BfS (D) and AMC Kamenna (CZ)

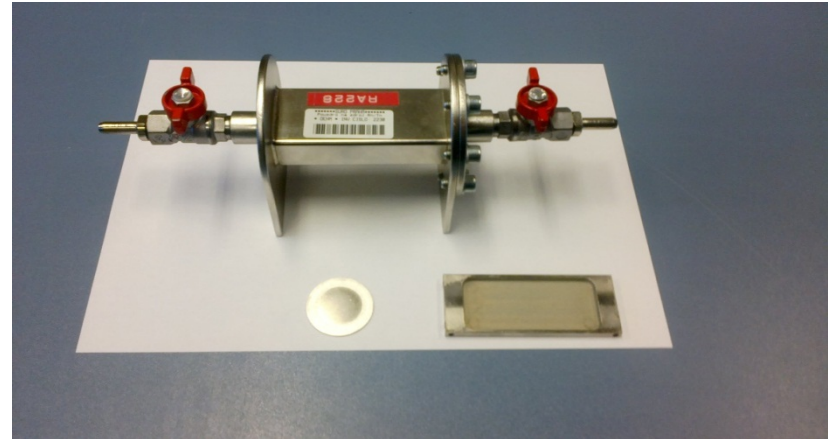
Comparing instrument BWLM plus 2S for $^{222}\text{Rn}/^{220}\text{Rn}$ progeny including their unattached and attached fractions



The NRPI low-level radon gas continuous monitor (LLRM)



LLRM - 100 l ss -cylinder with 20 kV HV and alpha spectroscopy with powerful pump



Low active ^{226}Ra source – 10 Bq, $E > 99\%$ and others

Window	A+C	A+C	A	A
Sampling time	60 min	30 min	60 min	30 min
MDA (Bq/m ³)	0,10	0,15	0,27	0,41
DU (imp/min/ Bq/m ³)	2,05		0,8	

The key LLRM parameters

The NRPI low-level continuous radon gas measurement instruments with MDA-concentration below 5 Bq/ m³

- A. Based on high volume (3l) scintillation cells with response corrected according to Ward approach.
B. Alpha spectrometric high volume (50l) instrument based on alpha PIN diode.

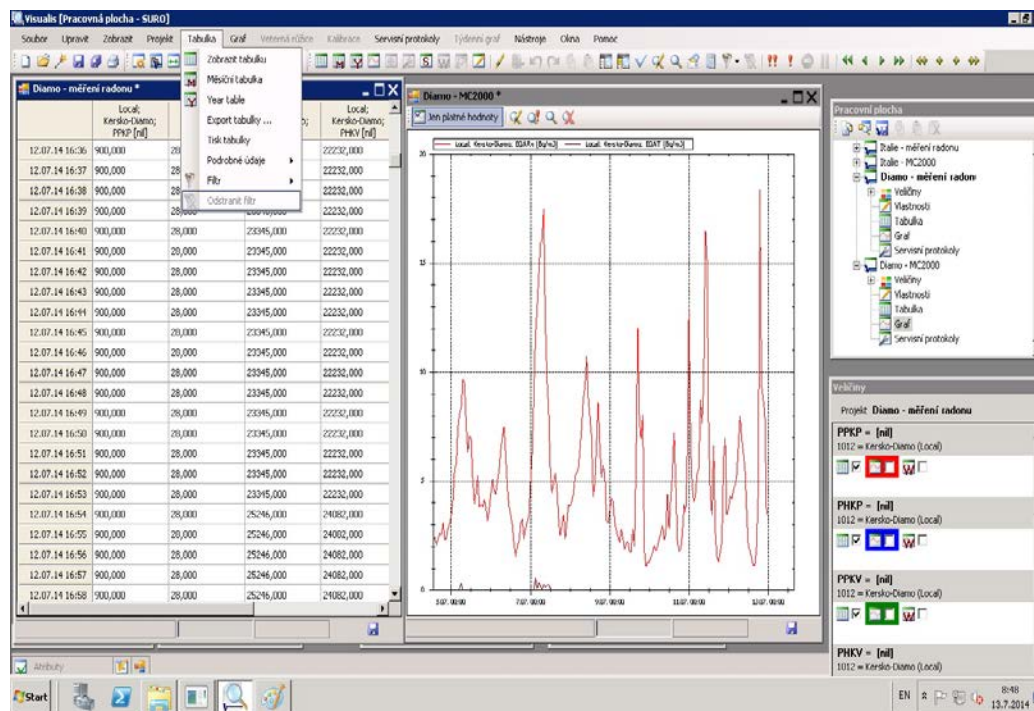


Fig.16 An example of measured radon outdoor daily variations and used radon low -level measurement instruments

The purpose of a (QA/QC) program is :

to ensure proper processes are followed by all individuals responsible for radon measurements so that if errors do arise they will be recognized early on.

The source of the error should be identified, and adjustments shall be made to correct issues to ensure that high quality results are obtained.

Quality Control —the process of measuring the ongoing performance of its devices to ensure continued conformance to established performance standards;

USEFUL DEFINITIONS:

Accuracy - refers to the closeness of a measured value to a standard or known value.

Precision —refers to the closeness of two or more measurement results to each other.

Calibration - correcting a measuring instrument by measuring values whose true values are known

Lowest Level of Detection (LLD) LLD is based on the detector and analysis system's background and can restrict the ability of some measurement systems to measure low concentrations.

Quality Assurance Plan — the planned and systematic activities implemented in a quality system to ensure that quality requirements are met.

Quality Control —the process of measuring the ongoing performance of its devices to ensure continued conformance to established performance standards;

Relative percent differences (RPD) – comparison of two measured values

Relative Percentage Error (RPE) – a measurement of inaccuracy

Reference value (RV) – conventionally true value

Uncertainty - is the total error generated by precision and accuracy errors.

Quality Assurance Plan must include the following elements:

- I. Background information on the organization
- II. Name of Organization's Radon QA/QC Coordinator
- III. Organization's Radon Services Offered
- IV. Identify Radon Devices Used
- V. Identify Employees Using Radon Measurement Devices and Personnel Qualifications
- VI. Work Flow Control and Radon Test Procedures (measurement Protocols)
- VII. Documentation and Record Control
- VIII. Change Control (measures that need to be taken after an error is discovered)
- IX. Calibration and Maintenance Plans
- X. Procedures for Non-Conformances and Corrective Action

Within QC process we should focus on following five procedures:

1. Calibration

Measurement device calibration is to be conducted in accordance with the manufacturer's recommended calibration schedule. The calibration is to be conducted by the manufacturer or an authorized representative of the manufacturer. Calibration ensures that the machine is operating properly and measuring accurately.

2. Spike/Performance tests or inter-comparisons (blind proficiency test)

are to be conducted according to recommended schedule. Blind performance testing or inter-comparisons involve sending the radon devices to the organizer of the inter-comparison, where the radon devices will be placed in an accredited radon chamber and exposed to a known radon concentration.

- To calculate the acceptance range you need to use the calculation to determine the Relative Percentage Error (RPE).

$$\text{RPE} = [(MV - RV) / RV] * 100\%$$

where:

RPE = relative percentage error;

MV = measured value of spiked measurement; and

RV = reference value.

Tests are acceptable if: (they are within +/- 25% at radon levels of 150 Bq/m³ or greater)

3. Field duplicate tests (or Duplicates) are quality control measurements designed to assess the precision of radon measurement devices. Duplicate tests are required for every 10% of tests deployed by an organization. The first detector deployed by the measurement provider should be a duplicate and every 10th test after that should also be a duplicate test.

- Duplicate tests should be conducted by placing two radon devices side-by-side, 10 cm (4”) apart. The test duration for the two devices must be the identical, meaning that the start and stop must be the same.

- Duplicates provide assurance to an organization that the tests are providing acceptable precision. Duplicate measurements should be compared by calculating their relative percent difference (RPD)

- Calculating Relative percent differences: $| \text{Test 1} - \text{Test 2} | / ((\text{Test 1} + \text{Test 2}) / 2) \times 100$
The following chart provides guidance on allowable variances in RPD for duplicate tests.

Average Test Measurement	Acceptable RPD	Warning Level
<75 Bq/m ³	No limits	No limits
75 – 149 Bq/m ³	Less than +/- 25%	+/- 25 to 50%
Over 150 Bq/m ³	Less than +/- 14%	+/- 14 to 28%

Duplicate measurements should be recorded and tracked on a spreadsheet by the organization and analyzed for changes or trends via control charts.

If Duplicate measurements fall outside the Acceptable RPD than action should be taken to determine why the error occurred and how many past tests may have been affected

4. Cross-checks are similar to duplicate tests however the device used needs to be **approved** device. In a situation where a professional is using a continuous radon monitor a cross check should be done every six months to identify any anomalies with the testing equipment in between calibrations.

If a standard check source is available for the device, it can be used for the cross-check purpose. i.e (such as an in-built one)

A cross-check should be conducted for at least a 48 h period and can be conducted using a second device setup up side by side (within 10 cm or 20 cm apart).

The devices should be with in allowable limits as specified in the previous section 3.
Duplicates.

5. Blank tests are designed to measure the limit of detection of the radon measurement device by assessing any background exposure which may increase the lowest limit of detection from the manufacturers stated levels.

- The detection limit is defined to be the lowest concentration of radon that can be measured with a certain confidence. The detection limit is calculated by the manufacturer of measurement devices but then it could be elevated by background exposure which interferes with the actual radon test.
- Blank tests are required for every 5% of tests deployed by an organization. The second test deployed by the organization should be a blank and every 20th test after that should also be a blank test.
- The lowest level of detection must be supplied to the measurement provider by the lab as this is a value calculated on each batch.
- There are multiple types of blank tests depending on the device type.
- **Lab Blanks** – these are blanks that are sent away by the lab after manufacturing to assess any background exposure that a device has while waiting to be deployed by a measurement professional. A lab must retain 5 % of its devices to expose at least one device each time a batch is sent to a purchaser.
- **Transit Blanks** – these are blanks that are sent back to the lab by the measurement professional to ensure that there was no contamination to the shipment of radon tests during transit from the lab to the measurement professional.
- **Field Blanks** – these are blanks that are sent to the lab as blind tests to verify that the devices are not being exposed to background exposure which would interfere with regular radon tests.

Field Blanks should be conducted by following the same procedure as used for duplicates – however the blank detector or device will not be opened or activated until the end of the test, where it would be opened and sent to the lab to be treated the same as one of the exposed devices when analyzed by the lab.

Blank tests are acceptable if they are within +/-10 % the devices lowest level of detection as determined by the manufacturer.

NOTE 1

The results of continuous monitors from the 2016 NRPI Rn/Tn intercomparison (taken from the Report for each participant)

TABLE 3 Analysis of all reported results for Exposure D

INSTITUTE ID	MEAN (Bq/m3)	1 SD (Bq/m3)	SEM (Bq/m3)	R	95% CI	ζ zeta score
1/D	8485	830	97	1,022	0,99-1,05	1,6
2/D	8200	510	61	0,988	0,97-1,01	-1,2
3/D	8839	1388	168	1,064	1,02-1,11	3,0
4/D	8727	453	53	1,051	1,03-1,07	5,5
5/D	8507	806	134	1,025	0,99-1,06	1,4
6/D	8208	476	56	0,989	0,97-1,01	-1,2
7/D	7607	372	44	0,916	0,90-0,93	-9,7
8/D	9172	515	60	1,105	1,08-1,13	10,5
9/D	7938	417	49	0,956	0,94-0,97	-4,9

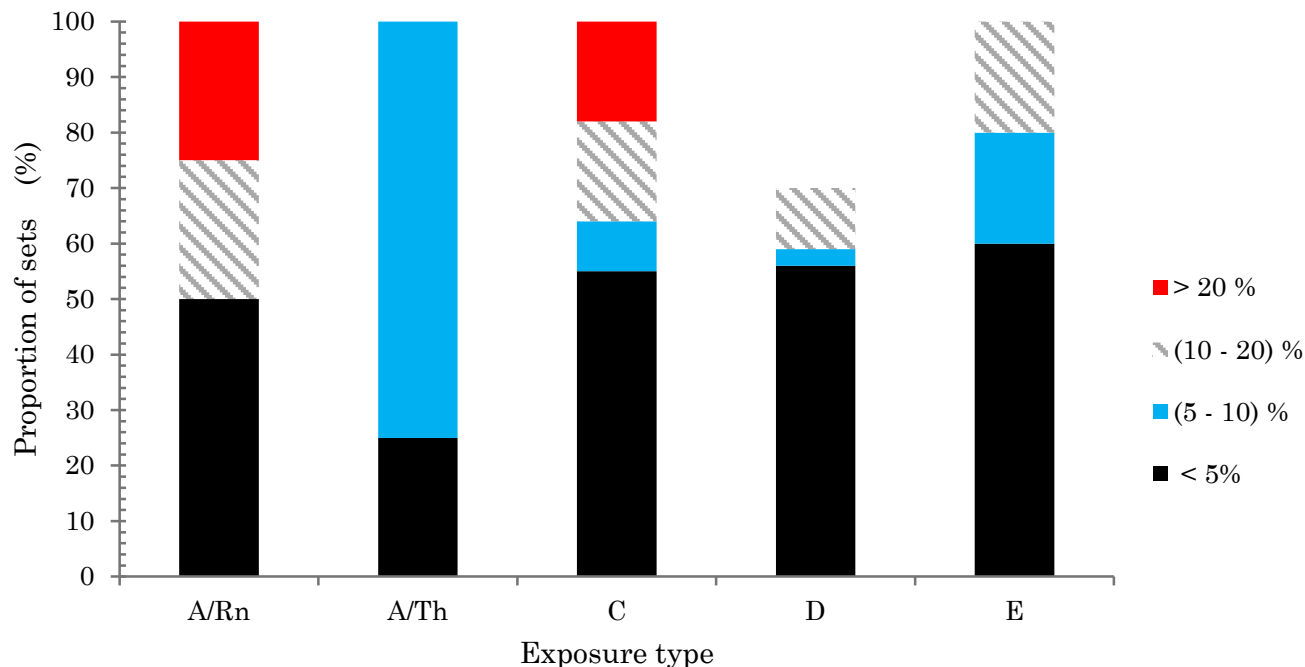
MEAN - total mean within whole exposure measured on an hourly basis

RED labelled – monitors AlphaGUARDS

R – ratio of the total instrument mean to the reference NRPI value

ζ zeta score – related to the NRPI reference value.

The complete results of the 2016 NRPI Rn/Tn intercomparison



E - EEC continuously	5 independent continuous monitors	
A - Rn/Tn tracks	4 independent sets of a passive detectors	
D - Rn continuously	9 independent monitors	
C - Rn passive detectors	11 independent sets of a passive detectors	

NOTE 2

LEGALLY CONTROLLED MEASURING INSTRUMENTS

Legally controlled measuring instrument have to conforms to prescribed legal metrological requirements.

We distiguish between a two basic metrological acts with respect to measurement instruments:

- a) calibration (provided by a private Accredited Labs
- b) verification (provided by an State Authorized Labs)

The goal:

To prevent customers against consequences of incorrect measurements made in the field of official transactions, working conditions, public health and work safety a Decree of Ministry of Industry and Trade sets list of instruments for mandatory verification.

The Decree is attached to the Czech version — [see on Czech version.](#)

CONCLUSIONS

- ★ The NRPI facility allows calibration for all the types of measurement instruments i.e. spot, passive detectors and for continuous monitors in a mix field of $^{222}\text{Rn}/^{220}\text{Rn}$.
- ★ The NRPI calibration way based on measurements standards eliminate a potential problems with „a reference“ instruments as the AlphGUARD, RAD 7 etc.
- ★ The NRPI radon gas metrology is based on the Czech ^{226}Ra standards produced by the Czech Metrological Institute and thoron metrology on PTB ^{228}Th .
- ★ Attention is also payed to radon/ thoron decay products in their mix fields QA/QC system for radon /thoron gas and radon its short- lived decay products including fp measurement instruments developed at the NRPI and cross-checked both within the intercomparisons and solo in the renowned Labs. as the PTB (D), BfS (D), AMC Kamenna (CZ) declares overall calibration uncertainties as follows :
 - less than 5% (K=1) for radon gas
 - less than 10% (K=1) for thoron gas
 - less than 10% (K=1) for short- lived decay products
 - less than 20% (K=1) for f_p .