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Technical report on the REM 2018 radon-in-water proficiency test

Viktor Jobbágy, Heiko Stroh, Gerd Marissens, Petya Malo, Mikael Hult, Raf Van Ammel, Valeria Gruber, Dietmar Roth

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Contents

Fo	reword	1
Ac	knowledgements	2
ΑŁ	ostract	3
1	Introduction	4
	1.1 Responsibilities and roles	5
	1.2 Collaborating partners	5
	1.3 Participating organisations, participation fee	5
	1.4 Timeline and announcements	6
	1.5 PT materials and logistics	6
	1.6 Packaging and sample preparation for shipment	8
	1.7 Reporting of the results	. 10
	1.8 Questionnaire	. 10
	1.9 Data treatment	. 11
2	Material characterisation	. 12
	2.1 Reference values	. 13
	2.2 Homogeneity study	. 15
	2.3 Stability study	. 18
	2.4 Establishing reference values	. 20
3	Reported results	. 22
	3.1 Uncertainties	. 24
4	Scores, evaluation and comparison of results	. 26
	4.1 Standard deviation for proficiency assessment (σ_{PT})	. 26
	4.2 Percentage difference (%)	. 26
	4.3 Z-scores	. 28
	4.4 Zeta (ζ)-scores	. 30
	4.5 E _n -numbers	. 32
	4.6 PomPlot	. 34
	4.7 Comparison of proficiency tests	. 34
5	Information on the participating laboratories: organisational and technical details \dots	. 38
	5.1 Methods used by the participating laboratories	. 39
	5.2 Participants` feedbacks	.41
	5.3 Sources of interferences	.43
	5.4 Reported impacts of this PT	.43
6	Summary and conclusions	. 44
	6.1 Conclusions about performances	. 44
	6.2 Conclusions for PT organisers	.45

6.3 Future JRC actions	45
References	46
Related recommended literature	48
List of abbreviations and definitions	49
List of figures	50
List of tables	51
Annexes (see next page)!	52
Annex 1. Nomination request, e-mail, invitation letter	53
Annex 2. Registration instructions	58
Annex 3. Reporting instructions	60
Annex 4. Accompanying letter	62
Annex 5. Sample receipt form	64
Annex 6. List of participating laboratories	65
Annex 7. Questionnaire	79
Annex 8. Feedbacks and comments from the questionnaire	84
Annex 9. Communication on preliminary results	87
Annex 10. Summary table on participants` scores	88
Annex 11. Calculation of performance evaluation scores	95
Annex 12. The PomPlot interpretation	96
Annex 13. Reference date and times of individual bottles	97
Annex 14. Temperature logger and a typical temperature profile recorded during sample transport	იი

Foreword

This report focuses on the technical evaluation of the REM (Radioactivity Environmental Monitoring) 2018 radon-in-water proficiency test. It contains details on the material characterisation, performance evaluation, information on the participants` organisation, the applied analytical methods. Feedback from participants is also included.

Another JRC report (JRC116812) contains the performance evaluation of the participants and different analytical methods in the REM 2018 PT giving an overview on the exercise and the key scores of the participants.

The REM 2018 proficiency test was performed within the institutional work programme of the JRC Directorate G (Nuclear Safety and Security) as described in the work Package SELMER (Support to European Member States Measuring Environmental Radioactivity in and the Project SARA (Science Applications of Radionuclides and Actinide materials). It is conducted on request of DG ENER to support their work in implementing Article 35 and 36 of the Euratom Treaty.

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 The following organisations for technical cooperation:
 - SCK•CEN (Belgian Nuclear Research Centre in Mol, Belgium)
 - Silesian Centre for Environmental Radioactivity at GIG (Katowice-Poland)

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- Members of the advisory group (Head of Unit ad interim: Arjan Plompen, 17043 Quality management: Petya Malo, Project Leader: Mikael Hult, Statistical advisor: Stefaan Pommé, Jan Paepen, External advisor: Piotr Robouch from JRC-Geel Directorate F.5 Food & Feed Compliance Unit),
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Viktor Jobbágy, Heiko Stroh, Gerd Marissens, Petya Malo, Mikael Hult, Raf Van Ammel, Valeria Gruber, Dietmar Roth

Abstract

A large scale Europe-wide proficiency test (REM 2018 PT) on the determination of the massic activity of ²²²Rn in drinking water was organised by JRC-Geel. The 101 participating environmental radioactivity monitoring laboratories were either nominated by their corresponding national authorities or invited by JRC to participate.

Spring water with an elevated ²²²Rn massic activity was used as the proficiency test material. The water was collected from a natural water source in Austria. Since the material for the PT was collected at a location more than 900 km away from the organiser's laboratories, it was challenging to execute the packaging and transport in a timely manner with short storage and delivery time. We conducted a PT at a smaller scale (Jobbágy et al., 2019b) to test, optimise and streamline the complete process before running the Europe wide PT. The acquired experience helped us to perform in a robust manner the large scale Europe-wide PT with an increased number of participants.

The process involved using state-of-the-art knowledge for the sampling and transport of water containing ²²²Rn (e.g. submersion sampling, temperature controlled transport, radon tight transport containers, etc.). A rapid homogeneity screening was performed immediately after sampling, showing a stable massic activity of ²²²Rn in the samples covering the whole sampling period.

For the reference value determination, homogeneity and short term stability study, two independent measurement methods were used: gamma-ray spectrometry and liquid scintillation counting. The assigned reference value was established by taking the power-moderated mean of the gamma-ray spectrometry measurement results. The uncertainty of the reference values includes the uncertainty related to stability, between-bottle homogeneity, characterisation measurements and sampling.

The performance of each participating laboratory was evaluated with respect to the reference value using relative deviations, z-score, zeta-score, E_n -number. Additionally PomPlots were made up to visualise the data. The results were evaluated grouped by the analytical methods used, to check for method dependency. It was found that the majority of the participants' results, 113 out of 135 (84%), were within the acceptance range, which was set to \pm 20% of the reference value. This suggests that each of the applied methods (emanometry, gamma-ray spectrometry and liquid scintillation counting) seems to be adequate for radon-in-water measurements. However, when the reported value with its uncertainty was evaluated (zeta score), fewer acceptable scores were found, 103 results out of 135 (76%). A key problem with ^{222}Rn is that almost all errors and problems that can occur lead to an underestimation of the massic activity as the inert ^{222}Rn gas can escape at any stage, from sampling until measurement.

1 Introduction

This report describes a large scale Europe-wide proficiency test (referred to as "REM 2018 PT") on radon-in-water organised by the European Commission's Joint Research Centre in Geel, Belgium (JRC-Geel). This report focuses on the technical details of the PT preparation, data evaluation and analysis. Furthermore, the questionnaire is evaluated and the participants` feedback is presented.

The REM 2018 radon-in-water PT was organised on request of the EU member states` Euratom article 35/36 experts with the approval of the European Commission's Directorate-General for Energy (DG ENER). This European scale PT underpins the new EURATOM Drinking Water Directive (EURATOM, 2013)¹.

The G.2 unit of JRC-Geel organises on request of DG ENER regularly proficiency tests (PTs) involving laboratories that monitor radioactivity in the environment. These support the implementation of the Euratom Treaty Article 35 (and 39). The aim is to check comparability of measurement results and verification of data submitted to the European Commission (EC) by European Union (EU) Member States (following Article 36). These PTs are usually linked to regulation dealing with radioactivity in environmental matrices, food or feed. One of the fundamental EU directives in this field is the Euratom-Drinking Water Directive or E-DWD (EURATOM, 2013), which covers several naturally occurring radionuclides including ²²²Rn (radon). Other radon isotopes, like ²²⁰Rn and ²¹⁹Rn are due to their short half-life and limited impact on human health excluded from the directive and consequently also from this exercise.

Radon-in-water analysis is one of the most frequently used radiological monitoring methods. The used analytical techniques are relatively simple and provide reliable results (Jobbágy, 2017a). Very few international PTs (Neznal et al., 2014; Björklöf et al., 2015; Björklöf et al., 2017, Celaya González et al., 2018) have been organised in Europe since the publication of the E-DWD. The main reason is that there are a lot of practical problems in setting up such PTs (Jobbágy el al., 2018; Jobbágy el al., 2019a). The difficulties are e.g. (i) the short-half-life of ²²²Rn (3.8 days), (ii) the fact that radon gas easily escapes from the sample and (iii) the problems to produce a large number of homogeneous samples. The PTs organised in the past had also difficulties related to the lack of metrological traceability, a missing or incomplete homogeneity and/or stability study of the material. This PT provides homogeneous high-quality interference-free material, a metrological traceable reference value and a transport chain free of radon-loss from sampling till arrival at the analytical laboratory.

The REM 2018 PT followed the ISO 35 Guide (2017), ISO 17043 (2010) and ISO 13528 (2015) standards on characterisation of reference materials, organising proficiency tests and performance assessments, respectively.

-

¹ Council Directive 2013/51/EURATOM of 22 October 2013 Laying Down Requirements for the Protection of the Health of the General Public with Regard to Radioactive Substances in Water Intended for Human Consumption.

1.1 Responsibilities and roles

The REM 2018 radon-in-water PT was organised by the European Commission, Joint Research Centre (JRC-Geel), Retieseweg 111, B-2440 Geel, Belgium.

The communication between the organiser and the participants was mainly done using the functional mail account:

JRC-GEE-REM-COMPARISONS@ec.europa.eu.

The responsibilities amongst the involved staff of the organiser:

- Viktor Jobbágy: PT coordinator, sampling, logistics, liquid scintillation counting analysis, reporting.
- Mikael Hult: team leader and quality control.
- Petya Malo: logistics assistant, administration, quality management.
- Heiko Stroh: sampling, logistics, gamma-ray spectrometry analysis.
- Gerd Marissens: sampling, logistics.
- Katarzyna Sobiech-Matura: quality assurance and logistics.
- Raf Van Ammel: internal review of the report
- Jan Paepen: data validation of participants' performance.
- Advisory group members (Arjan Plompen: Head of Unit ad interim, Petya Malo: 17043 Quality management; Mikael Hult: Team Leader; Jan Paepen, Stefaan Pommé: Statistical advisors; Piotr Robouch: External advisor).

1.2 Collaborating partners

JRC-Geel collaborated with external expert institutes in the field. The main contact person and the name of each collaborating institute are listed below:

- Valeria Gruber: Austrian Agency for Health and Food Safety (AGES) in Linz, Austria.
- Michel Bruggeman: SCK•CEN (Belgian Nuclear Research Centre in Mol, Belgium)
- Stanisław Chałupnik: Silesian Centre for Environmental Radioactivity (Katowice-Poland)

AGES in Austria actively contributed to the REM 2018 PT by enabling access to the natural water source, collecting the PT material, performing preliminary material characterisation, carrying out some logistical tasks and giving technical support throughout the PT. The measurement results from SCK•CEN and the Silesian Centre for Environmental Radioactivity were used to confirm the JRC-Geel measurement results.

1.3 Participating organisations, participation fee

The participation in the PT was open to all analytical laboratories. Priority was given to the environmental radioactivity monitoring laboratories nominated by the EU member states` Euratom article 35/36 contact points and authorities. In total 101 laboratories from all over Europe participated in the PT (from 26 EU countries and six EU candidate countries). In addition to the registered organisations, JRC-Geel received additional participation requests by e-mail.

Unfortunately, these requests were rejected because they were received after the registration deadline, shortly before the sampling.

Eight participating laboratories were from outside the European Union (but all H2020 associated countries), while 93 were from within the European Union. The full list of all laboratories with their affiliations is presented in Annex 6.

Participation in this PT was free of charge. All costs regarding the PT organisation were covered by the PT coordinator JRC-Geel, except the sample analysis related costs.

1.4 Timeline and announcements

Table 1 shows the REM 2018 PT exercise tentative time line.

Table 1. Timeline of the radon-in-water REM 2018 PT exercise.

13 July 2018	JRC-Geel contacted national authorities, laboratories requesting nominations and expression of interest
3 September 2018	Invitation letter sent to the nominated/interested laboratories
14 September 2018	Registration deadline
18 October 2018	PT material shipment to participants
14 November 2018	Laboratories` results and questionnaire submission deadline
14 December 2018	Preliminary results sent to participants
30 April 2019	Final report

Source: JRC

The announcements and communication documents are presented in Annex 1-5 and Annex 9.

1.5 PT materials and logistics

It was difficult to get suitable waters for the PT in the area close to JRC-Geel (Jobbágy, 2017b). Austrian natural spring water was used as PT material. The material was named "JRC-W1". It was selected on the basis of the successful use in the JRC pilot-proficiency test (Jobbágy et al., 2019b) organised in the first half of 2018. The PT material had an elevated ²²²Rn massic activity, a low ²²⁶Ra massic activity and complied with our basic requirements towards suitable radon-in-water PT material. When natural waters are used in a PT, not only the water needs to contain relatively high ²²²Rn massic activity but homogeneity within /and in between the individual PT samples and stability have to be assured. Participants should be able to measure the samples before the ²²²Rn decays to low-levels of activity. The half-life of ²²²Rn is only 3.8232 (8) days (Bé et al., 2008). The PT sample's ²²⁶Ra massic activity was below the minimum detectable massic activity (<1 Bq/kg) of the gamma-ray spectrometry system used. There was no interference linked to any presence of dissolved ²²⁶Ra.

For sampling, an immersion - overflow technique was used where a hose was connected with an adapter to the outlet of the spring water as shown in Figure 1. On the other side of the adapter, a plastic tube was connected leading the water into a 15 L bucket. This bucket had a continuous overflow of water. A sampling bottle was immersed into the fully filled bucket. The plastic tube was inserted close to the bottom of the sampling bottle. The bottle was flushed with three bottle-volumes of water before closing it with its cap still underwater. The sampling time of each bottle was registered. After filling, the bottle was wiped dry carefully using towels and paper tissues. There was approximately 1 L of water in each bottle. This volume was expected to be sufficient for the requested analyses. More sampling procedures can be found in details in the corresponding ISO standards (ISO 5667-1, 2006; ISO 5667-3, 2012; ISO 13164-1, 2013). Each participant received one sample bottle. The PT coordinator gave the opportunity to the labs to send their own sampling bottles for sampling. It was not allowed to send liquid scintillation vials with cocktails, because JRC-Geel experienced some difficulties with shipping and receiving LSC cocktails. In the pilot-PT, it was also observed that some of the LSC vials were leaking during the transport to JRC-Geel. Furthermore, according to European regulations (EC No. 1272/2008), the cocktails are classified as hazardous materials. The decision to abandon the shipping of LSC vials including cocktails was also taken from environmental protection point of view to avoid accidental contamination of the natural water sources.



Figure 1. Sampling arrangement at the Austrian sampling site.

Source: JRC

The water temperature at the source was (9.5 ± 0.5) °C and the flow rate of the water source fluctuated between 6-8 L/min. It was not possible to regulate the flow rate but with a special sampling adapter designed by AGES was used to reduce bubble formation. During the sampling period of 4 hours, 175 sample bottles were collected covering 1400-1900 L of spring water volume.

The samples were distributed by a logistics company. In general, the samples arrived at the participants laboratories within 1-7 days after dispatch. In some cases there were some delays due to e.g. customs procedure outside the EU area

or internal reasons (the sample was received by the organisation logistics/storage service but the laboratory personnel only received the sample a few days later).

The samples assigned to different studies were selected using a random stratified selection strategy covering the whole batch. The selection was made using the Sample Number Assignment Program (SNAP) developed and validated at JRC-Geel.

The 160 individual PT samples (Figure 2) were used in the following way:

- 107 samples were sent to the participants
- 10 samples were used in the homogeneity study
- 10 samples were used for the reference value determination
- 5 samples were used in the stability study
- 28 samples served as back-up.

Additionally, 11 samples were sent to participants in sampling bottles provided by them.



Figure 2. PT test samples for homogeneity and reference measurements.

Source: JRC

1.6 Packaging and sample preparation for shipment

Special precautions were taken to assure that the PT material arrived at the participants in good condition. Therefore, robust physical and thermal resistant EXAM packaging transport boxes were used (model: HIGH-Q Pack 20L). They are insulated containers moulded in technical polyurethane foam accommodated in water-resistant cardboard. The boxes can keep their content below 10°C up to 5 days using pre-chilled cooling elements. To confirm that the PT samples were not exposed to high temperature, thermo-buttons were placed into each transport box next to the samples. These thermo-buttons logged the

temperature every 15 minutes during shipment. They were returned after sample arrival to the PT coordinator for reading them out. A typical temperature profile during sample transport is presented in Annex 14.

The glass sampling bottles containing the water samples were put into a protective bubble foil layer or bubble foil envelop. The transport arrangement in a transport box is illustrated in Figure 3.

Figure 3. Interior view of six transport boxes whilst being prepared for shipment.

Source: JRC

Each package shipped to participants contained the following items:

- PT material (1 L glass bottle) wrapped in bubble foil and sealed in a plastic bag,
- an accompanying letter and sample receipt form,
- cooling elements (12-18 units),
- a thermo-button (electronic temperature logger) in a plastic bag.

Upon arrival of the package, the participants were requested to send back immediately the *Sample receipt form* (Annex 5) by e-mail and the thermo-button in an envelope to the PT coordinator.

Participants were instructed to store their samples in a dark place at maximum room temperature (preferably below) but well above 0°C. Before the analysis, the PT coordinator recommended to store the sample bottle at room temperature until it reached thermal equilibrium with its environment.

All samples arrived at the participants without any major problems. Only few participants commented the presence of a small volume (approximately 1 mL) of water in the protective plastic bag. This could be condensed humidity on the cooled sample and water from the thread of the cap since it was immersed into the sampling bucket.

1.7 Reporting of the results

The reporting of laboratory results was done via the JRC online reporting tool. Participants were requested to fill in the online questionnaire about their organisation and technical details of the analytical method(s) used. The link was sent via e-mail to the participants.

Participants were asked to submit their results via the following weblink using the personalised password key provided to each participant:

https://web.jrc.ec.europa.eu/ilcReportingWeb

Participants had the opportunity to report results obtained by different analytical methods (LSC, γ -ray spectrometry, emanometry or other methods) following the organiser`s instructions:

- One measurement result/mean value per method (²²²Rn massic activity in Bq/kg),
- Associated expanded uncertainty with coverage factor of k = 1,
- The applied analytical method.

The reference date for JRC-W1 was 18 October 2018 (Thursday). The exact reference date and times were communicated after shipping the samples. They were given as Coordinated Universal Time (UTC) (Annex 13). In addition, each participant received the information on the exact sampling time of their sample (hour and minute).

We recommended to use the decay data provided by the Decay Data Evaluation Project (DDEP) available at:

http://www.lnhb.fr/nuclear-data/nuclear-data-table/

The ²²²Rn half-life is 3.8232 (8) days (Bé et al., 2008).

It was not possible to accept modifications of results after the reporting deadline.

1.8 Questionnaire

Participants were asked to fill in a questionnaire (Annex 7) which was composed of four main parts concerning the information on the laboratory, experience, technical details on measurement methods, feedback. Information provided in the questionnaire was used to evaluate the results of the proficiency test in detail. The questionnaire was available on the EU-Survey website via the following link:

https://ec.europa.eu/eusurvey/runner/REM 2018 PT radon-in-water

1.9 Data treatment

All results were treated confidentially; identities were kept anonymous. However, the results and performance of <u>each nominated laboratory</u> is made available to the laboratory, its national representative(s) (the nominating authority) and to the relevant services of the European Commission at Directorate General for Energy as announced in the invitation e-mail (Annex 1).

In order to comply with the European regulation on the General Data Protection Regulation (GDPR), we asked for the participants` consent/approval to be able to list the organisation and the name of the contact person in the final report. Participants could express their consent by sending us an e-mail with the following statement:

"Hereby, I [give / DO NOT give] (delete as necessary) my consent to have my name and the name of my organisation listed in the final report of the REM 2018 Radon-in-Water PT organised by JRC-Geel."

However, we decided to include the name of the organisations only to avoid any privacy related complaints.

2 Material characterisation

The determination of ²²²Rn in water samples for the reference value assignment, homogeneity and stability studies was done at JRC-Geel by using standard analytical methods (ISO 13164-2:2013: Water quality - Radon-222 - Part 2: Test method using gamma-ray spectrometry; ISO 13164-4:2015: Water quality Radon-222 - Part 4. Test method using two-phase liquid scintillation counting) as introduced in the next paragraphs.

High purity germanium (HPGe) detectors were used for gamma-ray spectrometry (GS). The unopened sample bottles were placed on a specifically designed sample holder. This holder was directly placed on the endcap of the HPGedetectors. The space in the lead shield was continuously flushed with nitrogen that boils off from the liquid nitrogen Dewar used for cooling the detector. The measurements were performed for one to up to several half-lives to check the ²²²Rn decay and possible ²²⁶Ra presence. The Full Energy Peak (FEP) efficiency curve was established by measuring volume sources of a stable gel provided by Eurostandard (Czech Republic), a branch of the Czech Metrology Institute (ČMI). The sources contain a range of standardised radionuclides commonly used for calibration. The efficiency transfer (from calibration container to glass bottle) and coincidence summing corrections were calculated with the Monte Carlo simulation software EGSnrc (Electron Gamma Shower National Research Council, Canada). For the determination of the ²²²Rn massic activity the following gamma-ray energies of the decay products were used (assuming secular equilibrium between ²²²Rn and its decay products): ²¹⁴Pb (295 keV, 352 keV) and ²¹⁴Bi (609 keV, 1120 keV, 1238 keV, 1764 keV).

For liquid scintillation counting (LSC), approximately 10 g of samples were mixed with UltimaGold-F water non-miscible cocktail in 20 mL low-diffusion plastic vials. A 30 mL syringe connected to a plastic tube of 5 mm diameter and a length of 60 mm was found to be the optimal solution. This tube is used only for taking the test portion, and then replaced with a needle when ejected under the liquid scintillation cocktail surface in the vial as demonstrated in **Figure 4**.



Figure 4. Subsampling for LSC measurements

Two Quantulus 1220 (PerkinElmer) counters were used for the analysis. The typical counting time was set at 120 minutes. The measurement was started more than 3 hours after shaking the sample to allow establishing an equilibrium between ²²²Rn and its short lived decay products. Efficiency calibration, which includes extraction and counting efficiency, was established by measuring two ²²⁶Ra standard solutions in equilibrium with ²²²Rn. The combined extraction and counting efficiencies for ²²²Rn and its decay products was found to be between 430-470%. The high energy beta protocol without alpha-beta discrimination was used. The counts in the channels between 50 and 1024 were summed.

One of the most important performance indicators of a method is its detection limit (for definition, see ISO 11929:2010). For establishing detection limits, blank samples were prepared and compared in two different LSC vial materials, low-diffusion antistatic polyethylene and low-potassium glass. Background samples were prepared by boiling 200 mL deionized water for 10 minutes while stirring with a magnetic stirrer to facilitate removing residual radon. Both vials were closed with a screw cap with an aluminium liner to achieve radon tightness (Jobbágy et al., 2019a). The measurements were performed in the same conditions as the samples and the results are presented in **Table 2**.

Table 2. Comparison of background counts and MDA in LSC using glass and low diffusion polyethylene vial. High energy beta protocol without alpha-beta discrimination was set, channels between 50 and 1024 were considered (120 min data acquisition).

Vial material	# of replicates	Mean counts	MDA* (Bq/kg)
Low diffusion polyethylene	4	277	0.3
Low potassium glass	3	616	0.5

^(*) Minimum Detectable Activity is derived from the Detection Limit introduced by Currie (1968) and ISO 11929 (2010).

Source: JRC

For LSC measurements, the background can be reduced by choosing alpha/beta discrimination and using low background vial material. However, for drinking water analysis one can already achieve a reasonable low background (i.e. detection limit) without alpha/beta discrimination.

2.1 Reference values

For the reference measurements, the selected samples were measured on two HPGe detectors at two distances on each detector. In order to place the bottles in exactly the same positions relative to the detector, custom made sample holders were used. A certified multi-gamma source from CMI was used for establishing an efficiency curve. The detection efficiency of the sample bottle was determined by geometry transfer (including EGSnrc simulations). The data was corrected for background and decay. The ²²²Rn activity was determined by first calculating an activity value for the individual measurement. This was done by taking the weighted mean of the activity obtained from each of the gamma-lines used for analysis. Then, a weighted mean was calculated from the four different measurements (i.e. samples measured on two detectors at two distances). The massic activity was determined by dividing the activity by the sample-mass. The bottle average massic activities from GS measurements are presented in **Figure 5**.

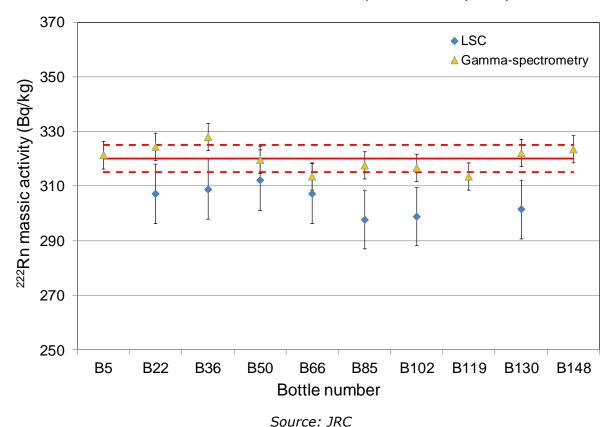
The highest contributions to the combined uncertainty come from counting statistics and the efficiency calibration. Other parameters have only low contributions.

The LSC measurements were performed by gravimetrically preparing three replicates with a sample size of approximately 10 g from the same bottles that were used for GS measurements. Due to repetition of GS measurements for three reference samples it was not possible to measure them with LSC. The measured data for the activity determination were corrected for background and decay. The mean results from the three replicates are calculated and are presented in Figure 5.

The liquid scintillation counting measurement results performed on the same 7 bottles at JRC-Geel (n=7) as measured by GS. It has to be noted that since GS measurements took longer than planned, we were not able to determine radon in three of the reference samples due to decay.

The reference values were established by calculating the power moderated means of the HPGe gamma-ray spectrometry only because the two datasets (LSC results and GS results) were identified as different after using the one-way ANOVA test (a=0.05).

Figure 5. Massic activity results of 222 Rn in the JRC-W1 PT sample. Yellow triangles GS results, blue diamonds LSC results. All uncertainties are combined uncertainties at 1σ level (k = 1). The solid red line: the power moderated mean of the GS results; the dashed red lines: combined uncertainty of the mean (k = 1).



The individual data were checked for outliers but none were found using the single Grubbs's test at p=0.05 level. The uncertainty budgets at the 1σ level

(k = 1) according to GUM (2008) for a typical single measurement by gammaray spectrometry and LSC for are given in **Table 3**.

Table 3. Typical uncertainty budgets for GS and LSC radon-in-water measurements for a single measurement of a sample at the 1σ level as relative values (%). The combined uncertainty is the square root of the quadratic sum of all components (k = 1).

Component	GS (%)	LSC (%)
Counting statistics (incl. background)	1.2	0.7
Weighing	0.05	0.05
Geometry repeatability	0.9	n.a.
Dead time	0.1	0.05
Detection Efficiency	2.2	3.0*
Gamma-ray emission prob.	0.2	n.a.
Half-life	0.2	0.2
Combined uncertainty (%)	2.5	3.1

^(*) combination of counting and extraction efficiency

Source: JRC

2.2 Homogeneity study

A rough homogeneity screening on-site done immediately after sampling to make sure that there are no big differences between sampling bottles within the whole batch. A detailed homogeneity study was done in the laboratory of JRC-Geel using LSC.

At the sampling site 27 samples were measured to verify whether the between-bottle homogeneity within the individual bottles meets our requirements (< 10%). Measurements were performed by measuring water samples in their original glass sampling bottles (approximately 1 L). Sampling containers were placed on the same sample holder positioned directly on the endcap of a germanium detector. The samples (n=27) were measured for 15-20 minutes each until < 5% counting statistical uncertainty was obtained. Data acquisition was done using the "MCA – Measurement System v1.0". The spectra were analysed using GammaVision-32 software. Only decay and mass corrected net peak counts-rates were determined and compared between the samples. A model was created on the basis of the sampling bottle geometries and its material composition. The decay corrected variation of the total count-rates from Pb-214 (295 keV and 352 keV) and Bi-214 (609 keV) were studied.

The on-site screening results from the gamma-ray spectrometry measurements of ^{214}Pb and ^{214}Bi are presented in **Figure 6**. The uncertainties are combined standard uncertainties (with k=1). The major uncertainty contributions come from the counting statistics and the geometry repeatability.

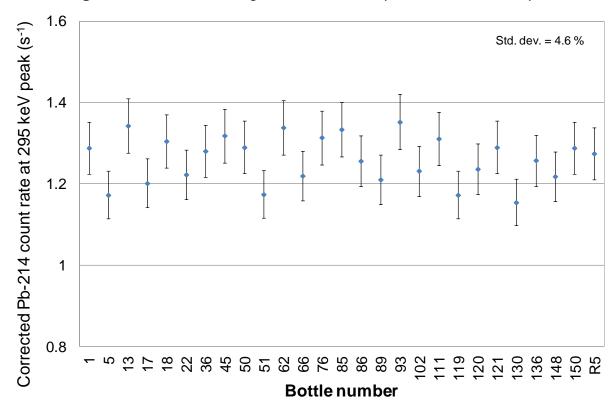


Figure 6. On-site screening results obtained by GS for JRC-W1 sample.

A homogeneity study was performed and evaluated according to ISO 13528 (2015). Ten samples for homogeneity measurements were selected using randomly stratified sampling scheme covering the whole batch (Linsinger et al., 2007).

The detailed homogeneity study was used for the calculation of the uncertainty on the reference value. The study was performed by liquid scintillation measurements using a priori randomly assigned bottles (n=10). Three samples per bottle were prepared for liquid scintillation counting. The sample preparation was identical to the one used for the reference value determination (ISO 13164-4). The replicates were measured with Quantulus detectors in random order as recommended in the ISO 35 guide (2017).

The reference value of a comparison material is assumed to be valid for the whole batch at the level of subsample with a minimum mass or volume (approximately 10 g). Therefore, between-bottle homogeneity in the radionuclide concentration increases the uncertainty of the corresponding reference value.

The results from the laboratory homogeneity study from LSC measurements in the PT material on 222 Rn are presented in **Figure 7**. The uncertainties are combined standard uncertainties (with k=1).

350 222Rn massic activity (Bq/kg) 325 300 275 250 **B**1 **B18 B45 B51** B62 **B89 B93** B111 B121 B136 Bottle number

Figure 7. Homogeneity study results obtained by LSC for JRC-W1 sample.

The between-bottle homogeneity of 222 Rn in the matrix was evaluated using the SoftCRM software-version 2.0.10 (Linsinger et al., 2001) following the certification principles for reference materials as given in ISO/IEC Guide 35 (2017). All individual results were normally and unimodally distributed. No measurement results could be identified as outlier at a level of significance $\alpha = 0.05$ using the single Grubbs' test. Therefore, the whole batch was considered homogeneous and retained for further analysis (homogeneity, characterisation and stability). As mentioned before, the homogeneity study was performed by liquid scintillation counting. The results were then evaluated by a one-way analysis of variance (ANOVA). The between-bottle standard deviation s_{bb} and within bottle standard deviation s_{wb} were calculated with the following formulae (ISO 35, 2017)

$$s_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n}}$$
 and
$$s_{wb} = \sqrt{MS_{within}}$$
 (1)

Where:

- MS_{between} is the between bottle variance,
- MS_{within} is the within bottle variance of the measurements used in the between-bottle homogeneity study,
- n is the number of observations per group.

The inhomogeneity that could be hidden by the method repeatability is calculated by the following equation (ISO 35, 2017):

$$u_{bb}^* = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{\nu_{MS_{within}}}} \tag{2}$$

Where:

- $v_{MSwithin}$ is the degree of freedom of MS_{within} .

This expression is based on the consideration that a confidence interval can be established for s_{bb} , and that the half-width of the 95% confidence interval, converted to a standard uncertainty, can be taken as a measure of the impact of the repeatability of the method on the estimate of s_{bb} (ISO 35, 2017). The evaluated relative uncertainties between units u_{bb}^* , hidden by the method repeatability, are given in Table **4**4. The uncertainty related to a possible between-bottle variation is then the larger of u_{bb}^* and s_{bb} in this case s_{bb} . The calculated uncertainty contributions due to inhomogeneity are presented in the **Table 4**.

Table 4. ANOVA test results of ²²²Rn massic activities for JRC-W1 sample.

	JRC-W1
S bb	0.010
S_{wb}	0.010
${u_{bb}}^*$	<0.001
	S_{Wb}

Source: JRC

An alternative evaluation approach uses only the standard deviation of all measured sub-samples. This overestimates the real physical inhomogeneity, since the reproducibility of the measurements (in this case counting statistics) is not corrected for but still contribute to the reference values.

2.3 Stability study

According to the ISO 17043 and ISO 13528 standards, the uncertainty from a stability study originates from two types of stability:

- The short-term stability of the samples which is related to sample transport (i.e. transport between the PT provider and the participants).
- The long-term stability of the samples is linked to sample storage.

The long-term stability of the material was checked. The only way to have increased uncertainty from instability, apart from decay, is radon loss through the sampling container material or its cap. If it can be proven that the containers are radon-tight, then no significant uncertainty contribution can arise from the stability. The tightness of the glass containers and the low diffusion polyethylene LSC vials was checked with GS and liquid scintillation counting, respectively in previous studies (Jobbágy et al., 2019a). An aliquot of a sample with elevated radon activity (appr. 400 Bq/L) was transferred into a sampling bottle or an LSC vial. The glass bottle or polyethylene vial was closed with its cap and measured

(on HPGe detector or in LS counter respectively). Measurement data were recorded until radon had decayed so far that only the background was detected.

The short term stability was checked by measuring samples before and after shipment. During long term stability testing, four bottles were placed in a temperature controlled climate chamber and kept at 10°C and one bottle was stored at 20°C. Radon tightness of the sampling container was tested at elevated temperature (45°C). Long term stability results are presented in Figure 8 and Table 5.

The deviation of the experimental half-life from the literature half-life value was less than 0.4% and 1% in case of GS measuring a sample in a glass bottle and a low-diffusion polyethylene LSC vial, respectively. The covered time-span varied between 18 and 29 days.

One sample was stored at 45°C in a climate chamber (Memmert GmbH) for three days in the same sampling bottle type as used for the PT samples. The bottle was also vigorously shaken manually for 5 minutes to simulate extreme road transport vibration. The radon massic activity was measured before and after the heat treatment and shaking with low-background GS until good counting statistics were reached (< 1%). If the bottle is fully filled without bubbles, tightly capped and if a proper storage container is used then normal air/road transport can deliver samples without significant detectable ^{222}Rn loss, even when the samples are exposed to elevated ambient temperature.

Long-term stability samples (n = 5) were measured by HPGe gamma-ray spectrometry for several weeks (i.e. covering the whole period between sampling-reporting dead line) to confirm that there was no other radon loss than radioactive decay during the PT exercise. Deviation of the experimental half-life from the literature values was considered as uncertainty arising from short term and long term stability which can be an overestimation of the real uncertainty from stability. However, the potential radon loss can be hidden by the method repeatability, therefore when the uncertainty components from stability studies are established, one has to correct for it during calculation. On the basis of the SoftCRM calculation data the uncertainty due to transport and storage conditions (i.e. short term and long term stability) was found to be < 2.0% as presented in **Table 5**.

Table 5. Summary of short and long term stability tests. Data includes the total number of measurements taken in the covered period, the deviation of experimental half-life from the literature value and the deviation of radon-222 massic activity from the reference value.

Measurement data per bottle	Covered period	Average deviation from half-life	Average deviation from reference value (uncertainty due to transport and storage)
33-107	19/10/2018-02/12/2018 (half-life) 19/10/2018-16/11/2018 (ref. value)	0.4%	2.0%

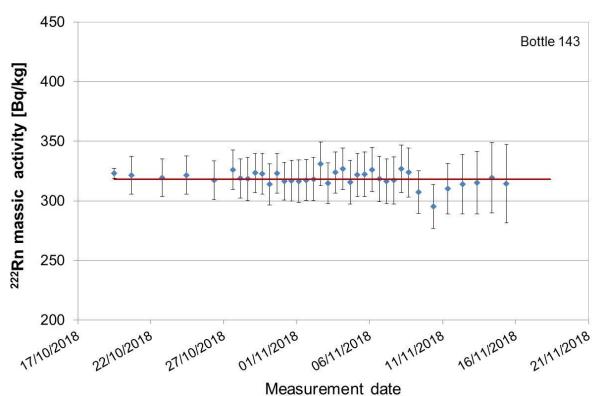


Figure 8. Radon-222 massic activities from the long term stability study.

The average deviation of the individual results of the stability study from the reference value was found to be <1.0%. However, we decided to use the highest uncertainty value from the stability study for the reference value uncertainty which was 2.0%.

2.4 Establishing reference values

For the radon-in-water PT samples, uncertainties from the long-term stability (u_{lts}) and short term stability due to transport conditions (u_{sts}) were included as a sum of the two components. As required by ISO 17025, an extra uncertainty component related to sampling (u_{smpl}) was also introduced due to possible interferences during sampling.

The combined uncertainty u_{ref} of the reference value can be estimated as

$$u_{ref} = k \times \sqrt{u_{char}^2 + u_{bb}^2 + u_{sts}^2 + u_{lts}^2 + u_{smpl}^2}$$
(3)

where

- k: coverage factor (k=1) at ~ 68% confidence interval
- u_{char}: combined standard uncertainty from the characterisation study,
- u_{bb}: uncertainty related to possible between bottles inhomogeneity,
- u_{sts}: uncertainty related to the possible short-term instability of the samples
- u_{lts}: uncertainty related to the possible long-term stability of the samples (longer than the duration of the comparison-exercise),
- usmpl: uncertainty related to sampling.

The reference value of the massic activity for the ²²²Rn is presented in **Table 6** and the uncertainty components are presented in **Table 7**. The main contribution to the uncertainty of the reference values comes from sampling and characterisation. The uncertainty from short and long term stability may be overestimated as the uncertainty from the counting statistics increased during the last two weeks of the measurements. In the first four weeks when better counting statistics were achieved, the uncertainty from the combined short term and long term stability was found to be below 1%.

Table 6. The reference ²²²Rn massic activity value (A_{ref}) in the REM 2018 proficiency test samples with its combined standard uncertainty (u_{ref}) with a coverage factor k = 1.

PT sample code	²²² Rn massic activity (Bq/kg)	Reference date
JRC-W1	318 ± 16	18 October 2018

Source: JRC

Table 7. The relative uncertainty of the reference value (u_{ref} , k=1) and its contribution from the characterisation study u_{char} and the relative homogeneity contributions u_{bb} .

PT sample code	<i>U_{char}</i> (%)	<i>u_{bb}</i> (%)	$u_{sts}+u_{lts}$ (%)	<i>u_{smpl}</i> (%)	<i>u_{ref}</i> (%)
JRC-W1	2.6	1.0	2.0	3.5	4.9

3 Reported results

From the 101 participating laboratories, 28 participants used more than one analytical method (**Table 8**). Therefore, in total 135 measurement results were reported.

Table 8. Overview on the number of participants and reported results for JRC-W1 PT samples.

Registered participants	Reporting participants	Results submitted	Filled in the questionnaire
101	101	135	97

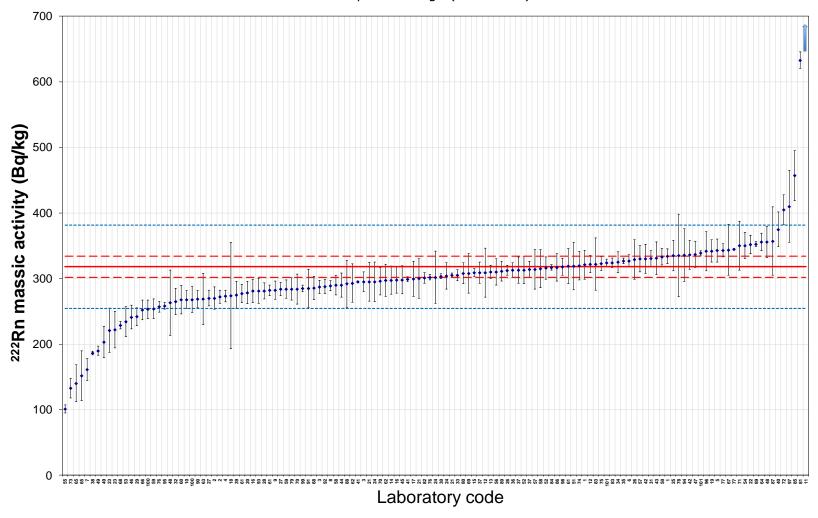
Source: JRC

The participants were requested to submit their results together with their combined standard uncertainties (coverage factor k=1). If the reported coverage factor differed from 1 then we recalculated the uncertainties for k=1.

The participants' results and their scoring using percentage difference from the reference value, z-score, $\zeta(\text{zeta})$ -score according to ISO 13528:2015 are presented in Annex 10. The detailed numerical measurement results as reported by the participants, further information on accreditation and used analytical methods can be found in the performance evaluation JRC report (Jobbágy and Hult, 2019c).

The reported 222 Rn massic activities in Bq/kg with their corresponding combined standard uncertainties (k=1) are plotted in ascending order in **Figure 9**. The solid red line indicates the reference 222 Rn massic activity (A_{ref}). The dashed red lines show the combined standard uncertainty (u_{ref} , k=1) of the reference values. Blue dashed lines represent the reference range within the **standard deviation for proficiency assessment** (σ_{PT}) **which was set to 20%**. The participants' identification numbers are indicated with the results.

Figure 9. The 222 Rn massic activity measurement results reported by participants with their combined standard uncertainties (k = 1). The solid red line: reference 222 Rn massic activity (A_{ref}). The red dashed lines: the uncertainty on the reference value (u_{ref}). Blue dashed lines: acceptance range (A_{ref} \pm 20%).



The presence of statistical outliers among the reported results was investigated using the Grubbs's test at a significance level of $\alpha = 1\%$ (two-sided) according to ISO/IEC 5725-2 (ISO, 1994).

One result from participant 11 was indicated as outlier in the Grubbs's test in the first run.

The ratio between the maximum and minimum reported 222 Rn massic activity (A_{max}/A_{min}) was approximately 27.

3.1 Uncertainties

Participants had to submit their results together with their combined uncertainty (coverage factor k=1). If the reported coverage factor differed from 1 then we recalculated the uncertainties for k=1. Furthermore, participants were requested to provide information on their uncertainty budget. Although 97 participants submitted the questionnaire only 61 (63%) of them provided information on the uncertainty budget.

The standard measurement uncertainty from a laboratory $u(A_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (Case a": $u_{min} \le u(A_i) \le u_{max}$). u_{min} is set to the standard uncertainties of the assigned values $u(A_{ref})$ but in this case excluding the uncertainty from sampling and stability study; $u(A_{ref}) = 2.8\%$. In general, it is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with (much) smaller measurement uncertainty than an expert laboratory which establishes the assigned value or the uncertainty of available calibration standards. u_{max} is set to the standard deviation accepted for the PT assessment ($\sigma_{PT} = 20\%$).

- Case "a" (blue colour in **Figure 10**): $u(A_{ref}) \le u(A_i) \le \sigma_{PT}$,
- Case "b" (orange colour in **Figure 10**): If $u(A_i) < u(A_{ref})$; the laboratory may have underestimated its measurement uncertainty,
- Case "c" (red colour in **Figure 10**): If $u(A_i) > \sigma_{pt}$; the laboratory may have overestimated its measurement uncertainty.

Note the colours in **Table 9** and **Figure 10**: Case "a" in blue, Case "b" in orange, Case "c" in red.

Table 9. Number of measurement results as a function of uncertainty cases.

PT sample	Case "a"	Case "b"	Case "c"
JRC-W1	112 (82.9%)	21 (15.6%)	2 (1.5%)

Source: JRC

The uncertainty budgets were correctly established for the majority of the submitted results. However there may be issues in case of 23 measurement results representing 17% of the submitted results. These have to be investigated by the participants. An overview on the relative standard uncertainty distribution as a function of the individual measurement results is plotted in **Figure 10**.

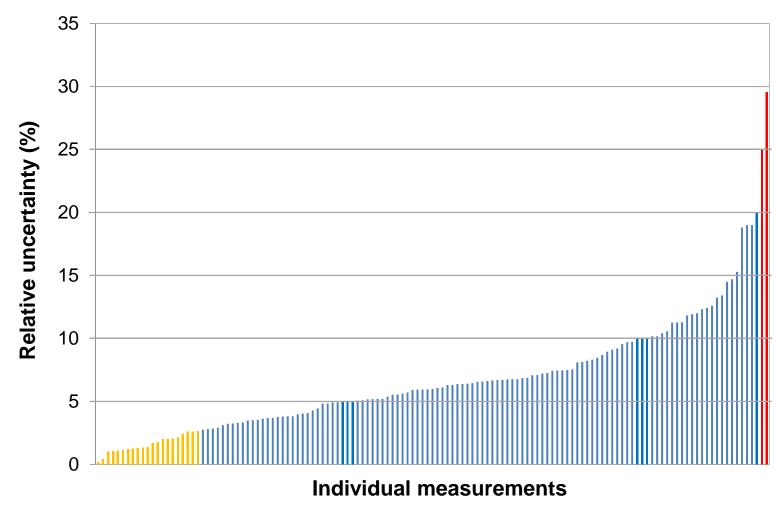


Figure 10. Relative standard uncertainty distribution of the individual measurement results.

4 Scores, evaluation and comparison of results

The participants` results were evaluated with respect to the reference values using percentage difference from the reference value, z-score, zeta-score and the E_n number according to ISO 13528:2015. Additionally, PomPlots were presented. An estimate of the uncertainty of the reported results was required from each participating laboratory. The detailed calculation of performance evaluation scores is presented in Annex 11 and Annex 12. The participants` scores (percentage difference, z and zeta score, E_n number) and PomPlot are displayed in Figure 11-15.

4.1 Standard deviation for proficiency assessment (σρτ)

The standard deviation for proficiency assessment (σ_{PT}) was initially set to 15% but the PT organiser decided to change it because an additional component related to adsorption of radon decay progenies to the sampling bottle was suggested by Cassette (2019) and Mitev (2019) during the follow-up JRC workshop held in Geel between 26 and 29 March 2019. This can affect the GS measurement results as it changes the measurement geometry and efficiency in case of efficiency transfer calibration. The detection efficiency would be underestimated thus consequently the reference values may be overestimated. Since the PT organiser could not confirm these findings yet, the reference value did not change on this ground but **the standard deviation for proficiency assessment** (σ_{PT}) was increased to 20% to compensate for this.

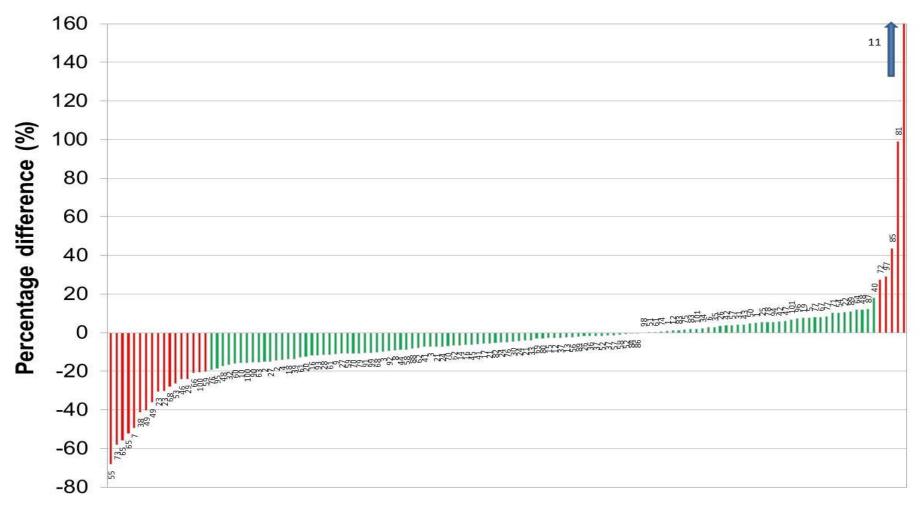
4.2 Percentage difference (%)

The percentage differences are plotted in ascending order in a deviation chart, where too low or too high measurement values become more visible (**Figure 11**). For the environmental radioactivity measurements the criterion of \pm 10-30% difference from the reference value is usually applied but on the basis of our experience on previous radon-in-water PTs, we used 20% as criterion.

About 84% of all the reported results were within 20% deviation. Approximately 16% of all results (22 out of 135 measurement results) deviated more than 20% from the reference values. There was one outlying measurement result (ID 11) which is indicated by the blue arrow symbol. It is also visible that the average of the submitted results is lower than the reference value.

From the results obtained by emanometry, there were nine measurement results (22.5% of the emanometry results) outside the one standard deviation of the proficiency test (σ_{PT}). The liquid scintillation measurements performed better. Nine results (13.2% of the LSC results) were outside one σ_{PT} . In case of gammaray spectrometry only three measurement results (11.1% of the GS results) were outside one σ_{PT} . Gamma-ray spectrometry is the best performing from the three measurement methods.

Figure 11. Participants` percentage differences for JRC-W1 sample plotted in ascending order. Green colour indicates results within the ± 20% difference from the reference value, red colour indicates outside this range. The participants` identification numbers are indicated on the horizontal axis.



4.3 Z-scores

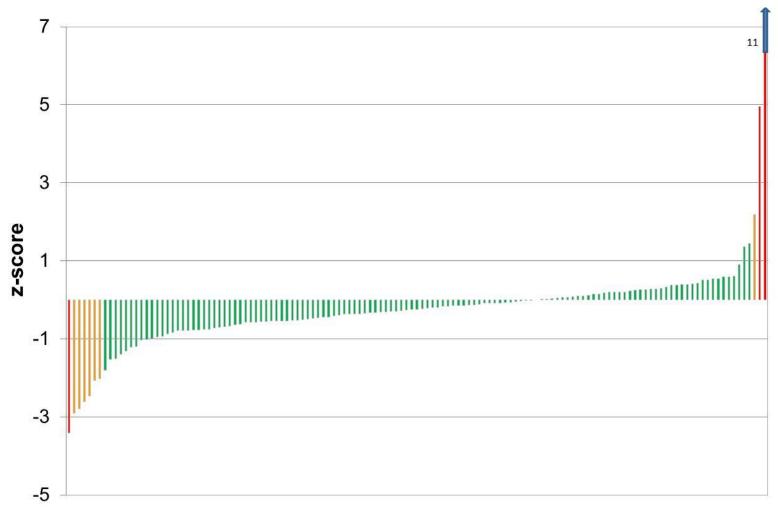
The z-score compares the participant's deviation from the assigned value with the standard deviation of the proficiency test assessment (σ_{PT}), used as common criterion.

The interpretation of the z- and ζ -scores is done according to ISO 13528:2015. The following scores and colour codes are used in Figure 12-14 and Table 11 in Annex 10:

- |score| ≤ 2 acceptable performance (green),
- 2 < |score| < 3 warning signal (orange),
- |score| ≥ 3 unacceptable performance (red).

Of all results 93% were satisfactory.

Figure 12. Participants` z-scores for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable performance, orange indicates warning signal, and red colour indicates unacceptable performance.

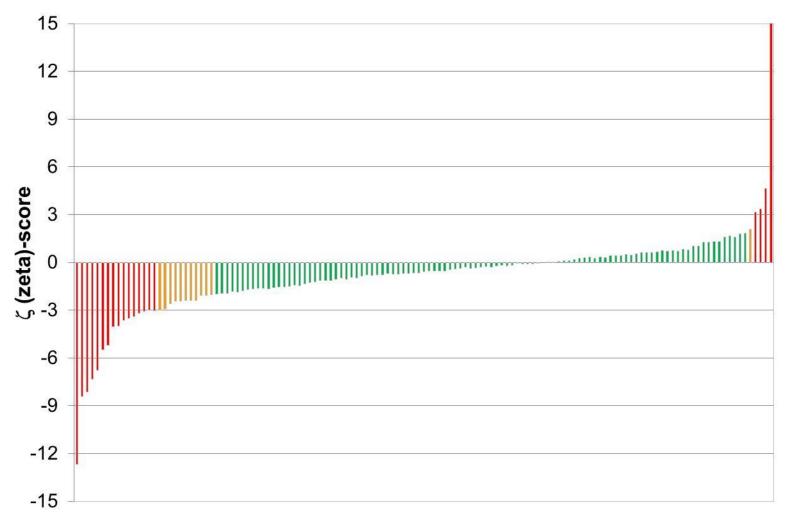


4.4 Zeta (ζ**)-scores**

The ζ -score states whether the laboratory's result agrees with the assigned value within the respective uncertainty. An unsatisfactory ζ -score can either be caused by an inappropriate estimation of the reported value or uncertainty, or both.

When the ζ -scores are evaluated, the satisfactory performances dropped to approximately 76%. This indicates that some participants have problems establishing a realistic uncertainty budget.

Figure 13. Participants` zeta-scores for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable performance, orange indicates warning signal performance, and red colour indicates unacceptable performance ("action" signal).



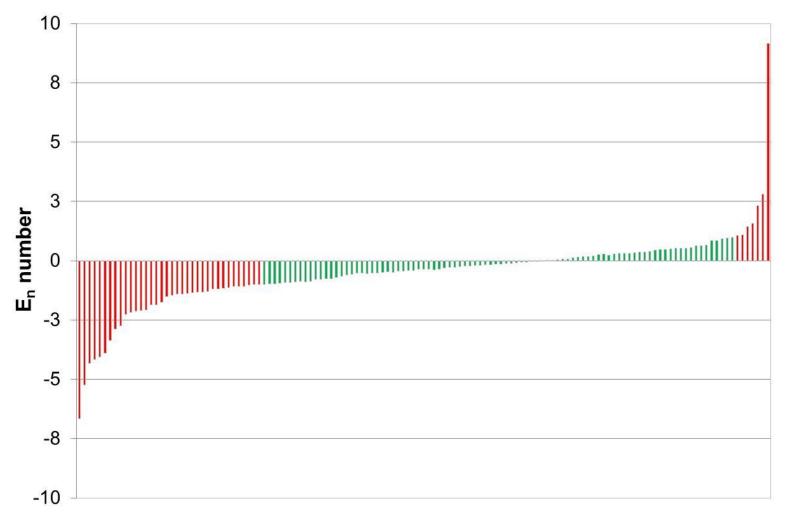
4.5 E_n-numbers

In order to take into account the expanded uncertainty of the reported value and the reference values, a performance test using E_n numbers was applied (ISO 13528, 2015).

When uncertainties are estimated according to the Guide to the Expression of Uncertainty Measurement (GUM) (ISO, 2008), a measurement result with its uncertainty interval giving a level of confidence of 95% should overlap with the reference value and its expanded uncertainty. Therefore, E_n numbers are interpreted as following:

- If $|E_n| \le 1$, the laboratory values can be considered acceptable,
- If $|E_n| > 1$, the laboratory values can be considered unacceptable, they differ significantly from the reference values. The sources of deviation should be investigated and corrected, "action signal".

Figure 14. Participants` E_n-numbers for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable results and red colour indicates unacceptable results ("action signal").

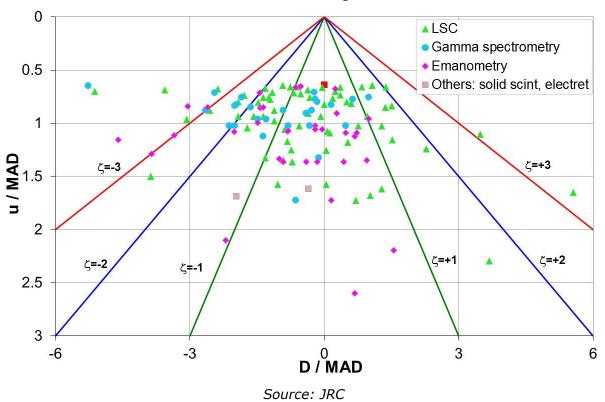


Source: JRC

4.6 PomPlot

In order to compare the results, a modern type of graph – PomPlot – that underlines the importance of the assigned uncertainties is applied. The PomPlot, an intuitive graphical method, is used for producing a summary overview of the participants' results (Spasova et al., 2007). It displays the relative deviations (D/MAD) of the individual results from the reference value on the horizontal axis and relative uncertainties (u/MAD) on the vertical axis (**Figure 15**). For both axes, the variables are expressed as multiples of MAD, which is the median of the absolute deviation from the reference value. The detailed PomPlot interpretation is presented in Annex 12.

Figure 15. PomPlot of the 222 Rn data sorted according to measurement method. Red square indicates the reference value. Green, blue and red solid lines represent ζ -scores = 1, 2 and 3, respectively. One outlying result cannot be displayed as it lays outside the X-and Y-axis range.



4.7 Comparison of proficiency tests

The main features, performance indicators of some PT for ²²²Rn in water from the past are compared to the JRC REM 2018 PT in **Table 10**.

Table 10. Comparison of intercomparisons and proficiency tests.

PT/ILC material	Acceptable results, number of participants	орт (%)	²²² Rn activities	Traceability	Comments	Reference
Ground waters from drilled wells	73% R1 sample 82% R2 sample 23 participants	10	R1 ≈ 3200 Bq/L R2 ≈3700 Bq/L	-	Two PT samples; 6-7% skewed towards lower values	Mäkinen and Hanste, 2009
Water from a drilled well	80 % 8 participants	20	25000 Bq/L	-	Lower results due to sample transfer from sampling bottle into measurement container	Vesterbacka et al., 2010
Low ²²⁶ Ra content ground water	14 participants	10-30	45000 Bq/L	-	LSCs only; Two ILC samples ; 5-10% skewed towards lower values (sample preparation and calibration)	Möbius and Salonen, 2001
In-house prepared	86% 21 participants	25	693 Bq/L	via ²²⁶ Ra standard	Results are skewed towards lower values (sample transfer, sample was exposed to air in syringe) Higher results: possible error: efficiency calibration	Kitto et al., 2010

PT/ILC material	Acceptable results, number of participants	орт (%)	²²² Rn activities	Traceability	Comments	Reference
Thermal water	Not calculated 4 participants	not established	Sample 1 ≈ 80 Bq/L Sample 2 ≈250 Bq/L Sample 3 ≈355 Bq/L	-	Three PT samples; Results were within 20% of the mean; Deviation due to calibrations, radon loss during storage, issues with sampling of running water	Neznal et al., 2014
Ground water samples	78% (76% GS; 82% LSC) 29 participants	G1:17 G2:25	G1: 399 Bq/L G2: 2732 Bq/L	-	Two PT samples; results skewed to lower values	Björklöf et al., 2017
Hot spring water	95% 17 participants 20 results	20	112 Bq/L	-	One PT sample in a 25 L canister; temperature was 34–37°C; Leak was observed from the LDPE sampling container	Celaya González et al., 2018
Spring water(S1) Well water (S2)	Total= 89% S1=90% (20 results) S2 = 88% 17 results 14 participants	15	S1 = 601 Bq/kg S2 = 2288 Bq/kg	via ²²⁶ Ra standard; mass standards	Two PT samples	JRC Pilot- PT 2017 report
Spring water(W1)	84% 101 participants 135 results	20	W1=318 Bq/kg	via ²²⁶ Ra standard; mass standards	One PT sample	This report; Jobbágy and Hult, 2019c

Source: JRC

In most of the PTs, ground water from wells was used as PT material. Some waters had an exceptionally high 222 Rn massic activity, which is not very likely in treated drinking waters due to extensive degasification during the water treatment procedure.

The standard deviation for proficiency test (σ_{PT}) from the reference values varied between 10-30%. The reported results were in average lower than the reference value. This is mainly due to factors like additional sample transfer, sample preparation, radon loss during storage or sample exposure to air. In one case, a possible cause for higher results due to efficiency calibration was mentioned.

It can be concluded that the general performance of participants in the different European radon-in-water PTs/ILCs is good. The majority reports results within 10-25% deviation from the reference values. The participants in the JRC pilot-PT2017 performed slightly better than participants in the other listed PTs/ILCs. The participants' performance in the REM2018 PT is comparable to the other PTs.

5 Information on the participating laboratories: organisational and technical details

Participants were requested to fill in a questionnaire (Annex 7). In this questionnaire the PT coordinator requested information on the participant laboratories' (i) experience, (ii) technical details on their methods and involvement in standardisation processes (iii). The participants were given the opportunity to give feedback and comments on the organisation of the PT. All feedback will, if relevant, be considered in the future PTs.

Participants were requested to use their routine analytical procedures. They were free to choose the analytical method. The information in this chapter was provided in the questionnaire by 97 out of the 101 participants.

Information on accreditation, application of standards:

- 42 out of 97 have ISO 17025 accreditation
- 36 are involved in international/national standardisation process, and from the non-involved organisations <u>71 would like to be involved</u>,
- 58 out of 97 performed their analysis according to ISO 13164 part 2-4.
- 28 followed other standard methods (e.g. EPA 913.0, ASTM_D5072D, national standards)

Type of laboratory:

- Measurements of radioactivity in the environment: 52 participants
- Research and development; Measurements of radioactivity in the environment: 21 participants
- Research and development: 11 participants
- Private commercial company: 3 participants
- Water company or company owned by municipality: 2 participants
- accredited calibrating laboratory, food and feed control laboratory, university, regulatory body: 1 participant in each type respectively.

Laboratory working according to a quality management system?

81 work according to a quality management system. The most commonly used quality management systems are: ISO 17025, ISO 9000 series, ISO/IEC 17043 and ISO 13528, ISO 14000, ISO 14001, EN 45000 series, DSTU ISO 10012:2005, ISO 14001.

How long is radon-in-water analysis performed routinely at your organisation (in years)?

Laboratories have been dealing with radon-in-water analysis between 1 and 35 years with an average of 11 years.

How many measurements of this type does your laboratory perform per year?

< 25: 38 participants 25-100: 28 participants > 100: 31 participants

What is the typical range of radon activity concentrations measured (Bq/L)?

1-50 Bq/L: 75 participants 50-100 Bq/L: 11 participants 100-500 Bq/L: 9 participants 500-1000 Bq/L: 1 participant. > 1000 Bq/L: 1 participant.

Did you perform the test in compliance with the ISO 13164 standards?

58 out of 97 participants followed one of the ISO 13164 standards regarding radon measurement in water.

5.1 Methods used by the participating laboratories

The methods used by the participating laboratories can be grouped into three categories based on the detection technique: Liquid scintillation counting, Gamma-ray spectrometry and emanometry. Liquid scintillation counting was the most often used method in this PT by 68 laboratories (50%). While gamma-ray spectrometry and different emanometry approaches were applied by 27 (20%) and 40 (40%) laboratories respectively. Note that there were participants who used multiple methods.

Regarding the test sample preparation approach which is one of the most crucial steps in terms of radon-loss, 22 measurement results were provided by using direct measurement of the sample in the sampling container. The rest transferred the sample (e.g. degassing, liquid extraction technique, simple sample transfer into another container) which could cause loss of radon from the sample.

In case of LSC, the majority of the laboratories (50) used two-phase cocktails, 16 used water miscible cocktails.

The sample volume used for a single measurement varied between 2 mL-1.15 L.

The material of sample containers were glass (51), HDPE (24), Metal (5), Teflon (12) or in 6 cases other materials (e.g. PET, PVC, polypropylene).

Efficiency calibration approaches

The different approaches for efficiency calibration used are listed below.

- Calibrated in radon chamber (²²²Rn in air calibration procedure)
- Calibration certificate/formula/coefficient provided by manufacturer or national metrology institute
- Degassing efficiency from experiments
- Standard ²²²Rn solution prepared by a national metrology institute

- Inter-calibration with other techniques/laboratories
- Multi-nuclide gamma source and efficiency transfer
- Calibration with ²²⁶Ra certified reference solution, measurement after equilibrium (14-30 days) set of standards from 3 Bq/L-1000 Bq/L
- Absolute activity measurements from triple-to-double coincidence ratio (TDCR) method
- LSC efficiency was assumed
- Polynomial minimum squared fitted to the mix nuclides calibrated isotopes.
- accumulation method
- NIST polyethylene encapsulated ²²⁶Ra/²²²Rn emanation standard

The following types of measurement instruments were used:

Quantulus, TriCarb, Alphaguard, RAD7, HPGe detectors, Lucas cell (Heger), Guardian, RackBeta 1217, NaI(Tl), Wallac 1414 LSC counter, Hidex, Luk 3P/3C with LUKVR system, PRM-145 (Portable Radon Monitor), own developed instrument, RadonMapper, MIAM degassing system, BetaScout/Triathler (HIDEX), etc.

List major uncertainty contributing parameters:

- Count rate, calibration
- Emanation procedure
- Sample mass, system volume, pressure, pipetting
- Statistical uncertainty, activity of the used standard
- Sampling time, Source variability (Homogeneity)
- Chemical yield, Laboratory repeatability, Ra-226 standard
- Uncertainties: geometry, summing correction, efficiency transfer
- Radon loss during sampling
- Cell sensitivity, humidity (Si detectors)
- unknown, the higher result out of two replicates is reported, sometimes differences are up to 25%
- Radiation background subtraction and others

The typical test sample volume needed for a single analysis:

LSC: 6-10 mL

Gamma spectrometry: 50-1150 mLLucas cell/emanometry: 40-500 mL

Did you observe any interference that might influence your measurement results?

Yes: seven labs.

There might have been a small leakage in the emanometry system.

- The way and time of probes storage.
- Temperature, background level of indoor sampling.
- Possible loss of activity by passing the sample into the measured sampler.
- The variance between background radiation on the day of the measurement of the sample and the day of the background measurement could interfere with the result, since our laboratory is not low background and we have radon progeny energies in the background.
- During the measurement the used radon monitor had a malfunction and I had to reconnect the bottle to another radon monitor that was available.
- The radon half-life was lower than the data from literature.

How did you calculate the final result?

- from a single analysis: 28 participants

- from the mean of replicates: 69 participants

5.2 Participants' feedbacks

The participants had the opportunity to comment any aspect of the proficiency test. In general positive feedback was received from the participants. Participants appreciated this PT as seen from their scores (average score given by the participants was 9.3 out of 10) which is very positive. The proposals to improve the organisation of a radon-in-water PT are listed below. A note from the PT organiser is given in italic after each comment (when relevant). The full list of comments as submitted by the participants is presented in Annex 8.

Remarks:

- Difficulties with the webpages
- Received a lot of information about the PT
- More information about how and when to perform the analysis
- The period between the preliminary and the final report is long

Organiser's comment: we try to reduce this period but the detailed evaluation takes some time.

- Presence of an air bubble in the sample
- Time gap between sample preparation and delivery was too long

Organiser's comment: We needed this time for transporting samples back to the laboratory, the pre-homogeneity screening, packaging.

Reporting results why not with coverage factor k=2?

Organiser`s comment: we considered the zeta score as one of the main scores and for the calculation the combined standard uncertainties with coverage factor k=1 are used.

- Send the reference time at the same time with samples
- Why not one common reference time?

Organiser's comment: The water sample is from a natural underground aquifer with a certain residence time. As soon as water leaves the geological environment Rn-222 starts decaying in the sampling bottle. The sampling

period spanned more than 4 hours therefore the measurement results would be already biased. The difference in massic activity between the first and last sample would be about 3%.

 A lab normally doesn't do manual calculations: it took a lot of time figuring out how to do the calculations.

Organiser's comment: a course on calculations would be desirable and help in calculating the decay.

In one case a "small leakage" was observed.

Organiser`s comment: bottles were closed while they were still submerged under water, water was thus trapped in threads which may be released during transport.

Suggestions:

- Provide more samples with different activities
- Schedule the reception of PT samples to the beginning of the week
- An activity around the parametric level (100 Bq/L) and about 10 Bq/L would be appreciated

Organiser`s comment: In case of 10 Bq/L more rapid shipment might be needed, the initial ²²²Rn activity would decay very quickly close to the detection limit of the most used analytical techniques.

- Put the questionnaire in the same page as the results

Organiser's comment: we will try to integrate the questionnaire and the reporting pages next time.

- Reporting: electronically, avoid paper
- Preference for using own bottle
- Check the validity of the calibration used and the routine working conditions
- Work with a bigger sample volume

Organiser's comment: it would be difficult and more expensive logistically.

Further comments:

- Well organised transport, protection of samples (temperature logger, transport box)
- Clear instructions
- Transport was relatively rapid and without complications but delays with shipments involving custom procedure
- No bottle was broken

5.3 Sources of interferences

There are some potential sources of interferences mentioned by the participants and the PT organiser (Jobbágy et al., 2019c).

- Bubble formation in the sampling container,
- Additional pouring/sample transfer,
- Elevated radon background in the laboratory,
- Adsorption of radon daughters on the glass bottle (Cassette, 2019; Mitev, 2019).

5.4 Reported impacts of this PT

Some participants could already use the results or the materials to improve the quality and reliability of their analytical results.

- Interesting and educating PT exercise for a "new in the field" participant,
- Bottles were useful, new procedure is adopted to that geometry,
- PT was useful regarding the quality system and the accreditation procedure.

6 Summary and conclusions

A proficiency test on the determination of the massic activity of ²²²Rn in drinking water was organised by JRC-Geel in October 2018. The proficiency test material with an elevated ²²²Rn massic activity was collected from a natural water source in Austria. The reference value traceable to SI units was established by calculating the power moderated means of the HPGe gamma-ray spectrometry measurements. Before organising the REM 2018 PT the sampling transport-storage conditions were tested and optimised to secure radon tightness during the whole PT chain from sampling until measurement. These experiments were done during different laboratory tests and a pilot-proficiency test.

6.1 Conclusions about PT performances

The participants were requested to treat and measure PT samples according to their routine procedures. Every participant submitted at least one measurement result, 30 participants submitted multiple measurement results from different analytical methods.

The results from LSC measurements showed less scatter around the reference value than GS and emanometry without any observable shift in the results. However, the mean results for GS and emanometry were lower than the reference value. A reason for this difference could be the additional transfer of a larger volume sample from the sampling container to a measurement container which makes these techniques more prone to radon loss. Another possible reason could be incorrect calibration of instruments.

The performance of the participating laboratories was evaluated with respect to the reference values using relative deviations, z-score, zeta-score and E_n -number. All results were also evaluated according the applied analytical method to check for method dependency.

The scores of the 135 measurement results submitted by the 101 participating laboratories were found to be satisfactory with only a few exceptions. 84% of the submitted results with their combined uncertainties were within the preestablished criteria ($\sigma_{PT}=20\%$). Only 1 outlying measurement result was identified by the Grubbs's test. The reason for this might be that the participant had problems with the instrument which was out of order for some weeks. They were only able to measure the sample a few weeks after reception. Calibration problems could not be excluded as well.

However, when the uncertainty was evaluated (zeta score) less acceptable scores were found: 103 measurement results out of 135 included a proper uncertainty budget, which represents 76% of the measurement results. Underperforming measurement methods need to be reviewed, e.g. to detect possible ²²²Rn loss during manipulation. Furthermore, calibration procedures and uncertainty budgets should be also re-evaluated by some participants.

The direct measurement using gamma-ray spectrometry has an undisputable advantage to other indirect methods: The sampling container can be directly measured on a detector, no sample transfer is needed. No radon loss can be expected through the sampling container. Consequently, it will represent more realistically the original radon activity concentration at the sampling point.

The importance of proper and accurate calibration has to be stressed in case of radon-in-water measurements. The biggest shortcoming at this moment is the availability of metrological traceable radon-in-water reference material. This leads to higher uncertainties due to calibration of direct as well as indirect measurement systems.

Each of the three applied methods (emanomery, gamma-ray spectrometry and liquid scintillation counting) is adequate for radon in water measurements. The methods are relatively well controlled by the participating laboratories. The loss of ²²²Rn during sample preparation leads to reported values that are too low.

6.2 Conclusions for PT organisers

Further conclusions can be drawn for participants but also for PT organisers.

- Influence of sampling approaches: turbulence and bubble free sampling shall be performed,
- Sample storage, transfer: ²²²Rn tightness of sample containers shall be secured,
- Sampling container material: glass is preferred,
- Speed of delivery is crucial,
- Customs procedure: outside the EU might take 2-10 days,
- Differences in local time can be important: reference date should be in Coordinated Universal Time (UTC). Info on the UTC can be found on this website: https://www.bipm.org/en/about-us/

6.3 Future JRC actions

Repetition of this PT can be considered if requested by the member state laboratories, Euratom 35/36 experts and the European Commission's Directorate-General for Energy (DG ENER).

It is considered to organise a radon-in-water sampling PT in the future. This has to be further discussed within the European Commission and the Euratom article 35/36 experts.

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List of abbreviations and definitions

Activity concentration* Activity per unit volume

AGES Austrian Agency for Health and Food Safety

Alab mean laboratory result of massic activity

A_{ref} reference value of massic activity

BIPM Bureau International des Poids et Mesures

D (%) Percentage deviation between the reported and the reference massic

activity

En Performance statistics number
DDEP Decay Data Evaluation Project

DG ENER European Commission's Directorate-General for Energy

EURATOM European Atomic Energy Community

GIG Silesian Centre for Environmental Radioactivity (Katowice-Poland)

GUM Guide to the Expression of Uncertainty in Measurement

HPGe high-purity germanium detector

ILC interlaboratory comparison

ISO International Organization for Standardization

JRC The Joint Research Centre of the European Commission

k coverage factor according to GUM

LOD limit of detection

LSC liquid scintillation counting

Massic activity* Activity per unit mass

MS member states of the European Union

PT proficiency test

REM Radioactivity Environmental Monitoring *
SCK•CEN Belgian Nuclear Research Centre, Belgium

SI Système International d'Unités, International System of Units

SIR Système International de Référence, International Reference System for

radionuclides

U expanded uncertainty according to GUM u standard uncertainty according to GUM

 u_c combined standard uncertainty according to GUM U_{lab} expanded uncertainty of average laboratory result

U_{ref} expanded uncertainty of reference value
UTC Coordinated Universal Time; time standard

σ_{PT} the standard deviation for proficiency assessment

49

^{*} In this report, the matrix was water, which has a density very close to 1. Although we clearly distinguish between massic activity (Bq/kg) and activity concentration (Bq/L), their numerical value would be almost identical.

[#]https://remon.jrc.ec.europa.eu/

List of figures

Figure 1. Sampling arrangement at the Austrian sampling site 7
Figure 2. PT test samples for homogeneity and reference measurements
$\textbf{Figure 3.} \ \textbf{Interior view of six transport boxes whilst being prepared for shipment.} \ \dots \dots 9$
Figure 4. Subsampling for LSC measurements
Figure 5. Massic activity results of ^{222}Rn in the JRC-W1 PT sample. Yellow triangles GS results, blue diamonds LSC results. All uncertainties are combined uncertainties at 1σ level (k = 1). The solid red line: the power moderated mean of the GS results; the dashed red lines: combined uncertainty of the mean (k = 1)
Figure 6. On-site screening results obtained by GS for JRC-W1 sample16
Figure 7. Homogeneity study results obtained by LSC for JRC-W1 sample17
Figure 8. Radon-222 massic activities from the long term stability study20
Figure 9. The 222 Rn massic activity measurement results reported by participants with their combined standard uncertainties (k = 1). The solid red line: reference 222 Rn massic activity (A _{ref}). The red dashed lines: the uncertainty on the reference value (u _{ref}). Blue dashed lines: acceptance range (A _{ref} \pm 20%)
Figure 10. Relative standard uncertainty distribution of the individual measurement results25
Figure 11. Participants` percentage differences for JRC-W1 sample plotted in ascending order. Green colour indicates results within the \pm 20% difference from the reference value, red colour indicates outside this range. The participants` identification numbers are indicated on the horizontal axis
Figure 12. Participants` z-scores for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable performance, orange indicates warning signal, and red colour indicates unacceptable performance
Figure 13. Participants` zeta-scores for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable performance, orange indicates warning signal performance, and red colour indicates unacceptable performance ("action" signal)31
Figure 14. Participants` E _n -numbers for JRC-W1 sample plotted in ascending order. Green colour indicates acceptable results and red colour indicates unacceptable results ("action signal")
Figure 15. PomPlot of the 222 Rn data sorted according to measurement method. Red square indicates the reference value. Green, blue and red solid lines represent ζ -scores = 1, 2 and 3, respectively. One outlying result cannot be displayed as it lays outside the X-and Y-axis range
Figure 16. Interpretation of a PomPlot

List of tables

Table 1. Timeline of the radon-in-water REM 2018 PT exercise. 6
Table 2. Comparison of background counts and MDA in LSC using glass and low diffusion polyethylene vial. High energy beta protocol without alpha-beta discrimination was set, channels between 50 and 1024 were considered (120 min data acquisition)13
Table 3. Typical uncertainty budgets for GS and LSC radon-in-water measurements for a single measurement of a sample at the 1σ level as relative values (%). The combined uncertainty is the square root of the quadratic sum of all components (k = 1)15
Table 4. ANOVA test results of ²²² Rn massic activities for JRC-W1 sample18
Table 5. Summary of short and long term stability tests. Data includes the total number of measurements taken in the covered period, the deviation of experimental half-life from the literature value and the deviation of radon-222 massic activity from the reference value.
Table 6. The reference 222 Rn massic activity value (A _{ref}) in the REM 2018 proficiency test samples with its combined standard uncertainty (u _{ref}) with a coverage factor $k=1,21$
Table 7. The relative uncertainty of the reference value (u_{ref} , $k=1$) and its contribution from the characterisation study u_{char} and the relative homogeneity contributions u_{bb} 21
Table 8. Overview on the number of participants and reported results for JRC-W1 PT samples. 22
Table 9. Number of measurement results as a function of uncertainty cases24
Table 10. Comparison of intercomparisons and proficiency tests
Table 11. Participants` results and their scores as percentage difference (D%), z-score, ζ (zeta)-score for JRC-W1 sample. ²²² Rn massic activity values (A _{ref}) with their combined standard uncertainties (u _{ref}) with a coverage factor $k = 1$ 88

Annexes (see next page)

Annex 1. Nomination request, e-mail, invitation letter

Ref. Ares(2018)3738457 - 13/07/2018



EUROPEAN COMMISSION

DIRECTORATE-GENERAL JOINT RESEARCH CENTRE

Directorate G - Nuclear Safety and Security

Standards for Nuclear Safety, Security and Safeguards

Geel, 13 July 2018

Subject: Article 35-36 of the Euratom Treaty

Nomination request: EC Proficiency Testing ²²²Rn activity concentration measurements in water organised under the ICS-REM* programme

Ms Barbara Vokal Nemec Mr Alar Polt

Ms Beata Vilimaite Silobritiene Mr Andras Donaszi-Ivanov
Ms Carmen Rey del Castillo Mr Andris Abramenkovs
Ms Claudia Landstetter Mr Antonis Maltezos
Ms Elena Simion Mr Árpád Vincze

Ms Eugenie Vial Mr Christian Katzlberger
Ms Hristina Halachliyska Mr Fabrice Leprieur
Ms Konstantina Kehagia Mr Giancarlo Torri
Ms Maria José Bação Madruga Mr Giuseppe Menna
Ms Marielle Lecomte Mr Josef Peter

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Ms Nathalie Reynal
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Mr Jurgen Claes
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Mr Martijn van der Schaaf

Ms Sarah FallonMr Michalis TzortzisMs Sofía LuqueMr Michel BaudryMs Sonia FontaniMr Michel CindroMs María Teresa SánchezMr Ondřej Chochola

Mr Pål Andersson Mr Paul Brejza Mr Pawel Lipinski Mr Pieter Kwakman Mr Reimund Stapel Mr Selwyn Runacres Mr Sven Poul Nielsen

Mr Vladimir Jurina

Dear colleague,

As you know, EU Member States are obliged under Art. 35 and 36 of the EURATOM Treaty to inform the European Commission (EC) on a regular basis of the radioactivity levels in their environment. In order to obtain more information on the measurement methods and on the quality of the values reported by the Member States, the Joint Research Centre of the European Commission is organising Proficiency Testing (PT) exercises for the MS laboratories. These PTs are organised under the ICS-REM* programme in which the EC is testing measurement capabilities as well as providing technical support to the participating laboratories.

After discussions with the Directorate-General for Energy of the European Commission and during the Euratom Treaty Art. 35-36 meetings, it was agreed that next PT exercise will be on ²²²Rn activity concentration measurements in water.

^{*}ICS-REM – International Comparison Scheme for Radioactivity Environmental Monitoring

The tentative schedule for the PT is as follows:

- July 2018 Announcement to Article 35/36 Experts and DG ENER
- September 2018: official invitation sent to participants,
- October 2018: PT material (natural water) shipped to participants,
- November 2018: participating laboratories results submitted to the JRC,
- December 2018: preliminary report distributed,
- 2019 t.b.d.: workshop and training course organised for participating laboratory practitioners.

We would like to ask you to investigate which laboratories in your country you would like to see participating and provide us with the contact data of the nominated laboratories (responsible person, complete postal address, telephone, and e-mail). To proceed according to the plan, we require your (nationally coordinated) response by **24 August 2018**. Should you have difficulties to nominate laboratories before the deadline, please let us know as soon as possible.

Please, send your replies to the functional e-mail box: JRC-GEE-REM-COMPARISONS@ec.europa.eu

We look forward to hearing from you with the laboratory nominations.

Yours sincerely,

Viktor Jobbágy Mikael HULT Petya MALO

Project Coordinator Team Leader** Logistics Assistant



European Commission Joint Research Centre (JRC) Nuclear Safety and Security Standards for Nuclear Safety, Security & Safeguards Retieseweg 111 B-2440 Geel, Belgium

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De Cort (JRC Ispra)
Messrs. Arjan Plompen, Mikael Hult, Katarzyna Sobiech-Matura, Petya Malo (JRC Geel)

JRC GEE REM COMPARISONS

From: JRC GEE REM COMPARISONS Sent: 03 September 2018 15:38

Cc: HULT Mikael (JRC-GEEL); MALO Petya (JRC-GEEL); PLOMPEN Arjan (JRC-GEEL); DE CORT Marc (JRC-ISPRA); TANNER Vesa (ENER); RYAN Alan (ENER)

CORT Marc (JRC-ISPRA); TANNER Vesa (ENER); RYAN Alan (ENER)
REM2018 radon-in-water proficiency test: registration and sample information

Invitation letter Eurolook REM2018_ARES reg.pdf; Email text and mail addressess_REM2018 invitation for registration_ARES reg.pdf; Instructions for

registration REM2018 PT ARES reg.pdf

Importance: High

Dear colleagues,

Subject:

Attachments:

You receive this mail because you were either nominated by your national Euratom article 35/36 contacts or you expressed your interest in the proficiency test on radon massic activity measurement in water organized by JRC-Geel.

Please find our official invitation letter in the attachment which includes all the important information regarding registration of participants, planning of the PT, result submission and reporting.

From this moment on please do not advertise this proficiency test and we reserve the right to refuse new participation requests.

Should you have any questions just feel free to contact us.

Best regards,

Viktor JOBBAGYMikael HULTPetya MALOPT CoordinatorTeam LeaderLogistics Assistant



European Commission

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JRC-IRMM-REM-COMPARISONS@ec.europa.eu EU Science Hub: https://ec.europa.eu/jrc

REM Proficiency Tests: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests

The views expressed are purely those of the writer and may not in any circumstances be regarded as stating an official position of the European Commission

Please consider the environment before printing this email



Geel, 30/08/2018

Subject: Invitation for participation in the EC-JRC-REM 2018 proficiency test on ²²²Rn massic activity measurements in water

Dear colleagues,

It is our pleasure to invite your laboratory to participate in the EC-JRC-REM 2018 proficiency test on radon massic activity (Bq/kg) in water (reference: REM 2018 PT) as part of the European Commission's verification scheme under Article 35 of the Euratom Treaty. You can find further instructions and information on the proficiency test below. Please read them carefully.

Material information

The proficiency test material is natural origin spring water with low carbonate content. This natural water sample contains environmental level of radon (222 Rn) and other radionuclides* but the material can be transported freely and handled in the laboratory without any radiological restrictions. However, it can be expected that the radon massic activity of this sample is above the parametric level (100 Bq/kg) indicated in the EURATOM Drinking Water Directive (Council Directive 2013/51). The PT material will be directly filled fully without headspace into 1 L borosilicate glass bottles with a PTFE coated silicone seal screw cap.

The organizer can provide only one bottle per participant. Each bottle will contain approximately 1 L of the material, which is expected to be sufficient for the requested analyses. If the sampling bottle is damaged or significant sample loss observed during transport, please contact us, in that case an extra sample can be shipped.

Reference values and scoring

Reference values of the comparison samples will be established by using liquid scintillation counting and gamma-ray spectrometry measurement methods. The comparison will be evaluated with respect to these reference values using percentage difference from the reference value, z-score, zeta-score and optionally the E_n number. Therefore, a well-founded estimate of the uncertainty of the reported results is required from each participating laboratory.

Homogeneity of the PT test items will be established by liquid scintillation counting from 10 mL sub-samples. This sample volume can be considered as minimum sample intake for each measurement technique.

Registration and reporting

The reporting of laboratory results will be done in the JRC online reporting tool. Therefore, we kindly ask you to register your laboratory via the following web link (instructions attached): https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparis on=2081

Please be aware that the deadline for registration is **14 September 2018**.

You will have the opportunity to report results obtained by different methods (LSC, γ -ray spectrometry, emanometry, etc.).

The exact reference dates will be communicated individually by e-mail after sampling.

Participation costs

We kindly draw your attention to the fact that the participation in this PT is **free of charge**. All costs regarding the PT organization are covered by JRC-Geel. However, the sample analysis related costs are covered by the participants and not by the PT organizer. The participant is

^{*}no interference on the radon measurement from the $^{226}\mathrm{Ra}$ content was proven.

responsible for possible clearance or customs fees. By registering for this PT, you accept these aforementioned policies and give your consent to the PT organizer to use your measurement results for reporting and publication purposes.

Protocol for the PT

- 1. Participants are requested to follow their own routine measurement methods.
- 2. A brief questionnaire is a part of this exercise and participants are requested to answer all relevant questions regarding the procedures that they have used. https://ec.europa.eu/eusurvey/runner/REM 2018 PT radon-in-water

Note: If you save your Questionnaire as a draft, don't forget to send the generated link by email to yourself otherwise your data will be lost.

- 3. Tentative timing and deadlines:
- July/August 2018 Announcement to Article 35/36 Experts, DG ENER and expert labs,
- 3 September 2018: official invitation for registration to participants,
- 14 September 2018: on-line registration deadline,
- 18 October 2018: Organizer will start shipping PT material to participants,
- 14 November 2018: deadline for results reporting,
- December 2018: preliminary report distributed,
- April 2019: Final report,
- 2019 t.b.d.: workshop and training course organised for PT participants.

Data treatment and privacy

Each laboratory's results will be treated with confidentiality; identities will be kept anonymous and will not be disclosed to third parties. However, the results and performance of each nominated laboratory will be made available to its national representative(s) (the nominating authority) and to the relevant services of the European Commission at Directorate General for Energy.

In order to comply with the European regulation on the General Data Protection Regulation (GDPR), we would like to ask for your consent/approval to be able to list your organization and the name of the contact person in the final report. You can express your consent by sending us an e-mail with this statement: "Hereby, I [give / DO NOT give] (delete as necessary) my consent to have my name and the name of my organisation listed in the final report of the REM 2018 Radon-in-Water PT organised by JRC-Geel."

Complaints

In case of complaints please send a mail to our functional mailbox immediately. We will investigate your complaint and try to resolve it.

If you have further questions, please contact us at: JRC-GEE-REM-COMPARISONS@ec.europa.eu

We will keep you updated on the preparations. We are looking forward to your participation in this comparison.

Yours sincerely,

Viktor JOBBAGY

PT Coordinator

European Commission, DG Joint Research Centre

Directorate G - Nuclear Safety & Security

Unit G2 - Standards for Nuclear Safety, Security and Safeguards

Retieseweg 111, B-2440 Geel, Belgium

+32 (0)14 571 251

JRC-GEE-REM-COMPARISONS@ec.europa.eu

REM Proficiency Tests: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests

https://ec.europa.eu/jrc/en

Subject: Instruction for registration EC-JRC-REM 2018 proficiency test on 222Rn massic activity measurements in water

Important note: Only one registration per laboratory is required. Please avoid multiple registrations.

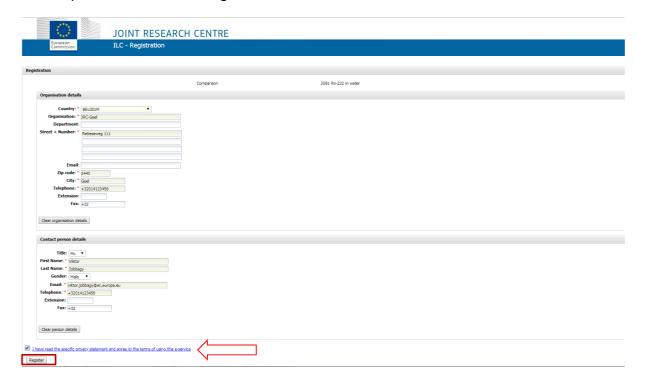
Weblink:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?s elComparison=2081

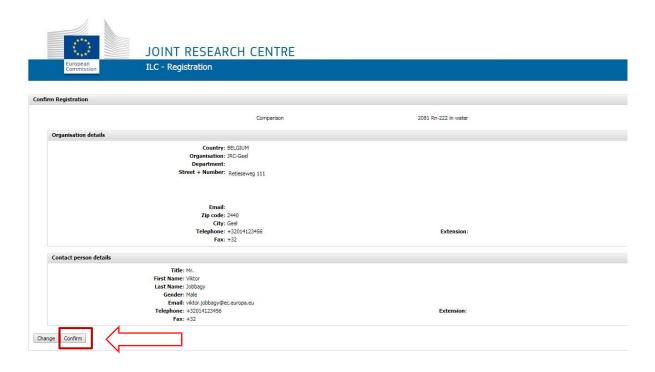
Step 1: Fill in the data, confirm that you have read the privacy statement and click "Register".

In the section "Organisation details", please provide the postal address of the laboratory which will perform the measurements (PO box is not acceptable). The sample will be sent to that address.

In the section "Contact person details", please provide the contact details of the person who will hold an overall responsibility for the measurements. This person will be our point of contact throughout the exercise.



Step 2: Check the data and if they are correct, click "Confirm" (if they are not correct, click "Change"). DO NOT CLOSE THE NEXT SCREEN!



Step 3: Download the registration form for proof of registration. Depending on your browser settings, the form will either open automatically or you should open it by clicking on the link "here".



After this step, your registration is complete. You **do not** need to print or sign the form.

Annex 3. Reporting instructions

Ref. Ares(2018)4956535 - 27/09/2018

How to submit your results for the reporting

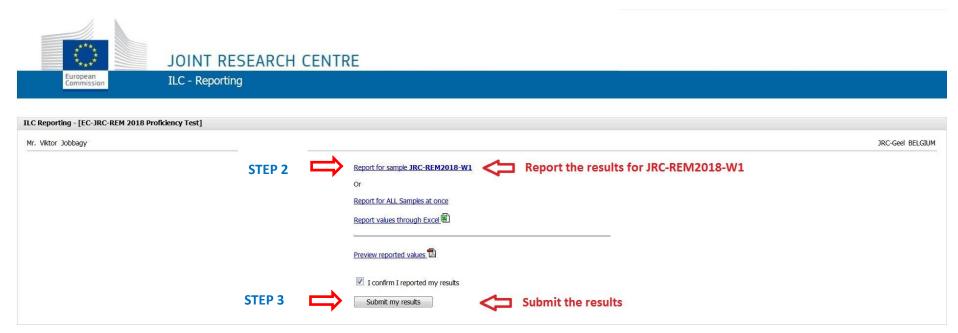
JRC-REM 2018 PT on radon-in-water

Step 1: Click on the link to the reporting module and insert your password key.

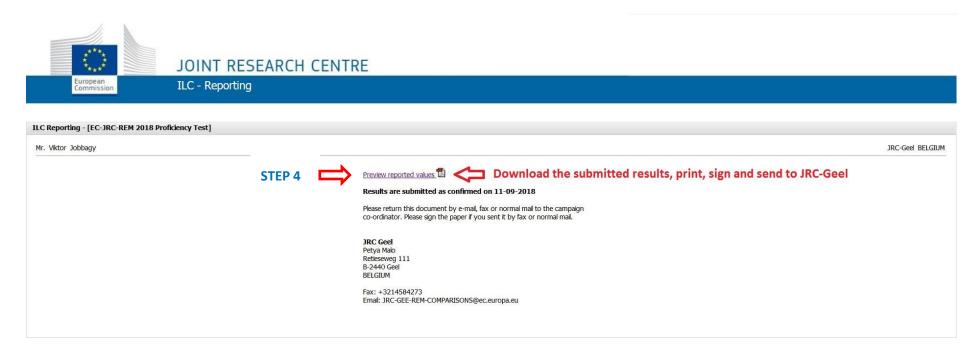
https://web.jrc.ec.europa.eu/ilcReportingWeb

Step 2. Report the results for JRC-REM2018-W1, validate and go back to the main page.

Step 3. Submit your results.



Step 4. Download the form with the submitted values and questionnaire, <u>sign it</u> and send it by <u>e-mail</u> (<u>JRC-GEE-REM-COMPARISONS@ec.europa.eu</u>) to finalise your results submission.



Annex 4. Accompanying letter



Geel, 27/09/2018 JRC.G.2/VJ/Ares(2018)4956535

Subject: Sample accompanying letter for the EC-JRC-REM 2018 proficiency test on ²²²Rn massic activity measurements in water

Dear colleague,

You will find information on the REM 2018 proficiency test material, requested measurement protocol and reporting in this letter.

Material information

The proficiency test material is natural origin spring water with low carbonate content. This natural water sample contains environmental level of radon (222 Rn) and probably other radionuclides but the material can be transported freely and handled in the laboratory without any radiological restrictions. However, it can be expected that the radon massic activity of this sample is above the parametric level (100 Bq/kg) indicated in the EURATOM Drinking Water Directive (Council Directive $^{2013/51}$).

The PT material was directly filled fully without headspace into 1 L borosilicate glass bottles with a screw cap with PTFE coated silicone seal. Each bottle contains approximately 1 L of the material, which is expected to be sufficient for the requested analyses. We can provide limited number of extra samples in case of damaged samples only.

The package you received contains in addition to this letter:

- PT material sealed in a plastic bag and bubble foiled envelope,
- a thermo-button (temperature logger) in a plastic bag and
- the *Sample receipt* form.

<u>Upon arrival of this package</u>, please check if the test item is undamaged after transport and send us the *Sample receipt form* by e-mail and the *thermo-button* in an envelope by post to the indicated address immediately.

Store your samples in a dark place maximum at room temperature (preferably below) but well above 0 °C (zero Celsius grade). By cooling samples below the sampling temperature, bubbles may form due to changes in the density of water which results in volume changes too.

Before the analysis, it is recommended to store the sample bottle at room temperature until it reaches thermal equilibrium with its environment. Participants are requested to follow their own routine measurement methods.

Timing

- 1. Material distribution of JRC-REM2018-W1 sample: 18-26 October 2018
- 2. Deadline for reporting results: 14 November 2018
- 3. Preliminary information on the individual laboratory performance will be sent by e-mail in December 2018. The final report of this comparison exercise is foreseen to be available by 30 April 2018.

Reporting

The reporting of laboratory results will be done in the JRC online reporting tool. Therefore, we kindly ask you to submit your results via the following weblink using the password key provided:

https://web.jrc.ec.europa.eu/ilcReportingWeb

Password key: will be given to each participant in personalized e-mails.

You will have the opportunity to submit results obtained by different methods (LSC, γ -ray spectrometry, emanometry, etc.). You are requested to report:

- the measurement technique you used,
- one measurement result/mean value per technique (massic activity in Bq/kg),
- associated expanded uncertainty with coverage factor of k = 1.

The exact reference dates will be communicated individually by e-mail after sampling.

For your calculations, we recommend to use the data provided by the Decay Data Evaluation Project (DDEP) at http://www.nucleide.org/DDEP_WG/DDEPdata.htm

Check your calculations and report before submitting your results. Please note that **we cannot accept modifications after the reporting deadline**.

A brief questionnaire is a part of this exercise. Therefore, you are kindly requested to **fill in the questionnaire** and answer all relevant questions regarding the procedures that you have used. In addition you can give feedback and comments about this PT.

https://ec.europa.eu/eusurvey/runner/REM_2018_PT_radon-in-water

If you save your Questionnaire as a draft, do not forget to send the generated link by email to yourself otherwise your data will be lost.

If you have further questions, please contact us via the following functional mailbox: JRC-GEE-REM-COMPARISONS@ec.europa.eu

Your participation in this proficiency test is highly appreciated.

Best regards,

Viktor JOBBAGY

PT Coordinator

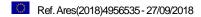
European Commission
DG Joint Research Centre
Directorate G - Nuclear Safety & Security
Unit G2 - Standards for Nuclear Safety, Security and Safeguards
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32 (0)14 571 251

e-mail: JRC-GEE-REM-COMPARISONS@ec.europa.eu

REM Proficiency Tests: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests

https://ec.europa.eu/jrc/en

Annex 5. Sample receipt form





EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate G - Nuclear Safety and Security
Standards for Nuclear Safety, Security and Safeguards

Geel, 27/09/2018

Subject: Sample receipt form
Reference: JRC-REM 2018 radon-in-water PT
Date and time of package arrival:
- (day/month/year):
(Coordinated Universal Time-UTC; hh:mm):
Sample code, bottle number:
Please return this form immediately after the receipt of your samples to confirm that the package arrived. If samples are damaged/missing, mention it under "Remarks, other comments" section and contact us immediately.
Broken containers (indicate sample code):
Leaking containers (indicate sample code):
Remarks, other comments:
Contact details
Organization:
Contact person:
E-mail address:
Thank you for returning this form by e-mail to: Viktor Jobbágy, REM 2018 PT coordinator

E-mail: <u>JRC-GEE-REM-COMPARISONS@ec.europa.eu</u>

Annex 6. List of participating laboratories

AUSTRIA

AGES GmbH Radiation Protection/Radiochem Spargelfeldstraße 191 1220 Vienna

AUSTRIA

AGES - Austrian Agency for Health and Food Safety Radon and Radioecology Wieningerstraße 8 4020 Linz

BELGIUM

Institute for radioelements (IRE ELIT) Radioactivity Measurements Lab 1 Avenue de l'Espérance 6220 Fleurus

BELGIUM

FANC
Surveillance of the territory
Ravensteinstraat 36
1000 Brussels

BELGIUM

SCK•CEN Low radioactivity measurements Boeretang 200 2400 Mol

BOSNIA - HERZEGOVINA

Institute for Public Health of Federation of Bosnia and Herzegovina Radiation Protection Center
M. Tita 9
71000 Sarajevo

BULGARIA

National Center of Radiobiology and Radiation Protection Public Exposure Monitoring Lab 3, Georgi Sofiiski Blvd. 1606 Sofia

BULGARIA

Sofia University "St. Kliment Ohridski" Atomic Physics 5 James Bouchier Blvd Building B10 1164 Sofia

BULGARIA

DIAL Ltd Mina Buhovo str, 111 1830 Buhovo

BULGARIA

Executive Environment Agency Radioactivity Measurement Labo 136, Tzar Boris III blvd. 1618 Sofia

BULGARIA

Institute of Soil Science, Agrotechnologies and Plant Protection "N. Poushkarov" Isotope laboratory
Shousse Bankya Str. 7
1080 Sofia

CROATIA

J. J. Strossmayer University of Osijek Department of Physics Trg Ljudevita Gaja 6 31000 Osijek

CROATIA

Ruđer Bošković Institute Bijenička cesta 54 10000 Zagreb

CYPRUS

University of Cyprus Chemistry Leoforos Panepistimiou 1 2109 Nicosia

CYPRUS

Radiation Inspection and Control Service Department of Labour Inspection Apellis str. 12 1080 Nicosia

CZECH REPUBLIC

National Radiation Protection Institute (Státní ústav radiační ochrany)

Radiochemie

Syllabova 21

70300 Ostrava 3

CZECH REPUBLIC

National Radiation Protection Institute

Branch in Hradec Kralove

Pileticka 57

50003 Hradec Kralove

ESTONIA

Environmental Board

Radiation Safety Department

Kopli 76

10416 Tallinn

FINLAND

Radiation and Nuclear Safety Authority (STUK)

Laippatie 4

00880 Helsinki

FRANCE

CEA/LIST

LNHB

CE-Saclay, bâtiment 602

91191 Gif sur Yvette cedex

FRANCE

IRSN

PSE-ENV / SAME

31 rue de l'Ecluse

Bâtiment C4, BP 40035

78116 Le Vesinet

FRANCE

PearL

20 Rue Atlantis

87068 Limoges

GERMANY

Federal Office for Radiation Protection

Rad. Protection & Environment

Koepenicker Allee 120-130

10318 Berlin

GERMANY

CVUA Freiburg 12.1 Radioactivity Lab Bissierstraße 5 79114 Freiburg

GERMANY

Federal Institute of Hydrology G4 Am Mainzer Tor 1 56068 Koblenz

GREECE

Greek Atomic Energy Commission Environmental Radioactivity Patriarchou Grigoriou & Neapol 15310 Attiki

GREECE

National Technical University of Athens Nuclear Engineering Department Heroon Polytechneiou 9 Zografos Campus, Mechanical Engineering Labs, Building "K" 15780 Athens

HUNGARY

RadiÖko Kft. Wartha Vince 1/2 8200 Veszprém

HUNGARY

University of Pannonia Institute of Radiochemistry and Radioecology Wartha V. u. 1. (N épület) 8200 Veszprém

HUNGARY

National Public Health Institute Dept. of Radobiology & Radiohygiene Anna u. 5. 1221 Budapest

HUNGARY

MECSEKÉRC Radiometriai laboratórium Akna utca 2. 7673 Kővágószőlős

HUNGARY

National Food Chain Safety Office Radioanalytical Reference Lab. Fogoly utca 13-15. 1182 Budapest

HUNGARY

Hydrosys Labor Kft. Botond u. 72. 1038 Budapest

ICELAND

Icelandic Radiation Safety Authority Raudararstigur 10 105 Reykjavik

IRELAND

Environmental Protection Agency ORM EPA, Block 3 Clonskeagh Square, Clonskeagh Road D14 H424 Dublin

ITALY

ARPACAL Dipartimento Reggio Calabria via Troncovito snc 89135 Reggio Calabria

ITALY

L.B. Servizi per le Aziende srl via Giuseppe Allievo, 81 00135 Rome

ITALY

Istituto Zooprofilattico Sperimentale della Puglia e della Basilicata Chimica - CRN-Radioattività Via Manfredonia 20 Via Del Feudo D'Ascoli 71121 Foggia

ITALY

Istituto Superiore di Sanità CNPRFC Via del Castro Laurenziano 25 00161 Roma

ITALY

ARPA Lazio Viterbo Via Montezebio 17 01100 Viterbo

ITALY

ARPA Valle d'Aosta Loc Grande Charrière 44 11020 Saint Christophe

ITALY

National Research Council (CNR) / Sapienza - University of Rome 7, P.le Aldo Moro, 00185 Rome 14, Via A. Scarpa, 00161 Rome 00100 Rome

ITALY

ARPAV Dip. Reg. Laboratori - CRR Via A. Dominutti 8 37135 Verona

ITALY

Arpa Sicilia S.T. di Catania via Varese 45 95123 Catania

ITALY

ARPA Piemonte Via Jervis, 30 10015 Ivrea

ITALY

MI.AM SRL via Alessandro Bolzoni 30 29122 Piacenza

ITALY

ARPA Marche - Dipartimento Prov.le di Ancona U.O. Radioattività Ambientale via Colombo 106 60127 Ancona

ITALY

ARPACAL
Catanzaro - Physics lab
Via Lungomare - loc. Mosca
Giovino - Catanzaro Lido
88100 Catanzaro

ITALY

APPA TN - Local Environmental Protection Agency Settore Laboratorio via Lidorno, 1 38123 Trento

ITALY

Agenzia Provinciale per l'Ambiente Bolzano Laboratorio di chimica fisica Via Amba Alagi 5 39100 Bolzano

ITALY

ARPA Puglia U.O.S. Polo R. I. Via Oberdan 18/E 70126 Bari

ITALY

Agenzia Regionale per la Tutela Ambientale ARTA Abruzzo Laboratorio Fisica ambientale Viale G. Marconi, 51 65126 Pescara

ITALY

ARPA Umbria Servizio Radiazioni Ionizzanti Via Pievaiola, 207/B-3 San Sisto 06132 Perugia

ITALY

ARPAE Emilia Romagna CTR Radioattività ambientale Via XXI Aprile, 48 29121 Piacenza

ITALY

Università "Roma Tre" Dipartimento di Scienze Largo San Leonardo Murialdo 1 00146 Roma

ITALY

ARPA Sicilia Palermo via Naiirobi N. 8 90129 Palermo

LATVIA

Latvian Environment, Geology and Meteorology Centre Laboratory Maskavas street 165 1019 Riga

LITHUANIA

Radiation Protection Centre of expertise and exposure monitoring Kalvariju 153 08221 Vilnius

LUXEMBOURG

Ministry of Health Radioprotection Villa Louvigny, Allée Marconi 2120 Luxembourg

NETHERLANDS

Rijkswaterstaat CIV Laboratory Zuiderwagenplein 2 8224 AD Lelystad

NORWAY

The Norwegian Radiation Protection Authority Grini næringspark 13 1361 Østerås

POLAND

Silesian Centre for Environmental Radioactivity plac Gwarkow 1 40-166 Katowice

POLAND

AGH University of Science and Technology Geophysics al. Mickiewicza 30 30-059 Krakow

POLAND

Technical University of Lodz Institute of Applied Radiation Wroblewskiego 15 90-924 Lodz

POLAND

Institute of Nuclear Physics PAN LER Lab. of Radiometric Expert Radzikowskiego 152 31-342 Kraków

POLAND

National Centre for Nuclear Research LPD A. Sołtana 7 05-400 Otwock

POLAND

Central Laboratory for Radiological Protection Konwaliowa st. 7 03-194 Warsaw

PORTUGAL

Instituto Superior Técnico/Laboratório de Proteção e Segurança Radiológica Estrada Nacional 10 (km 139,7) 2695-066 Bobadela LRS

ROMANIA

Cosma Constantin Radon Laboratory Str. Fintinele nr. 30 400327 Cluj-Napoca

SERBIA

University of Novi Sad, Faculty of Sciences Department of Physics Trg Dositeja Obraadovica 4 21000 Novi Sad

SERBIA

Vinca Institute of Nuclear Sciences Radiation and Envir.Protection Mike Petrovica Alasa 12-14 11000 Belgrade

SLOVAKIA

Regional Public Health Organisation Radiation Protection Ipelska 1 040 11 Kosice

SLOVAKIA

Regional Public Health Authority in Banska Bystrica Radiation Protection Cesta k nemocnici 1 975 56 Banska Bystrica

SLOVAKIA

Public Health Authority of the Slovak Republic Radiation Protection Trnavská cesta 52 82645 Bratislava

SLOVAKIA

Water Research Institute Radiochemistry Nábr. arm. gen. L Svobodu 5 81249 Bratislava

SLOVENIA

ZVD Zavod za Varstvo pri Delu D. O. O. CFM Chengdujska Cesta 25 1260 Ljubljana Polje

SLOVENIA

Jožef Stefan Institute Environmental Sciences Jamova cesta 39 1000 Ljubljana

SPAIN

Institut Tecniques Energetiques Universitat Politècnica de Catalunya Diagonal 647 08028 Barcelona

SPAIN

University of Extremadura Lab Environmental Radioactivit LARUEX-Faculty of Veterinary Avda. De La Universidad SN 10003 Caceres

SPAIN

University of Granada Inorganic Chemistry Radiochemistry Environmental Laboratory Faculty of Sciences Av. Fuentenueva, s/n 18071 Granada

SPAIN

CIEMAT

Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas Medioambiente Avenida de la Complutense, 40 28040 Madrid

SPAIN

Universidad del Pais Vasco Ing. Nuclear y Mec. Fluidos Escuela Ingenieria Bilbao Pza Ingeniero Torres Quevedo 1 48013 Bilbao

SPAIN

Universitat Politecnica de Valencia Lab. Radiactividad Ambiental Camino de Vera, S/n 46022 Valencia

SPAIN

University of Zaragoza Faculty of Sciences Theoretical Physics. Nuclear A Pedro Cerbuna 12 50009 Zaragoza

SPAIN

CEDEX Área Aplicaciones Isotópicas Alfonso XII, 3-5 (Edif. CETA) 28014 Madrid

SPAIN

Universitat Rovira i Virgili - Unitat de Radioquimica Ambiental i Sanitaria URAIS
Consorci d'Aigues de Tarragona
ETAP L'Ampolla / URAIS, Ctra.N340, Km. 1094
43895 L'Ampolla

SPAIN

LARUC (University of Cantabria)
Faculty of Medicine
Cardenal Herrera Oria s/n
39011 Santander

SPAIN

Universidad de Valencia Laboratorio de Radiactividad Ambiental Doctor Moliner, 50 Edificio de Investigación, Sótano -2 46100 Burjassot (Valencia)

SPAIN

ETSI Caminos, Canales y Puertos. UPM Laboratorio Nuclear C/Profersor Aranguren 3 28040 Madrid

SPAIN

Universitat de Barcelona Lab. Radiologia Ambiental Martí i Franqués 1-11 Planta 3 08028 Barcelona

SPAIN

University of Extremadura Physics Physics Av. w/n 06006 Badajoz

SWEDEN

SYNLAB Analytics and Services Olaus Magnus Väg 27 C 58110 Linköping

SWEDEN

Uppsala Vatten och Avfall AB Vattenlaboratoriet Stallängsgatan 3 75318 Uppsala

SWEDEN

Eurofins Environment Testing Sweden AB Sjöhagsgatan 3 Port 1 53140 Lidköping

SWEDEN

Radonova Laboratories AB Rapsgatan 25 75450 Uppsala

SWITZERLAND

Federal Office of Public Health Str/URA Schwarzenburgstrasse 157 3003 Bern

UKRAINE

SE "The O.M.Marzeev Institute of Public Health NAMSU" 50, Popudrenko str. 02094 Kyiv

UKRAINE

UHMI Environmental Rad.Monitoring av. Nauki, 37 03028 Kyiv

UK

Public Health England RHED CRCE Glasgow 155 Hardgate Road G51 4LS Glasgow

UK

SOCOTEC UK Limited Nuclear Chemistry Unit 12, Moorbrook Southmead Industrial Park OX11 7HP Didcot

UK

South West Water Ltd DWS Science and Water Quality Bridge Road Countess Wear, Exeter, Devon EX2 7AA Exeter

Annex 7. Questionnaire

QUESTIONNAIRE FOR PARTICIPANTS: REM 2018 Radon-inwater PT;

radon massic activity measurement in drinking water

Fields marked with * are mandatory.
Laboratory
Contact details
*Name of the contact person
*Name of organisation/laboratory
*Country
*E-mail
the state of any laborate of
*What is the type of your laboratory? Research and development
Measurements of radioactivity in the environment
Monitoring of nuclear facilities
Measurements for fissile material control or safeguards
Private commercial company
Other (please specify below)
Please specify

Ves, accredited No, not accredited No Yes No No No No No No No N	■ No, not accredited *Is your laboratory working according to a quality management system? ○ Yes ○ No *Select one or more □ ISO 9000 series □ EN 45000 series □ ISO/IEC 17025 □ Other (please specify below)	
*Is your laboratory working according to a quality management system? Yes No *Select one or more Is 05 9000 series EN 45000 series Is 04 5000 series Is 05 9000 series Other (please specify below) *Please specify *Does your National Standardisation Body involve you to comment on ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your organization (in years)? *Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *No *How many measurements of this type does your laboratory perform per year? *How many measurements of this type does your laboratory perform per year? *How ISO/European standards? *How many measurements of this type do	*Is your laboratory working according to a quality management system? Yes No Select one or more ISO 9000 series EN 45000 series ISO/IEC 17025 Other (please specify below)	
*Is your laboratory working according to a quality management system? Yes No *Select one or more Is 05 9000 series EN 45000 series Is 04 5000 series Is 05 9000 series Other (please specify below) *Please specify *Does your National Standardisation Body involve you to comment on ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *Would you be interested to collaborate more closely with your organization (in years)? *Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *Would you be interested to collaborate more closely with your National Standardisation Body in order to improve the ISO/European standards? Yes No *No *No *How many measurements of this type does your laboratory perform per year? *How many measurements of this type does your laboratory perform per year? *How ISO/European standards? *How many measurements of this type do	*Is your laboratory working according to a quality management system? Yes No Select one or more ISO 9000 series EN 45000 series ISO/IEC 17025 Other (please specify below)	
Select one or more Select one or more Select one or more Select one or more Six 9000 series No 45000 series No 150 9000 serie	 Yes No Select one or more ☐ ISO 9000 series ☐ EN 45000 series ☐ ISO/IEC 17025 ☐ Other (please specify below) 	
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Technical details

	d you perform the test in compliance with the following standards? (Select one or more)
-	ISO 13164-2 Gamma-ray spectrometry
	ISO 13164-3 Emanometry
	ISO 13164-4 Liquid scintillation counting
\equiv	Other:
f oth	er, please specify:
2. WI	nich detection technique did you use for this PT?
	Gamma-ray spectrometry
	Scintillation cell (e.g. silver-activated zinc sulfide ZnS(Ag))
	Air ionisation
	Semiconductor (alpha-detection)
	Liquid scintillation counting methods (LSC)
	Other
oth	er, please specify:
	se specify the type of measurement instrument you used (e.g. Quantulus, TriCarb, HPGe, Lucas cell, Guard, Rad7, etc.)
W	nich test sample preparation method did vou use?
-	nich test sample preparation method did you use?
	Direct measurement in the sampling container
-	Direct measurement in the sampling container Degassing technique
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Other			
□ Other			
If other, plea	ise specify:		
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software, if ap			
500 charact	er(s) maximum		
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Feedback, comments

Please give your brief feedback on the PT exercise (remarks, improvements or positive feedback):
500 character(s) maximum
Overall satisfaction score. How satisfied were you with the PT? (1: unsatisfied, 10: very satisfied)
Only values between 1 and 10 are allowed

Annex 8. Feedbacks and comments from the questionnaire

Please give your brief feedback on the PT exercise (remarks, improvements or positive feedback):

- 1. We are satisfied with this PT. Sample was received in suitable container for different type of analysis.
- 2. Just a little problem on web.jrc.ec.europa.eu/ilcReportingWeb but a rapid help to solve it.
- 3. Improvements: check the validity of the calibration used and the routine working conditions for this assay.
- 4. Very well organised PT exercise clear instructions and samples arrived really quickly and in good condition.
- 5. very good
- 6. Good exercise. Maybe some more samples with different activities
- 7. as already anticipated in the sample receipt form, I remark that one air bubble was present in the bottle
- 8. We were pleased with the organization of this PT!
- 9. It is very good to have the opportunity to participate in PT. It helps to check the procedure, calibration, reveals problems The important issues are as follows. The fast handling of the data and quick release of the final report. The another problem is the time gap between sample preparation and delivery. 3-4 days is OK, it's roughly the 1 half life of Rn-222. Longer time would be difficult to accept, as the uncertainty of the result would grow significantly.
- 10. The questionnaire does not seem to be too functional, having to report information relating to several measurement techniques.
- 11. The organization was very good and the sample we received has correct. We only have a small problem for the reporting but it was a mistake of our side. To be perfect, It should be nice to received such kind of PT sample in the beginning of the week
- 12. Improvements:

The period between the preliminary report and the final report seems rather long. An activity around the parametric level (100Bq/l) would be appreciated. Positiv: good documentation

- 13. We have had some difficulties with the web page while sending the results and also with this survey web page.
- 14. Plese note that the uncertainty budget is for Gamma spectrometry only.
- 15. It has been a very interesting and educating PT exercise. Especially for a lab that started doing Radon measurements 3 years ago regularly and not very frequently.
- 16. We would like to test our procedure with a lower concentration of about 10 Bq/L, that is the Minimum Concentration required by the Italian law.
- 17. This initiative is very good and helpful for quality assurance of our results. We hope that such PT will be organized also in future.

- 18. We are just developing the radon measurements procedure now. It is not a routine procedure in our laboratory. That is why we cannot answer (but we had to) for all questions in chapter Experience. Point Technical Details 9 was filled for LSC

 technique.
 - For AlphaGuard: uncertainty given by detecor: 18% relative uncertainty of sample volume: 2%
- 19. Very clear, detailed and helpful instructions received by e-mails; precise information regarding reference date, very well organised transport of water samples, excellent protection of water samples for transport, user friendly online reporting of results.
- 20. Regarding the question about Experience: We have a wide varity of radon activity in our samples from 1-10000 Bq/l. 1-1000 Bq/l is common results for radon activity in our samples.
- 21. We are satisfied with the PT organization. Just as a remark, there was a delay in the communication of the reference date of the sample (5 days after sample reception). Althought, we have the results of the analysis we cannot performe the quantification until the reception of this information.
- 22. Everything was perfectly organized.
- 23. Sample came in well packaged and arrived in the laboratory without the delay. Instructions were clear. Unfortunately due to the instrument breakdown lab was unable to perform analysis within 48 hours from sample arrival as per routine samples.
- 24. Normal geometry is a Kilner Jar which we would supply to customers for them to fill with no air bubbles, in this case in order to fill the geometry we had to dilute the sample which could have resulted in losses. The original sample was counted as received and found to be consistent. In the future it would be useful if we could have the option to send our standard geometry to be filled.
- 25. The organization of the PT test was excellent. Perhaps the reporting web-page should contain a field to indicate or enter the reference date/time.
- 26. It would be interesting to have the chance to work with a bigger sample volume (e.g. 2 L). We liked the way that sample was prepared, specially regarding the control of the temperature during the sample journey. A positive aspect to remark is the speed in the delivery of the samples and the continuous feedback provided by e-mail.
- 27. Sample delivery time was very quick! testing possible on day of arrival.
- 28. Ok! Just put the questionnaire in the same page of results
- 29. This PT was well-organized, precise, we received the sample shortly. I suggest, send the sample at the beginning of the week (there was a four-day holiday in Hungary).
- 30. A lot of information to submit. Didn't understand that I missed to fill in this form.
- 31. We are satisfied that JRC organized this interlaboratory comparison. The PT is very useful for our laboratory with regard to the QA system implemented in the laboratory. This PT will be highly esteemed to us in the pre-accreditation next year.

- 32. Additionally to p.4 We use both water miscible and organic scintillators Add to experiences: Radon range in water between 0.5 Bg/L to 3000 Bq/L
- 33. To take 12 mg of the liquid scintillation for Rn in water measurement, then take 10 mg of water and mix with liquid scintillation and measure by the alpha/beta liquid scintillation counter. For the elimination of the background from the surrounding the counter, cosmic ray, one sample prepared from distilled water. The distilled water sample was measured with the series of the studied water. The prepared samples were measured every day. The measurements should last at least one week.
- 34. It was very good that the samples were in standardized bottles. For this kind of bottles, we have devised a method of flawless transfer to the emanometry system.
- 35. It would have been better If we had received the ref. time at the same time as the sample. We thought that we should use the time that the sample was poured into our sample container. Since we normally don't do any manual calculations, this meant that it took a lot of time for us figuring out how to do the calculations. Also, we received a lot of information about the PT, but we would have liked more information about how and when to perform the analysis.
- 36. The PT exercise has been well programmed in long and form and the information provided clear and sufficient.
- 37. A very well organized PT
- 38. Interesting exercise, but I do not understand why there is not a single reference date.
- 39. Thank you for your kindliness with filling our sample bottles!
- 40. A well organised PT with clear and comprehensive instructions and adhered to a good timetable.
- 41. Usually in our TEST REPORTS the Cover factor (k) is: 2 not 1
- 42. Excellent. Very well organised and we look forward to participating in future JRC PTs.
- 43. Very well organized and, most of all, very usefull!
- 44. Appreciate this test. Can be difficult to fill up customer samples for performance test since customer samples can't be planned in time and concentration. It is an extra uncertainty in a possible loss of radon gas when water is refilled on the measurement bottles.
- 45. It was very helpful for us, that all important information was clearly specified in the text of messages.

Annex 9. Communication on preliminary results

From: JRC GEE REM COMPARISONS

Ref. Ares(2018)6449519 - 14/12/2018

Sent: Friday, December 14, 2018 12:07 PM

Cc: HULT Mikael (JRC-GEEL); MALO Petya (JRC-GEEL); PLOMPEN Arjan (JRC-GEEL); TANNER Vesa

(ENER); RYAN Alan (ENER); DE CORT Marc (JRC-ISPRA); JOBBAGY Viktor (JRC-GEEL) **Subject:** REM 2018 radon-in-water PT: Preliminary report (Ares(2018)6449519)

Importance: High

Dear participants,

We are sending the preliminary report of the REM 2018 radon-in-water PT focusing on the laboratory results and their scores.

Please note that the evaluation is still ongoing. Therefore, this document is **for information purposes only** and cannot be regarded as official final evaluation of the pilot-PT.

Your lab code will be sent to you in a separate e-mail later today. However, from your reported results you are able to identify your organization.

We would like to ask you to review this document and check your results before 1 February 2019. Should you notice mistakes, feel free to contact us immediately and then we will review our documents.

In order to comply with the European regulation on the General Data Protection Regulation (GDPR), we decided to list only the name of your organization and no person's names in the final report. Concerning the confidentiality of your results, only the lab codes assigned by us are used in the preliminary report. The correspondence between the laboratories and the assigned lab codes is not revealed.

We remind you that the <u>final</u> results and performance of each <u>nominated</u> laboratory will be made available to its national representative(s) (the nominating authority) and to the relevant services of the European Commission at Directorate General for Energy.

We are planning to make the final report available as soon as possible (before 30 April 2019).

We would like to remind you that a workshop and training courses will be organized at JRC-Geel between 26-29 March 2019. You will receive further information on the registration and travel/hotel arrangements soon.

You can also consult the planning of the forthcoming REM proficiency tests on this website: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests.

For your information, due to holidays at JRC we will have limited availabilities until 7 January 2019. For this reason, if you have any enquiries, please add Viktor Jobbagy (<u>viktor.jobbagy@ec.europa.eu</u>) in Cc of your e-mail.

We would like to express our gratitude to everyone who participated in this REM 2018 PT and for your kind collaboration. Your contribution was essential for the smooth organisation of the PT. We hope that we can welcome you in the coming JRC proficiency tests.

Best regards,

Viktor JOBBAGY Mikael HULT Petya MALO

PT Coordinator Team Leader Logistics Assistant

European Commission

Joint Research Centre (JRC), Standards for Nuclear Safety, Security & Safeguards

Retieseweg 111, B-2440 Geel, Belgium, +32 14 571 251

JRC-IRMM-REM-COMPARISONS@ec.europa.eu

EU Science Hub: https://ec.europa.eu/jrc

REM Proficiency Tests: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests

The views expressed are purely those of the writer and may not in any circumstances be regarded as stating an official position of the European Commission

Annex 10. Summary table on participants` scores

Table 11. Participants` results and their scores as percentage difference (D%), z-score, ζ (zeta)-score for JRC-W1 sample. ²²²Rn massic activity values (A_{ref}) with their combined standard uncertainties (u_{ref}) with a coverage factor k=1.

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
1	321	22	0.9	0.05	0.11	Liquid-scint. counting
1	334	11	5.0	0.25	0.82	Direct gamma-spec.
2	270	17	-15.1	-0.75	-2.06	Liquid-scint. counting
2	272	10	-14.5	-0.72	-2.44	Direct gamma-spec.
3	287	10	-9.7	-0.49	-1.64	Liquid-scint. counting
3	295	15	-7.2	-0.36	-1.05	Direct gamma-spec.
4	273.4	9.1	-14.0	-0.70	-2.42	Liquid-scint. counting
5	342.7	17.8	7.8	0.39	1.03	AlphaGUARD + AquaKIT
6	327	9	2.8	0.14	0.49	Liquid-scint. counting
7	161.3	16.8	-49.3	-2.46	-6.75	Emanometry
8	289	7.54	-9.1	-0.46	-1.64	Liquid-scint. counting
9	282.3	14.1	-11.2	-0.56	-1.67	Emanometry
10	268	13.5	-15.7	-0.79	-2.39	Direct gamma-spec.
11	2757.41	523.905	767.1	38.36	4.65	Liquid-scint. counting
12	309	37.1	-2.8	-0.14	-0.22	Electret Ion Chamber (EIC) technology
12	322	13.1	1.3	0.06	0.19	Direct gamma-spec.
13	310	10	-2.5	-0.13	-0.42	Liquid-scint. counting
14	297.44	21.61	-6.5	-0.32	-0.76	Emanometry

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
15	308.9	6.2	-2.9	-0.14	-0.53	Liquid-scint. counting
16	281	18	-11.6	-0.58	-1.54	Gamma spectrometry with NaI(Tl), water transfered from glass to plastic bottles
16	298	20	-6.3	-0.31	-0.78	Gamma spectrometry with NaI(TI), own bottles
17	299.7	26.8	-5.8	-0.29	-0.59	Liquid-scint. counting
18	274	81	-13.8	-0.69	-0.53	Radon monitor-Rad7
19	342	17	7.5	0.38	1.03	Liquid-scint. counting
20	278.56	17.01	-12.4	-0.62	-1.69	Liquid-scint. counting
21	295	29.5	-7.2	-0.36	-0.69	Liquid-scint. counting
21	300	30	-5.7	-0.28	-0.53	Liquid-scint. counting
21	305	3.2	-4.1	-0.20	-0.80	Emanometry
22	352	14	10.7	0.53	1.60	Liquid-scint. counting
23	221.11	33.82	-30.5	-1.52	-2.59	Liquid-scint. counting
23	221.87	27.94	-30.2	-1.51	-2.99	Emanometry
24	295	30	-7.2	-0.36	-0.68	alphaguard measurement
24	302	40	-5.0	-0.25	-0.37	Direct gamma-spec.
24	304	20	-4.4	-0.22	-0.55	Liquid-scint. counting
25	335	23	5.3	0.27	0.61	Emanometry
26	312.5	7.5	-1.7	-0.09	-0.31	Direct gamma-spec.
26	329	30	3.5	0.17	0.32	Emanometry

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
27	270	12	-15.1	-0.75	-2.40	LSC in organic LS scintilation coctail (3 sample 3 measurements each average)
27	284	10	-10.7	-0.53	-1.80	LSC in water miscible LS scintilation coctail (3 sample 3 measurements each average)
28	281.13	12.03	-11.6	-0.58	-1.84	Liquid-scint. counting
29	241.9	13.6	-23.9	-1.20	-3.62	Alfa counting, RAD7 + BigBottle System (soda bottles)
30	303.6	3.5	-4.5	-0.23	-0.88	Liquid-scint. counting
31	330.6	12	4.0	0.20	0.63	Liquid-scint. counting
32	265	20	-16.7	-0.83	-2.07	Direct gamma-spec.
33	305	8.5	-4.1	-0.20	-0.72	Liquid-scint. counting
34	325	16	2.2	0.11	0.31	Emanometry
35	327	4	2.8	0.14	0.55	Liquid-scint. counting
36	313	11	-1.6	-0.08	-0.26	Direct gamma-spec.
37	309	16	-2.8	-0.14	-0.40	Liquid-scint. counting
37	313	20	-1.6	-0.08	-0.20	Emanometry
37	314	12	-1.3	-0.06	-0.20	Direct gamma-spec.
38	186.4	2.55	-41.4	-2.07	-8.12	Direct gamma-spec.
39	274.9	20.5	-13.6	-0.68	-1.66	Liquid-scint. counting

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
40	375	26.5	17.9	0.90	1.84	Liquid-scint. counting
41	294.9	1.2	-7.3	-0.36	-1.44	Liquid-scint. counting
41	298.9	3.7	-6.0	-0.30	-1.16	Liquid-scint. counting
42	330	22	3.8	0.19	0.44	Emanometry
42	336	22	5.7	0.28	0.66	Emanometry
43	331	24.8	4.1	0.20	0.44	Liquid-scint. counting
44	290	18.5	-8.8	-0.44	-1.14	Liquid-scint. counting
45	298	21.5	-6.3	-0.31	-0.75	Emanometry
46	241.2	17.9	-24.2	-1.21	-3.20	Liquid-scint. counting
47	337	20	6.0	0.30	0.74	Liquid-scint. counting
48	263	50	-17.3	-0.86	-1.05	Emanometry
48	355.8	24	11.9	0.59	1.31	Liquid-scint. counting
49	190	7	-40.3	-2.01	-7.33	Liquid-scint. counting
49	203	24	-36.2	-1.81	-3.99	Emanometry
50	333	12.5	4.7	0.24	0.74	Liquid-scint. counting
51	319	36	0.3	0.02	0.03	Liquid-scint. counting
52	313	21	-1.6	-0.08	-0.19	RAD7
52	316	17	-0.6	-0.03	-0.09	Liquid-scint. counting
53	234.22	22.72	-26.3	-1.32	-3.01	Emanometry
54	350.1	20	10.1	0.50	1.25	Liquid-scint. counting
55	101	6	-68.2	-3.41	-12.70	Emanometry
56	310	20	-2.5	-0.13	-0.31	Direct gamma-spec.
57	314	30	-1.3	-0.06	-0.12	Emanometry

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
57	330	20	3.8	0.19	0.47	Direct gamma-spec.
58	290	15	-8.8	-0.44	-1.28	Liquid-scint. counting
58	315	29	-0.9	-0.05	-0.09	Direct gamma-spec.
59	254	15	-20.1	-1.01	-2.92	Liquid-scint. counting
59	284	14	-10.7	-0.53	-1.60	Emanometry
60	267.68	21.79	-15.8	-0.79	-1.86	Emanometry
61	277	14	-12.9	-0.64	-1.93	Direct gamma-spec.
61	282	8	-11.3	-0.57	-2.01	Emanometry
61	319	27	0.3	0.02	0.03	Liquid-scint. counting
62	293	29.3	-7.9	-0.39	-0.75	AlphaGuard
62	297.4	24.45	-6.5	-0.32	-0.70	Liquid-scint. counting
63	269	39	-15.4	-0.77	-1.16	solid scintilation
64	355.69	13.5	11.9	0.59	1.80	Liquid-scint. counting
65	140.4	28.1	-55.8	-2.79	-5.49	Liquid-scint. counting
65	151.6	37.9	-52.3	-2.62	-4.04	Emanometry
66	252	15	-20.8	-1.04	-3.01	Direct gamma-spec.
67	343.7	38.7	8.1	0.40	0.61	Liquid-scint. counting
68	229	6	-28.0	-1.40	-5.21	Liquid-scint. counting
68	285.5	18	-10.2	-0.51	-1.35	Direct gamma-spec.
69	308	30	-3.1	-0.16	-0.29	Emanometry
70	284	23	-10.7	-0.53	-1.21	Direct gamma-spec.
70	296	21	-6.9	-0.35	-0.83	Liquid-scint. counting

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
71	350.088	37.033	10.1	0.50	0.80	Liquid-scint. counting
72	405	22.5	27.4	1.37	3.15	Liquid-scint. counting
73	133	15	-58.2	-2.91	-8.44	Emanometry
74	319.7	21.6	0.5	0.03	0.06	Liquid-scint. counting
75	322.8	10.46	1.5	0.08	0.25	Liquid-scint. counting
76	257	8	-19.2	-0.96	-3.41	Direct gamma-spec.
76	302	4	-5.0	-0.25	-0.97	Emanometry
77	342.94	9.935	7.8	0.39	1.32	Direct gamma-spec.
77	344.89	0.655	8.5	0.42	1.68	Liquid-scint. counting
78	335	63	5.3	0.27	0.26	Emanometry
79	284	17	-10.7	-0.53	-1.46	Liquid-scint. counting
80	308	16	-3.1	-0.16	-0.44	Direct gamma-spec.
81	633	12.7	99.1	4.95	15.42	Direct gamma-spec.
82	301	8	-5.3	-0.27	-0.95	Liquid-scint. counting
83	322	40	1.3	0.06	0.09	Emanometry
83	324	7	1.9	0.09	0.34	Liquid-scint. counting
84	317	4.1	-0.3	-0.02	-0.06	Liquid-scint. counting
85	457	38	43.7	2.19	3.37	Liquid-scint. counting
86	317	21	-0.3	-0.02	-0.04	Emanometry
87	357	52	12.2	0.61	0.71	Emanometry
88	292	36	-8.2	-0.41	-0.66	Liquid-scint. counting
89	311	15	-2.2	-0.11	-0.32	Direct gamma-spec.

Lab ID	Value (Bq/kg)	Uncertainty (Bq/kg;k=1)	D%	z score	ζ (zeta) score	Technique
89	352.4	3.9	10.8	0.54	2.09	Liquid-scint. counting
90	268.9	13	-15.4	-0.77	-2.38	Direct gamma-spec.
91	285	29	-10.4	-0.52	-1.00	Liquid-scint. counting
92	288	11	-9.4	-0.47	-1.55	Liquid-scint. counting
93	281	19	-11.6	-0.58	-1.49	Emanometry
94	335.6	40	5.5	0.28	0.41	Liquid-scint. counting
95	258.5	5.25	-18.7	-0.94	-3.53	Liquid-scint. counting
96	341.7	29.7	7.5	0.37	0.70	Emanometry
97	410	55	28.9	1.45	1.61	Liquid-scint. counting
98	318	12.8	0.0	0.00	0.00	Liquid-scint. counting
99	285	5	-10.4	-0.52	-1.97	Liquid-scint. counting
100	253	14	-20.4	-1.02	-3.06	Alpha Guard Pro 2000 professional radon monitor
100	268	20	-15.7	-0.79	-1.95	Direct gamma-spec.
101	324	5.5	1.9	0.09	0.35	Emanometry
101	339	3.5	6.6	0.33	1.28	Liquid-scint. counting

Source: JRC

Annex 11. Calculation of performance evaluation scores

Percentage difference (D%)

The percentage difference from the reference activity value was calculated with the following formula:

$$D_{\%} = 100 \times \frac{A_i - A_{ref}}{A_{ref}} \%$$

$$\tag{5}$$

where

A_i is the participant's results

A_{ref} is the assigned reference massic activity value

z-score and ζ (zeta)-score

$$z_{i} = \frac{A_{i} - A_{ref}}{\sigma_{PT}}$$

$$\zeta_{i} = \frac{A_{i} - A_{ref}}{\sqrt{u_{i}^{2} + u_{ref}^{2}}}$$

$$(6)$$

Where:

- A_i is the measurement result reported by a participant;
- u(A_i) is the standard measurement uncertainty reported by a participant;
- A_{ref} is the assigned reference value;
- u(A_{ref}) is the standard measurement uncertainty of the assigned value;
- σ_{PT} is the standard deviation for proficiency test assessment.

En number

The calculation of the E_n numbers was carried out according to the following formula:

$$E_n = \frac{A_i - A_{ref}}{\sqrt{U_i^2 + U_{ref}^2}} \tag{8}$$

where

A_i is the participant's results

A_{ref} is the assigned reference value

 U_i is the expanded uncertainty of a participant's result (k=2)

 U_{ref} is the expanded uncertainty of the assigned reference value (k=2)

Annex 12. The PomPlot interpretation

The PomPlot, an intuitive graphical method, is used for producing a summary overview of the participants' results (Spasova et al., 2007). It displays the relative deviations (D/MAD) of the individual results A from the reference value A_0 on the horizontal axis and relative uncertainties (u/MAD) on the vertical axis (Figure 16). For both axes, the variables are expressed as multiples of MAD, which is defined as the median of the absolute deviation from the reference value

$$MAD = Median D_i | (i = 1, ..., n)$$
(1)

where D_i is the difference between the reported and the reference activity concentration:

$$D_i = \frac{A_i}{A_0} - 1 \tag{2}$$

where

- A_i activity value reported by Laboratory i
- A₀ assigned activity reference value for Laboratory i

The median absolute deviation MAD is used because of its robustness.

For every data point the uncertainty is calculated as an independent sum of the reported combined uncertainties on A_i and A_0 , $u_i^2 = u_c^2(A_i) + u_c^2(A_0)$ (3)

where

- $u(A_i)$ standard uncertainty of activity value reported by Laboratory i (k=1)
- u(A₀) standard uncertainty of assigned activity reference value for Laboratory i (k=1)

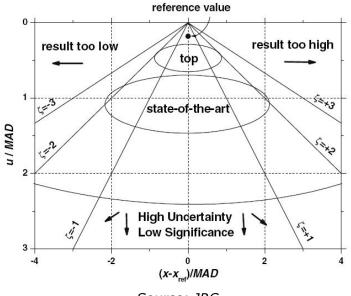


Figure 16. Interpretation of a PomPlot.

Source: JRC.

The ζ -scores, where $|\zeta|=|D/u|$, with values 1, 2 and 3, are represented by diagonal solid lines, creating the aspect of a pyramidal structure. The ζ -score is a measure for the deviation between laboratory result and reference value relative to the total uncertainty (ISO, 2015). The points on the right-hand side of the graph correspond to results that are higher than the reference value whereas lower values are situated on the left. When the uncertainty is small, the corresponding point is situated high in the graph. The most accurate results should be situated close to the top of the pyramid. Points outside of the ζ =±3 lines are probably inconsistent with the reference value.

Annex 13. Reference date and times of individual bottles

REM 2018 Radon-in-water PT, JRC-W1 sample

Reference date: 18 October 2018 (Thursday)

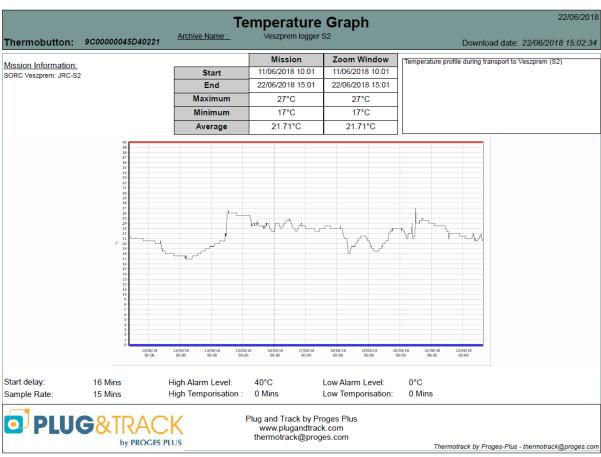
1 07:37 27 08:08 2 07:37 28 08:11 3 07:38 29 08:12 4 07:39 30 08:14 5 07:40 31 08:37 6 07:41 32 08:38 7 07:41 33 08:39 8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 36 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 <t< th=""><th>Bottle number</th><th>Reference time (UTC), hour:minute</th><th>Bottle number</th><th>Reference time (UTC), hour:minute</th></t<>	Bottle number	Reference time (UTC), hour:minute	Bottle number	Reference time (UTC), hour:minute
3 07:38 29 08:12 4 07:39 30 08:14 5 07:40 31 08:37 6 07:41 32 08:38 7 07:41 33 08:39 8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58	1	07:37	27	08:08
4 07:39 30 08:14 5 07:40 31 08:37 6 07:41 32 08:38 7 07:41 33 08:39 8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00	2	07:37	28	08:11
5 07:40 31 08:37 6 07:41 32 08:38 7 07:41 33 08:39 8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02	3	07:38	29	08:12
6 07:41 32 08:38 7 07:41 33 08:39 8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02	4	07:39	30	08:14
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8 07:42 34 08:40 9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	6	07:41	32	08:38
9 07:43 35 08:41 10 07:45 36 08:43 11 07:45 37 08:43 12 07:47 38 08:45 13 07:48 39 08:46 14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	7	07:41	33	08:39
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14 07:49 40 08:47 15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	12	07:47	38	08:45
15 07:50 41 08:49 16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	13	07:48	39	08:46
16 07:51 42 08:50 17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	14	07:49	40	08:47
17 07:53 43 08:51 18 07:54 44 08:52 19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	15	07:50	41	08:49
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19 07:55 45 08:53 20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	17	07:53	43	08:51
20 07:56 46 08:54 21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	18	07:54	44	08:52
21 07:58 47 08:56 22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	19	07:55	45	08:53
22 07:59 48 08:57 23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	20	07:56	46	08:54
23 08:00 49 08:58 24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	21	07:58	47	08:56
24 08:02 50 09:00 25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	22	07:59	48	08:57
25 08:03 51 09:01 26 08:06 52 09:02 27 08:08 53 09:03	23	08:00	49	08:58
26 08:06 52 09:02 27 08:08 53 09:03	24	08:02	50	09:00
27 08:08 53 09:03	25	08:03	51	09:01
	26	08:06	52	09:02
28 08:11 54 09:04	27	08:08	53	09:03
	28	08:11	54	09:04

Bottle number	Reference time (UTC), hour:minute	Bottle number	Reference time (UTC) hour:minute		
55	09:05 85		09:41		
56	09:06	86	09:42		
57	09:07	87	09:43		
58	09:08	88	09:44		
59	09:09	89	09:45		
60	09:10	90	09:46		
61	09:11	91	09:47		
62	09:12	92	09:48		
63	09:13	93	09:49		
64	09:19	94	09:50		
65	09:21	95	09:51		
66	09:22	96	09:52		
67	09:22	97	09:54		
68	09:23	98	09:55		
69	09:24	99	09:56		
70	09:25	100	09:57		
71	09:26	101	10:01		
72	09:27	102	10:02		
73	09:28	103	10:03		
74	09:29	104	10:04		
75	09:30	105	10:05		
76	09:31	106	10:06		
77	09:32	107	10:07		
78	09:33	108	10:08		
79	09:34	109	10:09		
80	09:35	110	10:10		
81	09:36	111	10:19		
82	09:38	112	10:19		
83	09:39	113	10:20		
84	09:40	114	10:21		

Bottle number	Reference time (UTC), hour:minute	Bottle number	Reference time (UTC), hour:minute
115	10:22	144	11:25
116	10:23 145		11:26
117	10:24	146	11:28
118	10:25	147	11:29
119	10:26	148	11:31
120	10:27	149	11:32
121	10:58	150	11:33
122	10:59	10:59	
123	11:00	11:00	
124	11:01	11:01	
125	11:02		
126	11:03		
127	11:05		
128	11:06		
129	11:07		
130	11:08		
131	11:09		
132	11:10		
133	11:11		
134	11:12		
135	11:13		
136	11:14		
137	11:15		
138	11:16		
139	11:17		
140	11:18		
141	11:22		
142	11:23		
143	11:24		

Annex 14. Temperature logger and a typical temperature profile recorded during sample transport





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