

JRC TECHNICAL REPORT

REM 2019 proficiency test on gross alpha/beta activity concentration in drinking water

JRC REM2019 PT

Viktor Jobbágy, Edmond Dupuis, Håkan Emteborg, Mikael Hult

2021



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Contact information Name: Viktor Jobbágy Address: Joint Research Centre (JRC), Retieseweg 111, B-2440 Geel, Belgium Email: viktor.jobbagy@ec.europa.eu Tel.: +32 (0)14 571 251

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Foreword

This report focuses on the technical evaluation of the REM (Radioactivity Environmental Monitoring) 2019 gross alpha/beta activity concentration measurements in drinking water proficiency test. It contains details on the material characterisation, performance evaluation (the key scores of the participants), information on the participants' organisation, the applied analytical methods and feedback from participants.

The REM 2019 proficiency test was performed within the institutional work programme of the JRC Directorate G (Nuclear Safety and Security) as described in the H-2020 JRC-Work Package SELMER (Support to European Laboratories Measuring Environmental Radioactivity) in and the JRC-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 and thereby also supporting Article 39.

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The authors want to express their sincere appreciation to all the 154 participating laboratories (listed in **Annex 6**) for their active collaboration.

This proficiency test exercise would not have been completed without the commitment and cooperation of Edmond Dupuis¹, Michel Bruggeman and his technical staff from the Low-level Radioactivity Measurements (LRM) expert group at SCK CEN (Belgian Nuclear Research Centre in Mol, Belgium) for doing gross alpha/beta measurements and nuclide specific analysis.

Also many thanks to the following JRC colleagues for supporting the PT exercise:

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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),
- JRC-Geel, F.5 Food & Feed Compliance Unit: James Snell and Geert Van Britsom for performing complimentary ICP-OES elemental analysis
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Authors

Viktor Jobbágy, Edmond Dupuis, Håkan Emteborg, Mikael Hult

¹ Since July 2021 freelance expert and available at <u>dupuis.edmond@gmail.com</u>

Abstract

A large scale Europe-wide proficiency test (sometimes referred to as *REM2019 PT*) on the determination of the gross alpha/beta activity concentration in drinking water was organised by JRC-Geel. The 154 participating environmental radioactivity monitoring laboratories were either nominated by their corresponding national authorities or invited by JRC to participate.

One spiked water sample (JRC-GAB2) and a commercially available natural mineral water (JRC-GAB1) were selected as reference materials for this proficiency test after initial testing using nuclide-specific analyses and gross alpha/beta measurements. The JRC-GAB1 reference material (mineral water) had intermediate mineral content and gross activity above the parametric values defined in the E-DWD (Euratom Drinking Water Directive). The original mineral water was collected from a natural water source in France.

Reference values were established in collaboration between the JRC-Geel and the Belgian Nuclear Research Centre (SCK CEN). The homogeneity, short-and long term stability of the batch of distributed PT reference materials were checked, their contribution to the uncertainty of the reference value was assessed.

The assigned reference value for the spiked PT reference material was established by gravimetric spiking (often referred to as formulation by weighing). For the natural PT reference material, the assigned reference value for gross-alpha activity was established by radionuclide specific analysis whilst the gross-beta reference value was established by calculating the gross activity from several reference measurements using the power-moderated mean of the individual measurement results. For the homogeneity and short term stability study, three independent measurement methods were used: alpha-particle spectrometry (AS), liquid scintillation counting (LSC) and inductively coupled plasma-optical emission spectrometry (ICP-OES). The uncertainty of the reference values includes the uncertainty related to stability, between-bottle homogeneity and characterisation of PT reference samples.

The performance of each participating laboratory was evaluated with respect to the reference value using relative deviations, z-score and zeta-score. Additionally, Youden plots and PomPlots were made to visualise reported data in comparison to the reference values. The reported results were evaluated and grouped by analytical methods to check for method dependency, accreditation, radionuclides used for efficiency calibration, time delay and if documented ISO standards were followed.

It was found that the close to 50% of the gross activity results still deviate more than the standard deviation for proficiency assessment (σ_{PT}) which was set to 30% for JRC-GAB1 sample and 20% for JRC-GAB2 sample, respectively. The general measurement performance is thus not satisfactory regardless of the methods used. This suggests that the existing analytical procedures and international standards need to be critically revised and harmonised for gross alpha/beta measurement in order to obtain more reliable and comparable measurement results. Furthermore, when the reported value with its uncertainty was evaluated using the zeta score, even fewer acceptable scores were found: 41% and 55% for gross alpha activity concentration; 38% and 62% for gross beta activity concentration in JRC-GAB1 and JRC-GAB2 PT reference materials, respectively. A key problem is that many variables influence and might interfere gross activity measurements. This makes it difficult to keep the analytical conditions under control and can lead to poor repeatability which affects accuracy as well. Therefore, it is of great importance to harmonise methods by fixing as many parameters as possible via true standardisation of the analytical methods.

However, in certain cases the performance of methods using LSC and proportional counting techniques seems to be better than those based on solid state scintillation counters or other detectors. On the basis of the 14 best and most consistently performing participants (or methods), JRC is proposing "*Best practices*" to follow briefly in this report.

1 Introduction, policy context

This is a detailed technical report describing a large scale Europe-wide proficiency test (referred to as "*REM2019 PT*") on gross alpha/beta activity concentration measurements in water organised by the European Commission's Joint Research Centre in Geel, Belgium (JRC-Geel). The purpose of the REM2019 PT was to assess the analytical capabilities of European environmental radioactivity monitoring laboratories on the determination of gross alpha/beta activity concentration in drinking waters.

The REM2019 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) as a repetition of the REM2012 exercise (Jobbágy et al., 2015, 2016). This European scale PT supports the EURATOM Drinking Water Directive (EURATOM, 2013)² (referred to as the E-DWD) and was considered as a high priority project after the outcomes of the REM2012 PT.

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 Articles 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 E-DWD, which covers several naturally occurring radionuclides and gross alpha and gross beta activity concentration due to their impact on human health.

Two types of PT reference materials were distributed to participants: (i) a natural mineral water containing naturally occurring radionuclides and minerals, named JRC-GAB1, and (ii) a spiked water sample prepared gravimetrically by spiking of demineralised laboratory water at JRC-Geel, named JRC-GAB2.

In total, 154 participants registered for this PT. Out of 154 participants 145 submitted at least one gross alpha/beta activity concentration measurement result which totalled 709 individual measurement results. In addition to the gross alpha and gross beta activity concentrations, we requested the participants to determine the total dissolved solid (TDS) content of the PT reference materials which gives information about the mineralisation of the water samples.

Due to the coronavirus pandemic, the initial deadline for reporting (20 March 2020) was extended by two month (17 June 2020) to allow participants to submit their measurement results. Since another closure was introduced at JRC-Geel in July 2020 and priority was given to quality management related tasks (e.g. accreditation) until February 2021, the preparation of the PT report was put on hold.

Some of the REM PTs organised prior to JRC-involvement in 2003 displayed some deficiencies related to the lack of metrological traceability, a missing or incomplete homogeneity and/or stability study of the material. This PT provides reference materials with homogeneity and stability tested according to ISO Guide 35:2017, ISO 13528:2015 and included interference-free material with metrological traceable reference values from measurements/certificated of individual radionuclides.

The REM2019 PT followed the ISO Guide 35:2017, ISO 17034:2016, ISO/IEC 17043:2010 and ISO 13528:2015 standards on characterisation of reference materials, production of reference materials, organising proficiency tests and performance assessments, respectively. The gamma-ray spectrometry measurements at JRC-Geel were done according to ISO/IEC 17025:2017, the gross alpha/beta measurements and the methods for U, Pb-210, Ra-226 and Ra-228 measurements at SCK CEN are under ISO/IEC 17025:2017 accreditation.

This report focuses on the technical details of the PT preparation, data evaluation and analysis. Furthermore, the questionnaire associated with this PT is evaluated and the participants' feedback is presented.

As a very important milestone, this project passed a rigorous assessment during an external audit by the Belgian Accreditation Body (BELAC) in February 2021 as part of the JRC Directorate G.2 accreditation procedure for ISO/IEC 17043:2010.

² <u>Council Directive 2013/51/EURATOM</u> 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.

2 **Project management and organisation details**

2.1 Responsibilities and roles

The REM2019 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: <u>JRC-GEE-REM-COMPARISONS@ec.europa.eu</u>.

The responsibilities amongst the involved staff of the organiser:

- Viktor Jobbágy: PT coordinator, packing, logistics, liquid scintillation counting and alpha-particle spectrometry analysis, reporting. main author of report.
- Mikael Hult: team leader, gamma-ray spectrometry and quality control.
- Håkan Emteborg: team leader, PT reference material processing and storage,
- Petya Malo: logistics assistant, administration, quality control.
- Heiko Stroh: packing, logistics, gamma-ray spectrometry analysis.
- Gerd Marissens: packing, gamma-ray spectrometry, logistics.
- Jan Paepen: packing, data validation of participants' performance.
- Piotr Robouch: quality control, data validation of participants' performance.
- Katarzyna Sobiech-Matura: internal review of the report.
- Ulf Jacobsson: G.2 Unit Quality Officer, developer of the REMPES application.
- Advisory group members: Arjan Plompen Head of Unit ad interim, Petya Malo ISO 17043 Quality management, Mikael Hult as Team Leader, Jan Paepen and Stefaan Pommé as Statistical advisors, Piotr Robouch as External advisor.

2.2 Subcontractors, collaborators

JRC-Geel subcontracted some of the tasks to other JRC directorates and an external expert institute in the field. The main contacts and the name of each collaborating entity are listed below:

- Edmond Dupuis and Michel Bruggeman: SCK CEN (Belgian Nuclear Research Centre in Mol, Belgium): performing preliminary material characterisation, radionuclide specific and gross activity measurements. SCK CEN is accredited according to ISO/IEC 17025:2017 to perform gross alpha and beta activity measurements in waters,
- Håkan Emteborg (JRC-Geel, F.6 Reference Materials Unit): PT reference material processing, packing and providing temporary sample storage rooms. JRC-Geel Dir.F.6 is an accredited Certified Reference Materials producer according to ISO 17034:2016,
- James Snell (JRC-Geel, F.5 Food & Feed Compliance Unit): performing complimentary ICP-OES elemental analysis.

Edmond Dupuis from SCK CEN actively contributed to the REM2019 PT by enabling access to the natural mineral water sample, performing preliminary material characterisation, radionuclide specific measurements and giving technical support throughout the PT. The measurement results from SCK CEN were either used to confirm the JRC-Geel measurement results or used solely for assigning reference value.

2.3 Participating organisations, participation fee

The participation in the PT was based predominantly on nominations and direct invitation by either JRC or the International Atomic Energy Agency (IAEA) through their network. 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 154 laboratories from all over Europe participated in the PT (from 26 EU countries and 11 EU associated countries). In addition to the registered organisations, JRC-Geel received

additional participation requests by e-mail. Unfortunately, these requests were rejected because they were either received after the registration deadline or participation requests were coming outside Europe (USA, African or Asian countries) where other financial or logistics issues could have emerged (e.g. customs). The full list of all registered 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 organisation (JRC-Geel), except the sample analysis related costs.

2.4 Timeline and announcements

Table 1 shows the REM 2019 PT tentative time line.

 Table 1. Timeline of the REM2019 PT exercise.

September 2018	EC Directorate for Energy and EURATOM Art. 35-36 experts' meeting: request to JRC			
July-Aug 2019	JRC-Geel contacted national authorities, laboratories requesting nominations and expression of interest			
3 September 2019	Invitation letter sent to the nominated/interested laboratories			
14 and 25 September 2019	Registration deadline			
7-14 January 2020	PT material shipment to participants			
20 March 2020	Initial submission deadline for laboratories' results and questionnaire			
17 June 2020	Extended submission deadline due to Covid-19 situation			
3 September 2020	Preliminary results sent to participants			
4-6 May 2021	Follow-up virtual-workshop on REM2019 PT			
2021	Technical report ("Final report")			

The announcements and communication documents are presented in **Annex 1-6**.

Note: Due to the coronavirus pandemic many laboratories running at limited capacity so the initial deadline for reporting (20 March 2020) was extended until 17 June 2020, to allow participants to perform measurements and submit their measurement result. Since another closure was introduced at JRC-Geel in July 2020, the preparation of this PT report was further delayed.

2.5 PT reference materials

To run a representative PT, the selection of test items (PT reference materials) is a crucial step. Therefore, our first objective was to select waters as close to the samples European laboratories usually measure as possible. For this reason, an initial radioanalytical survey to study the naturally occurring alpha emitting radionuclides was carried out in some different mineral waters from the European market. The activity concentrations of the most abundant naturally occurring alpha-emitting radionuclides (²²⁶Ra, ²¹⁰Po, ²³⁴U, ²³⁵U, ²³⁸U and ²²⁸Th) were determined. In order to find representative water samples of natural origin for the gross alpha/beta PT, the following important parameters were taken into account during the PT reference material selection: activity concentration of the alpha-emitting radionuclides, salinity and the chemical composition. In

terms of salinity, the selected PT test items were preferably in the range where the majority of drinking waters are (~50-1500 mg L⁻¹). It was also decided that apart from a natural matrix water, a spiked PT reference material would be prepared gravimetrically. The latter sample is one step away from the natural samples the European monitoring labs are measuring in their daily work but the reference activity concentration will have a lower uncertainty so it is useful to combine the two types of samples in a PT. Thus in total two types of waters were selected as PT reference materials, a natural origin mineral water (JRC-GAB1) and a deionised water that was spiked (JRC-GAB2) with alpha and beta emitting radionuclides and inactive inorganic salts to better represent typical water sample³.

Monitoring laboratories have to be confident in measuring activities near the screening levels of the recent WHO guidelines and E-DWD (WHO, 2017; EC, 2013) and should meet the recommendations on detection limits. Therefore, we selected the PT samples considering these performance quality parameters as shown in **Table 2**.

Parameter	Activity concentration (Bq L ⁻¹)	References
Limit of detection	0.02-0.1	ISO 9696:2007; ISO 9697:2008
	Gross α = 0.04; Gross β = 0.4	EC, 2013
Cereopine lovele	Gross α = 0.5; Gross β = 1	WHO, 2017
	Gross $\alpha = 0.1$; Gross $\beta = 1$	EC, 2013

Table 2. Sources and parameters used for establishing requirements for the REM2019 PT water selection.

Each water sample was filled in a 1-L bottle (See chapter 2.6). After filling, each bottle was wiped dry carefully using towels and paper tissues. There was approximately 1 kg of water in each bottle which was controlled with a balance during dispensing. This volume was expected to be sufficient for typical gross activity analyses.

Major chemical characterisation of both PT reference materials was performed. They contained calcium, magnesium, sodium, potassium, chlorides and nitrates and non-interfering trace elements as carriers. Both water samples were acidified with nitric acid to adjust pH \cong 1-2 following ISO 5667-3:2018 (section A.5). A detailed description of the preparation of the PT reference materials is described in Chapter 3.1.

JRC-GAB1 PT reference material contained only naturally occurring alpha - and beta emitting radionuclides. JRC-GAB2 PT reference material contained mainly artificial (anthropogenic) alpha - and beta emitting radionuclides with ⁴⁰K being the only naturally occurring radionuclide in the form of KCl (Merck, analytical grade, K assay content 99.5%).

2.6 Logistics: packaging and shipment

The PT reference materials were filled into regular acid proof 1L high-density polyethylene (HDPE) sample storage laboratory bottles. Crimp films were used to cover the screw caps serving as anti-tamper seal. They were in two different colours to facilitate visual identification of the two different PT samples.

The individual PT test items assigned to different studies (homogeneity, stability and reference value) 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 460 individual units of PT reference materials were split in the following way:

- 308 units per PT sample were sent to the participants,
- 10 units per PT sample were assigned for the homogeneity study and assigning reference value (JRC-GAB1) or verifying formulation/spiking (JRC-GAB2),
- 6 units per PT sample were used in the stability study,
- 96 units per PT sample served as back-up.

³ More details on the PT sample selection for the previous PT are described elsewhere (Jobbágy et al., 2013).

An example of labelled storage bottles containing JRC-GAB1 PT sample is presented on Figure 1.



Figure 1. REM2019 PT reference material (JRC GAB1) after dispensing into storage bottles.

Since temperatures below freezing point could be expected in some cases, special precautions were taken to ensure that the PT material arrived at all the participating laboratories in good condition. Therefore, robust physical and thermal resistant packaging transport boxes were used (model: EXAM, HIGH-Q Pack 20L). They are insulated containers moulded in technical polyurethane foam accommodated in water-resistant cardboard. Double layered card boxes were used for shipments where there was no risk of sample freezing.

The package contained the two units of PT reference materials; a natural mineral water (JRC-GAB1) and a spiked ASTM (American Society for Testing and Materials) D1193-06 type-II laboratory water (JRC-GAB2). The HDPE bottles containing the water samples were put into a layer of spill adsorbing material and eventually into a sealable plastic foil to contain any spillage during the transport.

Each package shipped to participants contained the following items:

- 2-4 units of PT reference material in 1 L HDPE bottles, each wrapped in bubble foil sealed in a plastic bag,
- accompanying letter,
- material information sheet,
- sample receipt form.

The packages containing the PT samples were distributed by a logistics company. In general, the packages arrived to the participating laboratories within 1 to 10 days after dispatch. In some cases there were some delays due to e.g. customs procedure outside the Schengen area or internal procedure reasons at the participants' organisation. The activity of the shipped PT samples were well below the exemption levels in terms of both activity concentration and total activity.

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

Participants were instructed to store their PT samples in a dark place between +4 °C and +20 °C.. The PT organiser recommendation was to store the sample bottle at room temperature prior to any analysis until it reached thermal equilibrium with its environment.

All samples arrived at the participants without any major problems. Only one participant requested additional samples to do extra measurements.

2.7 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: <u>https://web.jrc.ec.europa.eu/ilcReportingWeb</u>

Participants had the opportunity to report results obtained by different analytical methods following the organiser's instructions including:

- the measurement technique used,
- one mean result per measurement technique (in mBq/L),
- associated uncertainty and the coverage factor of k.

Note on reference date: in theory, decay correction is not possible for gross alpha/beta parameters. Therefore, reference date was not given.

2.8 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 via the following link on the REM2019 PT result reporting website:

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

2.9 Data treatment

All results were treated confidentially; identities were and will be kept anonymous even beyond the PT exercise. However, the results and performance of <u>each nominated laboratory</u> will be 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**).

Participants had to agree with our data treatment and privacy policy during the registration to comply with the European General Data Protection Regulation (GDPR). The participants were informed that the name of the organisation will appear in the final report.

2.10 Use of proficiency testing results by participants and accreditation bodies

The results and scores of a proficiency testing exercise should be used as described in Clause C.4 and C.5 of the ISO/IEC 17043:2010. The aforementioned clauses warn the laboratories and accreditation bodies to use proficiency testing (especially results from only one PT) as the only tool in the accreditation processes to determine competence. Performance scores from a PT are momentary evidence of competence for that particular exercise and may not necessarily reflect general long-term competence of a laboratory.

3 Proficiency test reference materials: processing and characterisation

3.1 PT reference material production and processing

The reference material processing and their treatment was identical for both PT-materials. The vessels used for PT reference material homogenisation and production steps are described in sub-chapter 3.1.1 and 3.1.2.

3.1.1 Vessels for homogenisation and processing

There were two large volume custom-made vessels used for homogenisation and processing (one vessel for each material) (Teblick, Antwerp, BE). Each vessel fulfils the requirements for trace elements in water reference materials since they can be rigorously cleaned with a sequence of strong acid and Type I ultrapure water. The wall of the vessels is a sandwich construction and consists of glass fibre reinforced plastic (GRP) as outer liner and Teflon[®] PFA (perfluoroalkoxy copolymer resin) as an inner liner. The dimensions of these vessels are such that the Dyna-mixer CM500 (WAB, Basel - Switzerland) can be used for easy cleaning of these vessels between projects. Consequently before filling with the water and the Type II pure water, the vessels were rinsed with >50 L Type II pure water and placed in the Dyna-mixer CM500. The whole system, when comprising of four inter-connected vessels, allows homogenisation of up to 2 m³ of water at the same time. The pneumatically driven bellow-pumps (Iwaki FS-30-HT2) are made so that all parts in contact with the water are made of PFA or PTFE. The vessels are also equipped with a level sensor and via a feedback circuit the pumping speed is individually controlled so that the level stays the same in all vessels during recirculation. A full re-circulation of 2 m³ can be achieved in approximately one hour with a flow of about 30 L/min per pump.

3.1.2 Production and processing

The natural mineral water sample (JRC-GAB1) was provided by a mineral water supplier in a 1 m³ transport container, whilst the water for JRC-GAB2 was produced in-house as described in the next paragraphs.

JRC-GAB1 PT reference material was produced from a commercial mineral water from France. For the homogenisation, one of the PFA-lined vessel with approximately 550 L was filled with the mineral water which was acidified to pH = 1.4 ± 0.1 with analytical grade concentrated HNO₃. The acidified water was recirculated for few days at 15 L/min using the inert Iwaki bellow pumps. During filling an intermediate PFA buffer tank of 20 L was used and the water was pumped from the main tank into the buffer tank. The buffer tank was placed inside a clean bench and the water bottles were filled automatically when placed on a balance subsequently reaching a mass set-point. Prior to filling, the buffer tank was rinsed with 2 x 10 L of Type I water (18.2 M Ω cm, 0.056 μ S/cm at 25 °C and TOC < 5 ng/mL) from a Milli-Q Advantage system (Millipore, Billerica, MA, USA) and 20 L of mineral water. In this manner 460 bottles were filled. The 1-L bottles were made of high density polyethylene (HDPE) with a leak-proof HDPE-screw cap (Nalgene).

JRC-GAB2 PT reference material was a spiked Type II water from a Millipore ELIX-35 system (>5 M Ω cm, 0.2 µS/cm at 25 °C and TOC < 30 ng/mL) with added inorganic salt mixture composed by KCl (Merck, assay content 99.5%), NaCl, CaCl₂, MgCl₂, Sm(NO₃)₃ and Sr(NO₃)₂. During several days, 500 L of Type II water was collected in portions into the main PFA-lined drum of 550 L. Subsequently the preliminary weighed salt mixture was added. Thereafter, analytical grade concentrated nitric acid was added to obtain the desired pH (pH = 1.4 ± 0.1) followed by ⁹⁰Sr/⁹⁰Y and ²⁴¹Am spikes from standardized solutions. The ⁹⁰Sr/⁹⁰Y with massic activity of (121.4 ± 1.0) Bq/g, and ²⁴¹Am (80.99 ± 0.40) Bq/g, radioactive solutions were standardized at the Czech Metrology Institute (Eurostandard). Reference date for both standardised solutions was 10 September 2019. The contents were thereafter mixed as previously described using the Iwaki inert bellows pump of the water handling system for 16 hours at 15 L/min. Subsequently, 460 of the 1-L HDPE bottles (Nalgene) were filled in the same way as for JRC-GAB1 reference material.

For both type of reference materials crimp films were used to cover the screw caps of the bottled materials serving as anti-tamper seal. The crimp films were used in two different colours to facilitate visual identification of the two PT reference materials.

After bottling, the PT reference materials were transported into their interim storage room within JRC-Geel premises. PT reference materials were stored in a dark and dry storage place at 4 °C.

Information on the radionuclide composition and chemical composition of the REM2019 PT samples are presented in **Table 3** and in **Table 4**.

Table 3. The radionuclide composition and total dissolved solid contents of the REM2019 PT reference materials

 (uncertainties at k=1).

JRC-GAB1 (Natural matrix)	JRC-GAB2 (QC Spiked water)
Total dissolved solids content: (966 ± 27) mg/L	Total dissolved solids content: (356 ± 20) mg/L
Source of alpha activity contribution: $^{\rm 234}\text{U}$ and $^{\rm 238}\text{U}$	Source of alpha activity contribution: ²⁴¹ Am
Source of beta activity contribution: ⁴⁰ K, ²³⁴ Th, ²³⁴ Pa	Source of beta activity contribution: ⁴⁰ K, ⁹⁰ Sr/ ⁹⁰ Y in equilibrium

Table 4. The chemical composition and concentration of JRC-GAB2 PT reference material from gravimetric weighing. The relative standard uncertainty on the chemical concentration was approximately 1.0%.

Chemical element/ion	Weighed amount (g)	Chemical concentration (mg/L)
Na (sodium)	19.650	38.1
Ca (calcium)	23.140	44.9
Sr (strontium)	4.160	8.1
Mg (magnesium)	10.750	20.8
K* (potassium)	8.890	17.2
Sm (samarium)	0.611	1.2
Cl (chlorine)	110.660	214.5
NO_3^- (nitrate)	5.900	11.4

Reference values for JRC-GAB2 PT reference material were determined by using gravimetric approach, where the standardized solutions were weighed on a calibrated balance which is traceable to the BIPM (SI) standard kilogram via JRC-Geel standard kilogram. The uncertainty on the weighing was approximately 0.1%.

3.2 Gross alpha/beta activity measurements

3.2.1 Measurements performed at SCK CEN

The gross alpha/beta activity measurements in water samples performed at SCK CEN were based on ISO 10704:2019 standard evaporation and co-precipitation approaches. The direct evaporation method using an automatic evaporator where 200-250 mL of water was directly evaporated on the cup before being measured in a ZnS counter and or in a proportional counter. Another applied approach was to pre-concentrate samples with the Buchi Syncore system and after, evaporate it on a planchet to obtain a homogenous dry deposit. Thanks to a self-absorption curve, a correction factor was calculated and used in order to be able to convert this activity into activity concentration values. Sample preparation started with evaporation of 250 mL sample. To keep all the soluble materials in solution, 5 mL of 10 % acetic acid were added and evaporated under vacuum in a BuchiSyncore Analyst system with a flush back option. With this system all the activity and salt were concentrated in a small volume of about 3 mL. This sample volume was transferred into a stainless steel planchet and the water was dried on a (glass-ceramic) hotplate until complete dryness. The residue was weighed and measured with the gross alpha/beta system.

Detector system for gross alpha counting: 5 inch (1 inch = 2.54 cm) ZnS(Ag) low background detector. To reduce the background of the counter, the counting cell is flushed with a low flow of dry nitrogen gas. Typical measurement time: 5×10000 s and 10×10000 s. Alpha background: 0.04 - 0.09 cpm.

Detector system for gross beta counting: the samples were counted in a proportional counter 5 inch very low background Canberra LB4200 and low background Canberra Tennelec LB 5500 with sample changer. Typical measurement time: 6×3000 s and 10×6000 s. Beta background: < 2.5 cpm. For quality check purposes background is measured before and after each sample measurement. The efficiency of all the counters is controlled each month with a certified source made at SCK-CEN. Radionuclides used for calibration: ²³⁹Pu for alpha and ⁹⁰Sr/⁹⁰Y for beta. Self-absorption factor is determined by using NaNO₃.

3.2.2 Measurements performed at JRC-Geel

The JRC-Geel method for the determination of gross alpha/beta activity concentrations in water samples was based on the ISO 11704:2018 standard. In the sample concentration step approximately an aliquot of 250-500 g of water was weighed into a glass beaker and acidified to approximately pH 2 using nitric acid if not acidified before. The sample was gently evaporated to approximately 20-30 mL on an electrical plate at maximum 80 °C . The beaker was cooled down and the remaining water was weighed. Then, an aliquot of 10 mL of water sample was dispensed into a 20 mL low-diffusion polyethylene liquid scintillation vial containing 10 mL of Ultima Gold AB liquid scintillation cocktail. A vial was closed with a cap and shaken vigorously by hand for 30 seconds. The LSC vial was placed into a cooled tray of the liquid scintillation (LS) counter for minimum 3 hours to reduce events from photoluminescence. Then samples were measured for 6 hours and the alpha/beta spectrum was recorded using the low background Quantulus 1220 counter (Perkin Elmer). Alpha particles were counted in a window between channels 500 - 1000, and beta particles were registered in a window between channels 500 - 1000, and beta particles were registered in a window between channels 500 - 1000, and beta particles were registered in a window between channels 500 - 1000, and beta particles were registered in a window between channels 500 - 1000.

Before measuring a batch of samples the LS counter's pulse shape analyser (PSA) value of the alpha/beta discriminator was adjusted by dispensing known activities of alpha emitting (²⁴¹Am) and beta emitting (⁹⁰Sr/⁹⁰Y) radionuclide standard solution to a concentrated water sample and measuring alpha and beta spectra. For both water samples (JRC-GAB1 and JRC-GAB2) the optimum PSA values were found to be 70. Thus the same settings were used for the LSC measurements.

Similarly, alpha and beta counting efficiencies were determined by dispensing a known activity of alpha or beta emitting radionuclide standard solution to thermally pre-concentrated water samples. Alpha counting efficiency (referred to ²⁴¹Am) was 0.98 ± 0.01, and beta counting efficiency (referred to ⁹⁰Sr/⁹⁰Y) was 0.96 ± 0.01. The alpha to beta spillover values for JRC-GAB1 and JRC-GAB2 samples were 1.12% and 1.24%, respectively. The beta to alpha spillover values for JRC-GAB1 and JRC-GAB2 samples were 0.97% and 0.89%, respectively.

Blank samples were prepared the same way as the routine samples. An aliquot of 10 ml pre-concentrated deionized water sample was dispensed into a 20 mL low-diffusion polyethylene liquid scintillation vial and mixed with 10 mL of Ultima Gold AB scintillation cocktail. Blank samples were measured before and after measuring a batch of samples.

3.3 Radionuclide-specific measurements

3.3.1 Alpha-particle spectrometry measurements of uranium isotopes and ²⁴¹Am

For both PT water samples a known amount of tracers (232 U for JRC-GAB1 samples or 243 Am for JRC-GAB2 samples) were added gravimetrically before the pre-concentration step. Pre-concentration of radionuclides was done prior to the separation phase from 0.5 L water samples by Fe(OH)₃ co-precipitation. The precipitate was dissolved in 15 mL 8 mol/L or 3 mol/L HNO₃ and loaded onto the extraction chromatography columns. TEVA, UTEVA and DGA solid phase extraction chromatographic resins were used for the sequential separation of U isotopes and Am from the interfering radionuclides and matrix elements.

Sources for alpha-particle spectrometry were prepared by electrodeposition from H_2SO_4/NH_4SO_4 media, uranium isotopes and ²⁴¹Am were electrodeposited onto stainless steel discs and measured by alpha-particle spectrometry. The detailed analytical procedures are described elsewhere (Jobbágy et al., 2013; Groska et al., 2016).

3.3.2 ⁴⁰K measurement with gamma-ray spectrometry

The water from three bottles for each of JRC-GAB1 and JRC-GAB2 were measured using gamma-ray spectrometry. The measurements were performed both in the 225 m deep underground laboratory HADES (Hult et al., 2021) and above ground at JRC-Geel. Due to the low count-rates, only the measurements from

detector Ge-5 in HADES were used for quantification. It is a 50% relative efficiency planar detector with a thin top deadlayer (so-called BEGe-detector). The count rates for 40 K in both water samples were low which indicated very long measurement times (about 1 week per measurement) were needed with this technique but it is very robust as it requires no pre-treatment of the sample (water). The full energy peak efficiency curve was derived from a reference sample (liquid solution of mixed radionuclides) from NPL (National Physics Laboratory, UK). The efficiency transfer to correct for small differences in filling height was performed using the Monte Carlo code EGSnrc. The reported uncertainties are the combined standard uncertainties (k = 1) with major components being counting statistics and the full energy peak efficiency. The massic activities of 40 K (as average of three bottles) are reported in **Table 5**. Due to the relatively high uncertainty (due to low count-rate) the value for JRC-GAB1 was not included in the determination of the reference values but served as a robust check of other methods. In addition, these measurements served to confirm the absence of other (gamma-ray emitting) radioactive impurities.

Sample	Massic activity (k=1)
JRC-GAB1	(290 ± 100) mBq·kg ⁻¹
JRC-GAB2	(480 ± 80) mBq·kg ⁻¹

Table 5. Massic activities of ⁴⁰K from underground gamma-ray spectrometry.

3.4 Summary of the radionuclide-specific and gross activity concentration results

It was important to confirm the radionuclide composition of the two PT samples in order to ensure that interference free measurements can be performed. The radionuclide-specific and gross activity measurements were done using independent measurement methods at JRC and SCK CEN. The measurement results of the individual radionuclides and gross alpha/beta activity concentration for JRC-GAB1 and JRC-GAB2 samples are summarised in **Table 6** and **Table 7**, respectively.

Radionuclide/parameter	Activity concentration	Related measurement
Gross alpha activity concentration (JRC)	(282 ± 22) mBq/L	alpha activity
Gross alpha activity concentration (SCK CEN)	Co-precipitation (354 ± 40) mBq/L Direct evaporation (350 ± 43) mBq/L	alpha activity
²³⁴ U (JRC)	(292 ± 22) mBq/L	alpha activity
²³⁴ U (SCK CEN)	(295 ± 16) mBq/L	alpha activity
²³⁵ U (JRC)	(3.3 ± 0.6) mBq/L	alpha activity
²³⁵ U (SCK CEN)	(2.8 ± 0.4) mBq/L	alpha activity
²³⁸ U (JRC)	(76 ± 6) mBq/L	alpha activity
²³⁸ U (SCK CEN)	(79 ± 4) mBq/L	alpha activity
²²⁶ Ra: RadDisk-alpha spec (JRC)	<10 mBq/L	alpha activity
²²⁶ Ra: LSC and Lucas method (SCK CEN)	<3 mBq/L	alpha activity
Gross beta activity concentration (JRC)	(432 ± 58) mBq/L	beta activity
Gross beta activity concentration (SCK CEN)	Direct evaporation (330 \pm 40) mBq/L	beta activity
²¹⁰ Pb (SCK CEN)	< 9 mBq/L	beta activity
²²⁸ Ra (SCK CEN)	< 9 mBq/L	beta activity
⁴⁰ K by ICP-OES (JRC)	(187 ± 19) mBq/L	beta activity
⁴⁰ K by ICP-AES (SCK CEN)	(210 ± 10) mBq/L	beta activity
Total beta activity (sum of ⁴⁰ K, ²³⁴ Th and ²³⁴ Pa)	(339 ± 17) mBq/L	beta activity

Table 6. Summary of the radionuclide-specific and gross activity measurement results for JRC-GAB1 PT reference material. The results are presented as activity concentrations [mBq/L], uncertainties are expanded uncertainties (k = 2).

The mean results for JRC-GAB1 PT reference material obtained by different independent measurement techniques were consistent and within the corresponding measurement uncertainties. The gross alpha/beta activity measurement results were also in agreement with the radionuclide specific measurement results. It was confirmed that there were no interfering radionuclides in the JRC-GAB1 PT reference material that could cause significant bias from the reference value in this type of sample.

Radionuclide/parameter	Activity concentration	Related measurement
Gross alpha activity concentration (JRC)	(635 ± 50) mBq/L	alpha activity
Gross alpha activity concentration (SCK CEN)	Co-precipitation (750 ± 80) mBq/L Direct evaporation (670 ± 80) mBq/L	alpha activity
²⁴¹ Am alpha-particle spectrometry (JRC)	(729 ± 56) mBq/L	alpha activity
²⁴¹ Am gravimetric spike (JRC)	(731 ± 10) mBq/L	alpha activity
²⁴¹ Am gamma-ray spectrometry (JRC)	(750 ± 60) mBq/L	alpha activity
²⁴¹ Am alpha-particle spectrometry (SCK CEN)	(700 ± 110) mBq/L	alpha activity
²⁴¹ Am gamma-ray spectrometry (SCK CEN)	(650 ± 160) mBq/L	alpha activity
Gross beta activity concentration, gravimetric spike (JRC)	(1610 ± 24) mBq/L	beta activity
Gross beta activity concentration, LSC (JRC)	(1625 ± 146) mBq/L	beta activity
Gross beta activity concentration, direct evaporation (SCK CEN)	(1400 ± 80) mBq/L	beta activity
⁴⁰ K gamma-ray spectrometry (JRC)	(480 ± 160) mBq/L	beta activity
⁴⁰ K gravimetric spike (JRC)	(474 ± 10) mBq/L	beta activity
⁴⁰ K ICP-OES (JRC)	(290 ± 88) mBq/L	beta activity
⁴⁰ K by ICP-AES (SCK CEN)	(290 ± 10) mBq/L	beta activity
⁹⁰ Sr/ ⁹⁰ Y by LSC (SCK CEN)	(1020 ± 180) mBq/L	beta activity
⁹⁰ Sr/ ⁹⁰ Y gravimetric spike (JRC)	(1136 ± 16) mBq/L	beta activity

Table 7. Summary of the radionuclide-specific and gross activity measurement results for JRC-GAB2 PT reference material. The results are presented as activity concentrations [mBq/L], uncertainties are expanded uncertainties (k = 2).

It can be concluded that the mean results for ²⁴¹Am and ⁹⁰Sr/⁹⁰Y in JRC-GAB2 sample obtained by different independent measurement techniques were consistent and close to the reference value (gravimetric spiking). These results were all within the corresponding uncertainties except JRC gross alpha activity measurements. In case of the measurement results of gross beta and beta emitting radionuclides, more inconsistencies were observed. The ⁴⁰K measurement results obtained by ICP-AES and ICP-OES versus gamma-ray spectrometry and gravimetrics are seemingly different and in case of comparing ICP-AES and ICP-OES with the gravimetric value it is even not within the measurement uncertainties of the results from radiometric methods. The reason for this difference was not found yet but these inconsistencies did not jeopardize the proficiency test exercise.

The following data were used for ⁴⁰K activity calculation: ⁴⁰K specific activity considering the beta particle emission probability was (27.9 ± 0.7) Bq/g of natural K (Maulard and Osmond, 2008; NIST and CIAAW websites). The beta particle emission probability data was taken from the Decay Data Evaluation Project (Bé et al., 2010; LNHB website).

3.5 Homogeneity study

Since inhomogeneity may occur within a batch and can lead to discrepant results, it had to be demonstrated that these samples are identical within the whole batch such that each laboratory receives samples with the same parameters. Therefore, a homogeneity study between bottles was necessary to establish its contribution to the uncertainty budget of the reference values. The uncertainty budget was built with respect to all contributing parameters like weighing, volumetric measurements, counting statistics and homogeneity.

Both PT waters were homogenised for several days and bottled at JRC-Geel in November and December 2019 as described in **Chapter 3.1**. For the homogeneity study a random stratified method was used to select bottles to avoid systematic errors within the batch. Bottles were selected with the help of SNAP excel application developed at Reference Materials Unit at JRC-Geel. From each batch of water, ten bottles were randomly selected and analysed using gross activity measurements and radionuclide specific analysis of the natural origin or artificial alpha and beta emitting radionuclides used for spiking as presented in **Table 8**.

PT reference material	Parameters checked				
JRC-GAB1	Gross alpha/beta activity; uranium isotopes, stable K				
JRC-GAB2	Gross alpha/beta activity; ²⁴¹ Am, stable K and Sr				

Table 8. Parameters/analytes checked during the homogeneity and stability studies.

The homogeneity and the short term stability of the radionuclides in the matrix was evaluated using the SoftCRM software version 2.0.21 (Linsinger et al., 2001; Bonas et al., 2003) following the certification principles for reference materials as given in ISO Guide 35:2006 and ISO Guide 35:2017. According to the software, all individual results were normally and unimodally distributed. SoftCRM did not identify any measurement results 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 and use. The homogeneity study was performed by alpha-particle spectrometry after radiochemical sample preparation and ICP-OES technique for stable K and Sr measurements. 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)

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

Where:

- MS_{between} is the between bottle variance,

MS_{within} is the within bottle variance of the measurements used in the betweenbottle 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 formula (ISO 35, 2017):

$$u_{bb}^{*} = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{V_{MS_{within}}}}$$
(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 *a priori* requirement on the uncertainty from the between bottle homogeneity (u_{bb}) was set to be maximum 10 %. When waters are bottled, the main contribution to their instability could be either from the adsorption of radionuclides to the container wall or from precipitation due to chemical reactions in the sample. Measurement results for homogeneity study are presented in **Figure 2-5**.





Figure 3. The elemental potassium concentration in JRC-GAB1 PT reference material for homogeneity study. All uncertainties are combined standard uncertainties at the 1 sigma level (k=1). The blue dashed line indicates the average potassium concentration.



Figure 4. The ²⁴¹Am activity concentration in JRC-GAB2 PT reference material for homogeneity study. All uncertainties are combined standard uncertainties at the 1 sigma level (k=1). The blue solid line indicates the average total uranium activity concentration and error bars indicate the ± 1sigma (k=1).



Figure 5. The elemental potassium and strontium concentration in JRC-GAB2 PT reference material for homogeneity study. All uncertainties are combined standard uncertainties at the 1 sigma level (k=1). The blue dashed line indicates the average potassium concentration and the orange dashed line indicates the average strontium concentration.



3.6 Stability study

According to the ISO/IEC 17043:2010 and ISO 13528:2015 standards, the uncertainty from a stability study originates from two types of stability:

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

The uncertainty contribution from short- and long-term stability of the material to the uncertainty on the reference values was determined. There are chemical processes that may lead to increased uncertainty from instability apart from decay. These are adsorption of radionuclides to the sampling container material, chemical precipitation or co-precipitation of radionuclides and due to biological activities in the sample.

The short term stability was checked by measuring PT samples before and after shipment. Short term stability analysis was done when a small aliquot of PT sample was taken from the 1 L bottle and analysed using gross alpha/beta activity concentration measurements.

The first stability measurements were done already before shipping the PT reference materials and the last stability samples were measured two weeks after the result submission deadline. During long term stability testing, five bottles (n = 5) were placed in a temperature controlled climate chamber (Memmert GmbH) and kept at 4 °C and one bottle was stored at ambient temperature (around 20 °C) in a laboratory room. Long-term stability of the PT reference materials were studied by measuring activity concentrations of uranium isotopes in JRC-GAB1 and ²⁴¹Am in JRC-GAB2 PT samples, respectively. In addition, gross alpha/beta activity concentration measurements were performed on both PT samples by liquid scintillation counting according to ISO 11704:2017. The stability study covered the whole period between sample processing and result submission deadline to confirm that there was no loss of radionuclides other than from the radioactive decay during the PT exercise. The potential loss of radionuclides can be hidden by the method repeatability. Therefore when the uncertainty components from stability studies are established, one has to correct for repeatability during calculation as the SoftCRM software does. On the basis of the SoftCRM calculation data the uncertainties due to transport and storage conditions (i.e. short term and long term stability) were found to be < 7.0% as summarised in **Table 10** in Section 3.4.

3.7 Assigned values and the standard deviation for proficiency assessment

The reference gross alpha and beta activity concentration values x_{PT} were calculated from the powermoderated mean (Pommé and Keightley, 2015) of a series of reference measurements in case of JRC-GAB1 water. While the reference gross alpha and beta activity concentration values x_{PT} were calculated from the formulation (gravimetric spiking) for JRC-GAB2 water. By principle, decay correction is not possible on gross alpha/beta activities. Therefore, a reference date is not given.

The combined uncertainty $u(x_{PT})$ of the assigned reference values can be estimated as

$$u(x_{\rm PT}) = k \times \sqrt{u_{char}^2 + u_{bb}^2 + u_{sts}^2 + u_{lts}^2}$$

where

- k: coverage factor (k=1) at ~ 68% confidence interval,
- $u(x_{\rm PT})$: 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.

The relevant parameters needed for calculating scores were: the reference values/assigned values x_{PT} of the proficiency test samples, its associated combined standard uncertainty $u(x_{PT})$ and the standard deviation for proficiency assessment σ_{PT} as presented in **Table 9**. The standard deviation for proficiency assessment σ_{PT} was set to 30 % for PT reference material JRC-GAB1 and to 20 % for PT reference material JRC-GAB2, respectively. The σ_{PT} for JRC-GAB1 was chosen higher than for JRC-GAB2 since JRC-GAB1 is a natural water

with lower gross activities. JRC-GAB2 is an easier-to-measure, gravimetrically spiked PT sample, free from interferences and with elevated gross activities compared to JRC-GAB1.

Table 9. The reference gross alpha and beta activity concentration values (x_{PT}) of the REM 2019 PT reference materials and each combined uncertainty $u(x_{PT})$ and the standard deviation for proficiency assessment (σ_{PT}).

PT reference material code	Parameter	$x_{\rm PT}(u(x_{\rm PT}))$	σ_{PT} (% of x_{PT})	$u(x_{ m PT})/\sigma_{ m PT}$
	alpha	372(29) mBq/L	112 mBq/L (30%)	0.26
JUC GADI	beta	333(27) mBq/L	100 mBq/L (30%)	0.27
	alpha	731(34) mBq/L	146 mBq/L (20%)	0.23
JKC-DADZ	beta	1610(53) mBq/L	322 mBq/L (20%)	0.16

In **Table 9**, the number in parentheses is the numerical value of the combined standard uncertainty $u(x_{PT})$ referred to the corresponding last digits of the quoted value x_{PT}^4 . The uncertainties on homogeneity, stability and characterisation were taken into account in establishing the uncertainties of the assigned reference values as presented in **Table 10**.

Table 10. Summary of the reference values and their uncertainty components (in brackets from alpha spectrometry).

PT reference material code	Parameter	U _{char}	U hom	u _{sts} +u _{lts} t=30 weeks	u (X _{PT})
	Gross alpha	0.031	0.02	0.069 (0.018)	0.078
JUC GADI	Gross beta	0.062	0.01	0.053	0.082
	Gross alpha	0.007	0.02	0.041 (0.022)	0.046
JKC-UADZ	Gross beta	0.007	0.004	0.032	0.033

To be more cautious with the uncertainty on the reference values we decided to use the higher uncertainty values from the stability study obtained by LSC gross alpha/beta measurements instead of the values from radionuclide specific measurements (see in brackets in **Table 10**).

3.8 Metrological traceability

Metrological traceability of the measurement values were established via a documented unbroken chain of calibrations and/or using certified reference materials with stated uncertainties on their property values characterised by metrology institutes (Czech Metrology Institute, National Physical Laboratory-UK). Furthermore, JRC participated in BIPM (International Bureau of Weights and Measures) key comparisons (K) and supplementary (S) comparisons. ²⁴¹Am and ⁹⁰Sr solutions were used from those exercises for certain measurements:

- CCRI(II)-K2.Sr-90 solution,
- CCRI(II)-K2.Am-241 solution,
- CCRI(II)-S3: Radionuclide (²⁴¹Am, ^{239,240}Pu, ²³⁸Pu, ²³⁸Pu, ²³⁸U, ²³⁴U, ²³⁵U, ²³²Th, ²³⁰Th, ²²⁸Th, ²²⁸Ra, ¹³⁷Cs, ²¹⁰Pb, ⁹⁰Sr and ⁴⁰K) activity measurements in reference materials shellfish.

Another important milestone from a metrological point of view is that the JRC officially re-joined EURAMET which was a pending issue since the reorganisation of the JRC in 2016.

⁴ Evaluation of measurement data — Guide to the expression of uncertainty in measurement, BIPM, 2008. <u>https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf</u>

4 Participants' results, scores and evaluation of results

In total, 154 participants of which 140 from the European Union Member States registered for this PT. From the 154 participants, 145 submitted at least one measurement result which totalled 709 individual measurement results. This chapter presents the evaluation of the participants' performance using their calculated performance scores.

The participants were requested to submit their results together with their combined standard uncertainties indicating the coverage factor (k) they used. To be able to compare the submitted results adequately, the organiser recalculated (when necessary) the reported uncertainties so that the same coverage factor is employed in all the graphs of this report.

We have to note that for JRC-GAB1 there were 7 and 11 "less-than" values for gross alpha and gross beta activity concentration, respectively. No less-than values were reported for sample JRC-GAB2. For "less-than" values, no scores (percentage deviation, z-score and zeta-score) could be calculated.

ISO 13238:2015 (**Annex 10**) requires the uncertainty of the assigned value $u(x_{PT})$ to be smaller than $0.3 \sigma_{PT}$ for enabling the organiser to express the laboratory performance in terms of z_i^- and $zeta_i^-$ scores. This was the case for all four σ_{PT} of this PT (see last column of **Table 9**). The detailed calculation of performance evaluation scores including formulae is presented in **Annex 11**.

The z-score divides each participant's deviation from the assigned value with the standard deviation of the proficiency test assessment (σ_{PT}).

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

The interpretation of the z_i -score and $zeta_i$ -score was done according to ISO 13528:2015. The following scores and colour codes are used in **Figure 6** and **Table 21-23** in **Annex 10** (in parenthesis the ISO/IEC 17043:2010 synonym is given):

- |score| ≤ 2 acceptable (or satisfactory) performance (green),
- 2 < |score| < 3 warning (or questionable) signal (yellow),
- |score| ≥ 3 unacceptable (or unsatisfactory) performance (red).

For the percentage deviation a value is acceptable if the reported value is within $\pm \sigma_{PT}$, i.e within $\pm 30\%$ of the assigned value for JRC-GAB1 and within 20% of the assigned value for JRC-GAB2.

The gross alpha and beta activity concentrations x_i in mBq/L are plotted in ascending order in **Figure 6**. (JRC-GAB1 gross alpha), **Figure 7** (JRC-GAB1 gross beta), **Figure 8** (JRC-GAB2 gross alpha) and **Figure 9** (JRC-GAB2 gross beta). The uncertainties reported by the participants are presented as expanded uncertainties $U(x_i)$, with k = 2.

The solid red line on the S-plots indicates the reference gross alpha or beta activity concentration (assigned value, x_{PT}). The dashed red lines show the expanded uncertainty $U(x_{PT})$ (with k = 2) of the reference value, while the blue short-dashed lines represent the acceptance range $x_{PT} \pm 2 \sigma_{PT}$ for z_i -scores.

Results without error bars on the S-plots (symbols at the right side of each graphs) represent results reported by the participants as "less-than" values.



Figure 6. The gross alpha activity concentration measurement results for JRC-GAB1 PT reference material, as reported by the participants, x_i , and their expanded uncertainty $U(x_i)$, k = 2. Solid red line: reference value (x_{PT}). Red dashed lines: assigned range ($x_{PT} \pm U(x_{PT})$, k = 2). Blue dashed lines: $x_{PT} \pm 2 \sigma_{PT}$ acceptance range.



Figure 7. The gross beta activity concentration measurement results for JRC-GAB1 PT reference material, as reported by the participants, x_i , and their expanded uncertainty $U(x_i)$, k = 2. Solid red line: reference value (x_{PT}). Red dashed lines: assigned range ($x_{PT} \pm U(x_{PT})$, k = 2). Blue dashed lines: $x_{PT} \pm 2 \sigma_{PT}$ acceptance range.

Laboratory code



Figure 8. The gross alpha activity concentration measurement results for JRC-GAB2 PT reference material, as reported by the participants, x_i , and their expanded uncertainty $U(x_i)$, k = 2. Solid red line: reference value (x_{PT}). Red dashed lines: assigned range ($x_{PT} \pm U(x_{PT})$, k = 2). Blue dashed lines: $x_{PT} \pm 2 \sigma_{PT}$ acceptance range.



Figure 9. The gross beta activity concentration measurement results for JRC-GAB2 PT reference material, as reported by the participants, x_i , and their expanded uncertainty $U(x_i)$, k = 2. Solid red line: reference value (x_{PT}). Red dashed lines: assigned range ($x_{PT} \pm U(x_{PT})$, k = 2). Blue dashed lines: $x_{PT} \pm 2 \sigma_{PT}$ acceptance range.

Laboratory code

The overview plots of percentage deviation, zeta-and z-scores in percentage (%) and number of laboratories with satisfactory, questionable and unsatisfactory measurement results for both water samples and the gross activity concentrations are presented in **Figure 10-13**.





Figure 11. Overview of percentage deviation, zeta-and z-scores in percentage (%) and number of laboratories with **satisfactory**, questionable and **unsatisfactory** measurement results for JRC-GAB1 PT reference material gross beta activity concentration.



Figure 12. Overview of percentage deviation, zeta-and z-scores in percentage (%) and number of laboratories with **satisfactory**, **questionable** and **unsatisfactory** measurement results for JRC-GAB2 PT reference material gross alpha activity concentration.







We observed that many reported results deviate by several orders of magnitude from the reference values. A general conclusion for all four parameters in this exercise is that only around 50% of the submitted results were satisfactory for percentage deviation and zeta score. The success rate for z-score was much higher thanks to the interpretation of this score, as it extends by two times the standard deviation for proficiency tests, i.e. "acceptance range". In the context of reducing the number of blunders or grand errors⁵ in the EU, it is useful to compare the ratio of maximum to minimum reported gross activity concentrations as presented in **Table 11**. The number of satisfactory percentage deviation (D%) and zeta scores together with the number of laboratories are collected in **Table 12**.

Table 11. Ratio of the reported maximum to minimum gross activity concentrations. (Not including results reported as detection limits)

	A _{max} / A _{min}			
rarameter	JRC-GAB1	JRC-GAB2		
Gross alpha activity	1.96×10 ⁷	7.30×10 ³		
Gross beta activity	4.52×10 ⁷	5.11×10 ⁶		

⁵ We define a blunder or grand error as a single error that generates an erroneous result by at least a factor 10 and can be to write mBq instead of Bq or to lose a digit like for example writing 100 instead of 1000.

Number of parameters with acceptable results	Percentage deviation (D%): Number of laboratories	Zeta (ζ)-score: Number of laboratories		
4	26 (17.9%)	22 (15.2%)		
3	36 (24.8%)	36 (24.8%)		
2	45 (31.0%)	37 (25.5%)		
1	24 (16.6%)	28 (19.3%)		
0	14 (9.7%)	22 (15.2%)		

Table 12. Number of laboratories with a certain number of parameters with acceptable results. The values in parentheses are percentage values of the total number of submitted results.

There were 131 participants out of 145 (about 90% of the participants) that managed to report at least one acceptable result. There were still 14 laboratories, which represent almost 10% of participants, that did not succeed to submit any acceptable results on the basis of percentage deviation and zeta score. There were 62 laboratories (43% of the participants) that reported minimum three acceptable results for percentage deviation. When the zeta score is considered, 56 laboratories (40% of the participants) reported minimum three acceptable results.

Before starting a gross alpha/beta activity measurement in water the total dissolved solid content has to be determined in order to verify the water sample falls within the scope of the corresponding ISO standard/method. Therefore, the TDS content of the two PT waters had to be also determined by the participants. The indicative values were determined by JRC-Geel in case of JRC-GAB1 PT reference material experimentally. For JRC-GAB2 PT reference material it was possible to calculate TDS from weighing of inorganic salts and the water used for formulation (see Chapter 3.1). However, we also measured the total dissolved solid (TDS) content of both PT reference material by direct evaporation of 50 mL water sample and thermal treatment of the dried residue at 180 °C until constant mass. The JRC-Geel experimental results and the participants' values are presented in **Figure 14** and summarised in **Table 13**.

 Table 13. Indicative values of the total dissolved solid (TDS) content of the REM2019 PT samples.

	Indicative values (mg L⁻¹); at <i>k=1</i>	Reported total dissolved solids (mg L^{-1})					
		Average	Median	Standard deviation	Minimum	Maximum	
JRC-GAB1	966 ± 27	1039	978	940	0.98	8783	
JRC-GAB2	356 ± 20	487	335	935	0.27	10000	_

Considering the participants' median TDS results it was close to the JRC-Geel indicative values which shows that the majority of participants determined TDS values relatively correctly. However, a large spread of data can be observed covering a range of several orders of magnitude. The reasons for this can be linked to (i) issues with the procedure, (ii) balance calibration (iii) improper weighing or (iv) incorrect result submission.



Figure 14. The laboratory results for total dissolved solid content for JRC-GAB1 (upper graph) and JRC-GAB2 (lower graph). The solid red lines: the indicative values; the dashed blue lines: standard uncertainties (± u at k = 1).

4.1 Youden plots

Since measurement results were obtained by analysing two similar proficiency test samples, Youden Plot can be used as a graphical approach to give information on repeatability and identify random/systematic errors as explained by Youden (1959) and ISO 13528 (2015). A scatter plot is drawn in which the x-axis shows the z-scores or D% scores of the first sample and the y-axis shows the same score for the other sample.

The interpretation of the Youden Plot (see **Figure 15**) is the following. The results can be grouped in four quadrants. When the variation in results is dominated by random errors, then the points are randomly distributed in all quadrants with approximately the same number of results in each quadrant. When systematic errors are significantly larger than random errors, then the points occur primarily in the upper right and lower left quadrants.

The distance of a point from the 45° line (blue dashed line) is proportional to the contribution of random error on the corresponding laboratory's results (purple arrow). The distance of a point from the zero points (intersection of the axes) on the 45° line is proportional to the laboratory's systematic error (orange arrow). Points in the far upper left and lower right quadrants show poor repeatability (grey circles). The acceptable results are distributed within the green box (|z-score| ≤ 2 ; |D%| $\leq 30\%$ and 20% for JRC-GAB1 and JRC-GAB2, respectively).





Two types of Youden-plots were created for gross alpha and gross beta activities separately. For the first Youden-plots pairs of z-score values from the two PT samples were used (**Figure 16**), while for the other ones pairs of percentage difference scores were used (**Figure 17**). Every plot contains information on the total number of pairs of scores (n_{total}) and the number of acceptable scores ($n_{acceptable}$) and their percentage value in parenthesis.



Figure 16. Youden Plot of z-scores for REM2019 PT gross alpha and gross beta activity concentration measurement results. The acceptable results are spread within the green box.



Figure 17. Youden Plot of percentage deviation (D%) for REM2019 PT gross alpha and gross beta activity concentration measurement results. The acceptable results are spread within the green box.

As seen in **Figure 16**, the majority of data is within the acceptance range in the Youden plots using z-scores. However, for the percentage deviation (**Figure 17**) the trend is the opposite, around 60%-70% of the results are outside the acceptance range.
4.2 Sorted results

Results were also sorted on the basis of counting technique, use of written standard, ISO/IEC 17025:2017 (or any predecessor, which is labelled as ISO 17025) accreditation, radionuclides used for efficiency calibration and the time delay between sample preparation and counting which presented in this chapter. The first parameter to analyse is the use of written ISO standards in combination with the counting techniques (liquid scintillation counter, proportional counter, solid-state scintillator) as shown in **Figure 18-20**. The percentage reported acceptable percentage deviation scores, zeta scores and the average bias from the reference values are presented comparing the non-sorted global pool of reported data (all data) to the sorted results as a function of counting techniques where all data is considered and in the next columns with the counting technique and use of ISO standard.

Figure 18. Sorted results for JRC-GAB1 PT reference material gross alpha and gross beta activity concentration measurements as a function of detection technique and use of ISO method. Percentage of acceptable results for percentage deviation, zeta score and average bias from reference values are presented.





GAB1 Gross beta





As seen, regardless the counting techniques there is a slightly improved performance in all three parameters when written ISO standard methods were followed. In case of JRC-GAB1 gross beta activity measurement the LSC technique was seriously underperforming. It might happen that this was due to calibration-settings issues rather than interference from sample composition. The reason for this bias and performance is still under investigation. Another interesting point to scrutinise from quality control point of view is ISO 17025 accreditation. In the next section measurement data is presented in S-plots separating the results of non-accredited laboratories from the results of accredited laboratories (**Figure 20-24**). On the bar charts (**Figure 24-28**), the percentage reported acceptable percentage deviation scores, zeta scores and the average bias from the results are presented comparing the non-sorted global pool of reported data (all data) to the results with- and without accreditation.



Figure 20. Sorted results for JRC-GAB1 PT reference material gross alpha activity concentration measurements as a function of ISO 17025 accreditation.



Figure 21. Sorted results for JRC-GAB1 PT reference material gross beta activity concentration measurements as a function of ISO 17025 accreditation.



Figure 22. Sorted results for JRC-GAB2 PT reference material gross alpha activity concentration measurements as a function of ISO 17025 accreditation.



Figure 23. Sorted results for JRC-GAB2 PT reference material gross beta activity concentration measurements as a function of ISO 17025 accreditation.





Figure 25. Comparison of ISO 17025 accredited and non-accredited laboratories for JRC-GAB1 PT reference material gross beta activity concentration measurements. Percentage of acceptable results of D%, zeta score and average bias from reference values.



GAB1 Gross beta

Figure 26. Comparison of ISO 17025 accredited and non-accredited laboratories for JRC-GAB2 PT reference material gross alpha activity concentration measurements. Percentage of acceptable results of D%, zeta score and average bias from reference values.



Figure 27. Comparison of ISO 17025 accredited and non-accredited laboratories for JRC-GAB2 PT reference material gross beta activity concentration measurements. Percentage of acceptable results of D%, zeta score and average bias from reference values.



Results sorted on the basis of radionuclides used for alpha and beta counting efficiency calibration are presented in **Figure 28-31**.



Figure 28. Sorted results for JRC-GAB1 PT reference material gross alpha activity concentration measurements as a function of the radionuclides used for alpha counting efficiency calibration.



Figure 29. Sorted results for JRC-GAB2 PT reference material gross alpha activity concentration measurements as a function of the radionuclides used for alpha counting efficiency calibration.



Figure 30. Sorted results for JRC-GAB1 PT reference material gross beta activity concentration measurements as a function of the radionuclides used for alpha counting efficiency calibration.



Figure 31. Sorted results for JRC-GAB2 PT reference material gross beta activity concentration measurements as a function of the radionuclides used for alpha counting efficiency calibration.

The last parameter to analyse is the delay between sample preparation and starting of a gross measurement. This delay can cause significant positive bias in case of waters with elevated ²²⁶Ra activity concentration. The decay products of ²²⁶Ra can build-up in the counting source which can lead to an overestimation of the true gross alpha/beta activity concentration in the sample. The opposite can happen if a particular water sample contains ²²⁴Ra which can be missed due to the delay of starting gross measurements. The reported gross alpha and gross beta activity concentrations are plotted against the elapsed time between sample preparation and starting measurement in **Figure 32-34**.



Figure 32. Sorted results for JRC-GAB1 PT reference material gross alpha and beta activity concentration measurements as a function of time delay between sample preparation and measurement.

Elapsed time between sample preparation and analysis (hours)



Elapsed time between sample preparation and analysis (hours)



Figure 33. Sorted results for JRC-GAB2 PT reference material gross alpha and beta activity concentration measurements as a function of time delay between sample preparation and measurement.



Elapsed time between sample preparation and analysis (hours)

We did not observe any trend or shift in the reported results as a function of delays which confirms that in these particular PT reference materials the time delay did not influence the measurement results.

4.3 PomPlots

The PomPlot is an intuitive graphical method that is used for producing an overview of the participants' results (Spasova et al., 2007). Its initial use was in the metrology field as a tool to analyse results from BIPM's key comparison exercises but its use is spreading to many other fields. It displays the relative deviations, D/MAD (absolute deviation divided by the median absolute deviation) of the individual results from the reference value on the horizontal axis and relative uncertainties (u/MAD) on the vertical axis. Red square indicates the reference value; green, blue and red solid lines represent ζ -scores = 1, 2 and 3, respectively in **Figure 34-38**. For both axes, the variables are expressed as multiples of MAD, which is the median of the absolute deviation from the reference value. A PomPlot works best when the reference value is established via consensus from the participants' results (like for the BIPM's key comparisons). Note that since the participants' data is used to establish the *MAD*, it may happen that data-points are located outside a certain zeta score value on the PomPlot while they fall within the same value when reference value is established by other approach than consensus value. The detailed PomPlot interpretation is presented in **Annex 12**.



Figure 34. PomPlot of the JRC-GAB1 PT reference material gross alpha activity data.

Figure 35. PomPlot of the JRC-GAB1 PT reference material ISO 11704:2017 gross alpha activity data sorted on the basis of accreditation.





Figure 36. PomPlot of the JRC-GAB1 PT reference material gross beta activity data non-sorted.

Figure 37. PomPlot of the JRC-GAB1 PT reference material ISO 11704:2017 gross beta activity data sorted on the basis of accreditation.



4.4 Comparison of REM2019 PT and REM2012 ILC

We tried to identify if there were any improvements or changes in the performance of the participants that took part in both the REM2012 ILC and REM2019 PT. Some general information of these two PTs are presented in **Table 14**.

Table 14. Summary on REM2019 PT and REM2012 ILC.					
PT identifier	Number of registered participants	Number of participants reported results	Type of PT reference materials	Volume of PT reference materials (produced units)	
REM 2012	76 (all from EU)	71 (93%)	2 natural, 1 spiked	500-700 L (1883 units)	
REM 2019	154 (140 from EU)	145 (95%)	1 natural, 1 spiked	500 L (860 units)	

There were 54 common registered participants from both PTs of which 51 submitted results at both occasions. The common score for comparison was percentage difference (D%) because this was the only score used in REM2012 ILC. The percentage of satisfactory results per PT sample and parameters were collected in Table 15.

	REM2012 PT			REM2019 PT		
Parameter	Water A (nat.)	Water B (nat.)	Water C (spiked QC)	GAB1 (nat.)	GAB2 (spiked QC)	
Gross alpha activity	36%	39%	63%	51%	55%	
Gross beta activity	45%	27%	61%	63%	51%	

For 34 participants the performance improved, while for 12 participants the performance became worse and for 5 participants stayed unchanged.

Only 28 participants submitted results for all 10 parameters for both exercises and there was not a single participant that had 100% success rate. However, 3 participants reported 9 acceptable results and further 6 participants reported 8 acceptable results.

5 Information on the participating laboratories: organisational and technical details

The 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 (iii) involvement in standardisation processes. The participants were also 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. The 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 145 participants.

It has to be noted that for some questions more replies can be counted than the total number of participants which is due to the possibility of multiple-choice selection options and in certain cases laboratories also replied to questions non-applicable to them.

Information on accreditation, application of documented standards:

- 88 out of 145 have ISO 17025 accreditation
- 54 are involved in international/national standardisation processes, and from the non-involved organisations **91 (i.e. all) would like to be involved**,

Note: During the follow-up workshop it was explained how to take the first steps to join standardisation process.

- 91 out of 145 performed their analysis according to an ISO standard.
- 31 followed other standard methods (e.g. EPA, ASTM, national or in-house developed standards)

Type of laboratory:

- Measurements of radioactivity in the environment: 125 participants
- Research and development: 40 participants
- Private commercial company: 12 participants
- Monitoring of nuclear facilities: 33
- Water supply company: 4 participants
- Other: 8

Laboratory working according to a quality management system?

139 organisations work according to a quality management system. The most commonly used ones are: ISO 17025, ISO 9000 series, ISO 14000, ISO 14001, EN 45000 series, and some reported "internal".

How long is gross alpha/beta activity analysis performed routinely at your organisation (in years)?

Laboratories have been dealing with gross alpha/beta activity in water analysis between 0 and 60 years with an average of 21 years.

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

- < 50: 25 participants</p>
- 50-100: 36 participants
- 100-250: 29 participants
- 250-500: 19 participants
- 500-1000: 26 participants
- >1000: 10 participants

From minimum 1 measurement up to 8000 measurements per year (in total approximately 70540 gross alpha/beta measurements are performed at the participants' laboratories).

How many individual gross alpha/beta samples can you measure in the same time?

Laboratories can measure between 1 and 360 samples in the same time with a median of 48 samples.

The typical test sample volume needed for a single analysis (in mL)

Minimum sample volume was 8 mL and maximum 5000 mL water sample with a median of 250 mL.

Acidifying the sample is part of the procedure

113 participants said acidification is part of the routine procedure and 32 replied no.

Having a procedure to treat hygroscopic residues

40 participant have procedure, while 71 participant indicated that they do not have procedure to treat hygroscopic residues. 32 replied not applicable.

Applying quenching correction

17 participant applied, while 65 participant did not apply quench correction. 63 replied not applicable.

Correction for surface density of the prepared source

50 participant made correction for surface density, 64 participant did not make correction. 31 replied not applicable.

The time delay between finishing sample preparation and starting gross measurements

The delay varied between 0 and 3001 hours with an average 66 hours.

Membership in analytical networks.

- National/regional analytical network: 93 participants
- IAEA-ALMERA network: 48 participants
- European NORM network: 5 participants
- Other networks (listed below): 12 participants
- PROCORAD (Association for the Promotion of Quality Control in Radiotoxicological Analysis)
- RIMNET (Radioactive Incident Monitoring Network, UK)
- UNE ASOCIACIÓN ESPAÑOLA DE NORMALIZACIÓN
- CTBTO
- EDF
- University network
- Not specified
- Not members of networks: 8 participants

How did you learn about this PT?

- Nominated by national authorities/contact points: 82 participants
- Invited by the JRC: 50 participants
- Announced by the IAEA: 23 participants
- from the JRC website (<u>https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests</u>): 11 participants
- Some laboratories got information about this PT from multiple channels.

5.1 Methods used by the participating laboratories

Details of the methods used by the participating laboratories including sample preparation and efficiency calibration approaches, background-blank sample preparation are given in the next paragraphs.

Sample preparation and measurement techniques

- Direct measurement after evaporation: 107 participants
- Thermal pre-concentration for liquid scintillation counting: 26 participants
- Co-precipitation: 15 participants
- Direct measurement with liquid scintillation counting: 12 participants
- Other (not specified): 5 participants.

Note: 18 participants indicated using multiple sample preparation methods.

- gas-flow proportional counter: 93 participants
- liquid scintillation counter: 39 participants
- solid-stated scintillation counter: 28 participants
- Other (not specified): 29 participants.

Note: 20 participants indicated using multiple counting techniques.

Efficiency calibration approaches

- Majority of participants followed efficiency calibration procedures described in standards
- Radionuclides used for alpha efficiency calibration:
 ²⁰⁹Po, ²¹⁰Po, ²²⁶Ra, ²³⁶U, U_{nat}, ²³⁹Pu, ²⁴¹Am, ²⁴³Am, ²⁴⁴Cm, ²⁴²Pu
- Radionuclides used for beta calibration: ³⁶Cl, ³⁵Cl(?), ⁴⁰K, ⁹⁰Sr, ¹³⁷Cs, ²¹⁰Bi, ²¹⁰Pb, U_{nat}
- TDCR approach for LSC was also used

Detection limits

Comparing the reported gross alpha/beta detection limits with the detection limits given in the E-DWD it can be noted that there are laboratories not complying with that requirements **Table 16**. There were 29 and 5 participants that reported higher limit of detections than what is stated in the E-DWD for gross alpha and gross beta activity concentration, respectively.

Table 16. Limit of detection of gross alpha/beta activity concentrations reported by the participant laboratories in mBq L⁻¹

Description	Gross alpha activity concentration	Gross beta activity concentration
Reported limit of detection range (The numbers in brackets are probable blunders)	1– 1000 (0-20000)	1 – 790 (0-54000)
Median	24	50
Average	43 (without blunders)	90 (without blunders)
Limit of detection (mBq L ⁻¹) from the Euratom Drinking Water Directive (EC, 2013)	40	400

Background-blank preparations

There were six different background-blank sample preparation approaches reported as listed below. In addition to that, a few participants mentioned that they do not prepare any blank or background samples.

- pure demineralised water
- acidified demineralised water
- demineralised water and adding salts (CaSO₄)
- demineralised water with same water:cocktail ratio as samples
- empty planchet
- empty detector
- no blank at all: 6 participants

5.2 Participants' feedback

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 evaluation scores (average score given by the participants was 8.9 out of 10) which is positive but there is still room for improvement. The proposals to improve the organisation of a gross activity-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

- Samples are too much acidified

Organiser's comment: samples were acidified to comply with the ISO 5667-3:2018(EN) A.5 section saying acidify to pH<2 with HNO_3 but do not acidify if sample is immediately processed after sampling. Since samples were stored for some weeks, sample preservation was needed.

- Covid-19: not enough time to perform the analysis

Organiser's comment: we continuously evaluated the Covid situation in the course of the reporting phase and decided to extend the reporting deadline by three months. We believe it was sufficiently enough time for doing the analysis.

- Some initial problems with the reporting software

Organiser's comment: it was solved very soon after notifying us.

- Concentration of the samples GAB1 and GAB2 is not typical for drinking water

Organiser's comment: it is difficult to find or prepare a fit for all/typical average drinking water sample since the geological/hydrogeological property of aquifers are very diverse in Europe even within a country. However, we aimed to provide waters with moderate but slightly elevated salinity (Low-intermediate mineral content: 50-1500 mg/L).

- Residue from sample JRC-GAB2 has an elastic consistency, difficult to spread in the counting tray

Organiser's comment: it might be also due to the method applied.

- GAB2 gross beta gave not repeatable results, despite several tests
- Gross beta is misleading: not clear if low-energy betas (⁴⁰K, ³H) are included or not

Organiser's comment: it is also one of the pitfalls of gross methods. Certain methods do not include some of the radionuclides (e.g. ISO 10704 co-precipitation approach excludes ⁴⁰K during the sample preparation), therefore they are in principle already biased. Also, consider the difference between residual beta and gross beta activity concentration.

- Provide questionnaire for different measurement procedures

Organiser's comment: it was limitations of the on-line reporting tool. Hopefully, after the upgrade of the reporting system, it will be also possible.

- Questionnaire and reporting files sometimes unclear

Organiser's comment: we try to improve the clarity of our documents and tools. However, the menu and the text on the on-line tool is given by the software developers we cannot change it.

- Announcements for sample shipping was too immediate

Organiser's comment: we believe it was properly communicated and in a timely manner. Participants were informed two times about the delivery dates: 1) on 22 November 2019 (i.e. few weeks before shipment took place). 2) on 13 January 2020 a reminder was sent to the participants that we started shipping the samples. We also indicated the shipment dates in our email when laboratories were first contacted.

Suggestions

- Provide non-acidified samples

Organiser's comment: see our comments on acidification in the previous section (Remarks). We had to do proper sample preservation to comply with the ISO 5667-3:2018(EN).

- Paper copies for reporting template/questionnaire

Organiser's comment: We prefer going paperless to save resources and to keep track on the date of reporting. Papers might be lost during the postal service or mislaid at the participant laboratory.

- Quantity of sample should be larger

Organiser's comment: considering the replies from the questionnaire, we can conclude that sample quantity was appropriate. The typical sample volume needed for a single measurement was 250 mL (median value).

- Send a single bottle package

Organiser's comment: Package containing more bottles are more economical options saving more CO_2 and money.

- Add questions about number of replicates, counting statistics

Organiser's comment: we consider this comment. However, we wanted to keep the questionnaire as simple and short as possible. During the REM 2012 ILC exercise we asked for more details about counting statistics and replicates, detection limit calculations and participants found that too heavy and exhausting to fill in.

- Organise this PT more frequently

Organiser's comment: we would like to organise PTs more frequently and keep some in the regular PT scheme but due to the current staff limitations and available resources JRC-Geel can organise maximum one PT per year in the field of environmental radioactivity which includes many different matrices and radionuclides.

Further comments, appreciations

- General feedback from participants: it was a well-organized PT
- Overall satisfaction score: 8.9 out of 10,
- Flexibility due to Covid situation was appreciated (submission deadline extension twice)
- PT was useful regarding the QA system
- PT was used for method validation
- Educating PT exercise for new-in-the-field laboratories
- PT helped to identify errors linked to method and calculation,
- Concentrations were within normal operation range
- Sample quantity was sufficient for doing multiple analyses
- Quick email answers/immediate communication was highly appreciated
- Instructions were clear enough,
- Delivery and packaging were fast and very robust
- All the needed information was provided in advance
- The documents were of high quality, clear and very detailed.
- Many participants are interested in future JRC PTs

5.3 Follow-up workshop

A virtual-workshop dedicated to the REM2019 proficiency test was organised by the JRC G.2 unit Radionuclide Metrology team between 4-6 May 2021. The objective of this workshop was to gather REM 2019 participants and discuss related hot topics with the aim to improve the level of radiation monitoring in Europe to ensure the health and safety of citizens.

This virtual event attracted 140 registered participants from 26 European countries representing environmental radioactivity monitoring laboratories, metrology institutes, universities, regulatory bodies, Euratom Article 35-36 experts, colleagues from JRC Dir.F and Directorate General for Energy (DG-ENER). During the three-day event, presentations were given by invited speakers and also participants had the opportunity to present their work on linked to the following topics:

- REM2019 Proficiency Test evaluation
- Quality assurance, proficiency tests in Europe
- Best practice, method harmonisation and standardisation efforts
- General staff issues in Europe: resources, trainings, "next-generation"
- JRC future policy support activities

In addition to the aforementioned topics, there were very constructive discussions during the workshop concerning education of future generation, retirement of proficient analysts/expert needs of hands-on trainings to improve proficiency. A question could be also raised: Are the current resources (human and financial) sufficient to operate environmental monitoring laboratories or are they at risk of being closed or operate in a way that jeopardize health & safety of the citizens? Usually governments/public need services of the laboratories urgently in case of emergency but under normal conditions they are "invisible"/unrecognised. After the discussions the following requests/conclusions could be drawn linked to proficiency tests, gross methods and radionuclide specific methods:

- the gross alpha and beta methods should be harmonised in Europe
- Collect information from PT participants on rapid radionuclide specific methods compare them to gross methods. Select "best ones" in terms of cost, time and complexity.
- JRC should consider including indicative dose (ID) calculation as part of the PT.
- A list of PT providers in Europe would be useful.

JRC's comment: there are websites of PTs on the internet (e.g. EPTIS website) as highlighted by the workshop participants. The participants also named some PT providers from Europe. A document with these PT providers were drafted and circulated to the workshop participants immediately after the workshop. This list is planned to be made available on the REMON website⁶ where the JRC PTs are summarized.

A discussion was about the requirements on how to give characteristic limits (decision threshold, detection limit and limits of the coverage interval) in measurement reports. The ISO standards dealing with radioactivity in water measurements recommend the use of ISO 11929:2019 standard which according to many participants does not reflect the real situation, the formulae are too complicated and taking a lot of space in a measurement report. Clients have problems already to understand the meaning of uncertainty and the situation gets more complicated (even for the laboratory personnel) with the introduction of characteristic limits following the ISO 11929:2019 standard.

At last but not least, the participants emphasised the importance of JRC as an impartial and competent organisation in the field of proficiency testing and its efforts were appreciated. Considering the policy impact and the importance of proficiency tests from quality assurance point of view a strong request was expressed by both the "nuclear and non-nuclear" Member States to continue this important task in future.

The JRC G.2 Unit-Radionuclide Metrology team was seeking for more feedback from Member States' authorities and REM PT participants in form of letter of support in which they could specify how they see these services and if they are important to authorities, nominating bodies and the radioactivity monitoring laboratories in the corresponding country. This feedback was needed to support JRC G.2 unit's activity within JRC evaluation of the JRC activities within the framework programme Horizon-2020. Another reason for seeking for written support is linked to the new budget cuts of JRC's Euratom programme and therefore many activities are at risk of being stopped. A continuation of the REM proficiency tests depends ultimately on how member states value this work.

5.4 Reported impacts of this PT

Participants could already use the PT results or the PT reference materials to improve the quality and reliability of their analytical results. Many participants reported that they could use the REM2019 PT preliminary report (which included the reference values and laboratory scores) to support their quality management system and accreditation efforts by showing their scores during external accreditation assessments. The main impacts of this PT were linked to the followings:

- PT was important regarding the quality system and the accreditation procedure (i.e. to maintain or to gain accreditation),
- Method validation,
- Useful learning exercise for participants that are new in the field,
- Helped to highlight calculation or method related errors.

5.4.1 A detailed impact case

A concrete detailed impact case including corrective actions as a positive example is given below. The PT organisers would like to thank the anonymous participant⁷ for this very important and thorough description.

A participant from an EU member state developed and validated a new method for the measurement of 'Gross Alpha and Beta Activity Concentration in Non-Saline Water' by a liquid scintillation counting method based on ISO 11704:2015. The method was routinely used for the analysis of drinking water under the national monitoring programme for radioactivity in drinking water since April 2017. By the first quarter of 2020, over 650 water samples had been analysed for gross alpha/beta activity where none of the results exceeded the corresponding gross activity screening levels, contrary to results from previous surveys.

In early 2020, an error was identified in the calculation of gross alpha/beta activity as a result of participation in the European Commission's Joint Research Centre REM2019 PT. All historical data back to 2017 was reviewed and the method was revalidated to confirm that the new method complied with the analytical

⁶ <u>https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests</u>

⁷ The identity of the participant is undisclosed for confidentiality reasons.

performance characteristics laid down in Annex III/Part 3 of the Euratom Drinking Water Directive (Council Directive 2013/51).

Besides the JRC REM2019 PT, this laboratory participated in another PT in the same period. The results submitted by the participant for the JRC PT and the other PT were both evaluated as 'satisfactory' for gross alpha and gross beta measurements.

All stakeholders (authorities, water supplies and other clients) were informed about the calculation error and the previous reports were withdrawn. The participant requested the relevant local authorities and water supplies to resample sources with elevated values to re-measure them according to the new validated method.

The participant re-issued all previous reports for the period of 2017-2020 and the national monitoring programme for radioactivity in drinking water resumed in September 2020 employing the new gross alpha/beta activity measurement method.

6 Best practices and recommended methods

Taking the percentage deviation (D%) and zeta scores into consideration separately 26 and 22 laboratories could submit acceptable results for all four parameters, respectively. However, there are only 14 participants that have acceptable results when combining the percentage deviation and zeta scores of all four parameters.

In case of evaluating the joint performances from both REM2012 and REM2019 proficiency tests (in total ten parameters reported) only 3 laboratories showed outstanding performance with reporting 9 acceptable results out of 10 (**Table 17**). There were another 6 laboratories that managed to submit 8 acceptable results.

REM2019 PT ID	Number of acceptable results (maximum 10)	Number of acceptable zeta scores (maximum 4) (only for REM2019)
17434	9	4
17503	9	4
17568	9	3
17354	8	2
17357	8	4
17394	8	3
17409	8	4
17431	8	3
17538	8	1

 Table 17. Best performing laboratories in the REM2012ILC and REM2019 PT joint evaluation on the basis of percentage differences and zeta scores.

The data from the best-performing 14 laboratories from the REM2019 PT and another 4 best performing laboratories (bold in **Table 17**) from the combined REM2012-REM2019 PTs are presented in **Table 18 and Table 19**, respectively. These data were further analysed in order to find parameters or conditions that lead to outstanding performance. We have to note that these 14 laboratories might not have always provided the most accurate results (i.e. closest to the reference values) but they were consistent, always within the standard deviations of the proficiency test and provided realistic uncertainties as well.

Lab ID	Accredited (17025)	Standard technique	Sample preparation	Measurement technique	Alpha calibration	Beta calibration
17357	a) yes	ISO 10704	Direct measurement after evaporation, coprecipitation	a) gas-flow proportional counter, c) solid-state scintillation counter	²³⁹ Pu	⁹⁰ Sr
17373	b) no	Other	Direct measurement after evaporation, coprecipitation	a) gas-flow proportional counter, c) solid-state scintillation counter	²⁴¹ Am	⁹⁰ Sr
17379	b) no	ISO 10704	Direct measurement after evaporation	a) gas-flow proportional counter	²⁴¹ Am	⁹⁰ Sr
17385	a) yes	ISO 11704	Thermal preconcentration for liquid scintillation counting	b) liquid scintillation counter	²³⁶ U	⁴⁰ K
17409	a) yes	ISO 11704	Thermal preconcentration for liquid scintillation counting	b) liquid scintillation counter	²⁴¹ Am	⁹⁰ Sr
17468	a) yes	ISO 11704, Other	Thermal preconcentration for liquid scintillation counting	b) liquid scintillation counter	²⁴¹ Am	⁹⁰ Sr
17491	a) yes	ISO 10704	Direct measurement after evaporation	a) gas-flow proportional counter	²⁴¹ Am	⁹⁰ Sr
17492	a) yes	ISO 11704, not applicable	Direct measurement after evaporation, Thermal preconcentration for liquid scintillation counting, coprecipitation	a) gas-flow proportional counter, b) liquid scintillation counter	²⁴¹ Am	⁴⁰ K
17500	a) yes	ISO 10704	Direct measurement after evaporation	a) gas-flow proportional counter	²³⁹ Pu	⁹⁰ Sr
17503	a) yes	ISO 10704	Direct measurement after evaporation	a) gas-flow proportional counter	²³⁹ Pu	⁹⁰ Sr
17513	a) yes	ISO 9696	Direct measurement after evaporation, other	a) gas-flow proportional counter	²⁴¹ Am	⁹⁰ Sr
17537	a) yes	not applicable	Direct measurement after evaporation, Direct measurement with liquid scintillation counting	b) liquid scintillation counter	²⁰⁹ Po	⁹⁰ Sr
17542	a) yes	ISO 9696, ISO 9697	Direct measurement after evaporation	a) gas-flow proportional counter	²⁴² Pu, ²⁴¹ Am	¹³⁷ Cs, ⁴⁰ K
17543	b) no	not applicable	Thermal preconcentration for liquid scintillation counting	b) liquid scintillation counter	²²⁶ Ra	Not defined

Table 18. Information on the best performing laboratories and their methods.

Lab ID	Accredited (17025)	Standard technique	Sample preparation	Measurement technique	Alpha calibration	Beta calibration
17434	a) yes	ISO 11704	Thermal preconcentration for liquid scintillation counting	b) liquid scintillation counter	²³⁶ U	⁴⁰ K
17568	a) yes	not applicable	Direct measurement after evaporation, Direct measurement with liquid scintillation counting	a) gas-flow proportional counter, b) liquid scintillation counter	²⁴¹ Am	Not defined
17394	a) yes	ISO 10704	Direct measurement after evaporation	a) gas-flow proportional counter	²³⁹ Pu	⁹⁰ Sr
17431	a) yes	ISO 11704	Direct measurement with liquid scintillation counting	b) liquid scintillation counter	²⁴¹ Am	⁹⁰ Sr

Table 19. Information on the best performing laboratories and their methods from the REM2012 ILC and REM2019 PT joint evaluation.

After reviewing the information from these most consistently performing 14 methods/organisations, we attempt to propose "*Best practices*". The most relevant information we used as the basis of this evaluation were: possession of ISO 17025 accreditation, following a written standard, measurement technique, sample preparation approach, radionuclides used for counting efficiency calibration. First number represents the number of cases from REM2019 PT, after the plus (+) symbol the number shows the cases from REM2012 ILC.

Having 17025 accreditation

- accredited: 11+4 cases
- non-accredited: 3 cases

Using standard technique

- ISO 10704: 5+1 cases
- ISO 11704: 4+2 cases
- ISO 9696: 2 cases
- ISO 9697: 1 case
- Other: 2 case; Not applicable: 3+1 cases

Measurement technique

- gas-flow proportional counter: 9 cases
- liquid scintillation counter: 6 cases
- solid scintillation counter: 2 cases

Sample preparation

- Direct measurement after evaporation: 10+2 cases
- Thermal pre-concentration for LSC: 5+1 cases
- Co-precipitation: 3 cases
- Direct measurement with LSC: 1+2 cases
- Other: 1 case

Alpha efficiency calibration, self-absorption correction

- ²⁴¹Am: 8+2 cases
- 239Pu: 3+1 cases
- ²³⁶U: 2 cases
- ²⁰⁹Po, ²⁴²Pu, ²²⁶Ra: 1 case

Beta efficiency calibration, self-absorption correction

- 90Sr: 10+2 cases
- ⁴⁰K: 3+1 cases
- ¹³⁷Cs, not defined: 1+1 case

6.1 Recommended methods

On the basis of the aforementioned information and the frequency of a parameter appeared in the **Table 18** and **Table 19** from the most consistently performing laboratories, the following practices can be recommended as summarised in **Table 20**.

We propose that the laboratory should follow a documented standard method, either ISO 10704 or ISO 11704. Sample preparation is done by either direct measurement with gas flow proportional counter after evaporation or thermal pre-concentration for liquid scintillation counting. The following radionuclides are preferred for counting efficiency calibration, self-absorption and quench correction: ²⁴¹Am or ²³⁹Pu; ⁹⁰Sr or ⁴⁰K.

We can note that the majority of these high performing laboratories were accredited following ISO 17025 for gross alpha/beta activity concentration measurement in water. However, it has to be stressed that having accreditation does not automatically mean better performance and good quality work. For laboratories with limited human resources it can be difficult to manage the administrative work related to accreditation.

Standard technique	Sample preparation	Measurement technique	Counting efficiency calibration*	
ISO 10704	Direct measurement after evaporation or	gas-flow proportional counter or	Alpha:	Beta:
ISO 11704	Thermal pre-concentration for liquid scintillation counting	liquid scintillation counter	²⁴¹ Am or ²³⁹ Pu	⁹⁰ Sr or ⁴⁰ K

Table 20. Summary of the "Best practices" parameters.

*also for self-absorption and quench correction determination.

7 Summary, key findings

In Europe today, gross alpha/beta activity concentration measurements in (drinking) water are still not reaching a satisfactory quality level because almost 50% of the results deviated more than accepted in this PT. On the basis of the percentage deviation score the acceptable scores varied between 51% and 63%. The situation seemed to be better when z-scores are compared with acceptable scores between 75%-87% success rate. This need some caution since z-score is interpreted in a way that the acceptance range is doubled comparing to percentage deviation. However, in case of zeta-score when reported uncertainties are also used for performance evaluation success rate was decreased between 38% and 62%.

Only 14 participants had acceptable performance with regards to percentage deviation and zeta score for all 4 parameters to report. There were 14 participants that could not report any acceptable result at all. Therefore, to some extent, the same conclusions are valid for the REM2019 PT as for the REM2012 ILC. Gross methods should be used with caution due to the numerous sources of interferences and there is a need for true standardisation/harmonisation (Jobbágy et al, 2016). However, comparing the two exercises (REM2019 PT and REM2012 ILC) some small improvements were noticed in performances especially in case of the natural water analysis on one hand but on the other hand worse performance was observed in case of the quality control (spiked) sample. As an average it shows no improvement.

At first glance it was difficult to identify superior method(s) but analysing the data thoroughly from the most consistently performing 14 methods/organisations *"Best practices"* can be proposed.

Following ISO standards for gross alpha/beta activity measurements in water could be a possible reason for improved performance in comparison to results from non-standard methods.

Method pitfalls

As was observed, gross methods were not performing with the desired accuracy and in some cases they fail to detect certain radionuclides. They give only "activity index numbers" rather than an approximate activity concentration, as explained by Schönhofer (2012) and re-confirmed by the data spread from this proficiency test as well after the REM2012 ILC exercise. The difference between laboratory results were between two to seven orders of magnitude, which falls far outside the measurement uncertainties.

The large spread of the results may be due to influencing factors during both the sample preparation and the measurement procedure. These influencing factors cannot generally be predicted and it is already difficult to define the measurand for gross activity analysis since the radionuclide composition of the sample is *a priory* not known.

On top of it, this is probably the only radiometric method where just metrological traceability cannot be established (except for reference materials) due to the lack of measurands. Measurement values are linked to particular sources/radionuclides used for gross alpha/beta efficiency calibration. Using different radionuclides would give different measurement result. Many different radionuclides used for calibration cover a wide energy range, which may be another reason for scattered results.

Since many combinations/variations of parameters are possible and might vary from one laboratory to another, it is not surprising that results show large spread.

In certain cases even using the same ISO standard method in different laboratories does not guarantee comparable results which shows poor method reproducibility.

The most important pitfalls and sources of interferences of the gross alpha/beta methods are related to (i) sample preparation methods (loss of volatile radionuclides), (ii) time delay between sample preparation and measurement (decay of ²²⁴Ra or ingrowth of radon and its progenies from ²²⁶Ra), (iii) detection technique and (iv) radionuclides used for counting efficiency calibration as detailed in a journal article by Jobbágy et al. (2014).

Other parameters like incorrect quench correction or alpha/beta discrimination for LSC can also introduce bias. In case of co-precipitation sample preparation approach ⁴⁰K is not co-precipitated and therefore excluded from the gross beta results (residual beta activity) unlike in case of direct evaporation or LSC where ⁴⁰K contributes to the gross beta activity. When using direct evaporation approach then source matrix and uniformity, surface density (i.e. self-absorption) and hygroscopic property of the sample play important roles.

The outcome of the analysis is certainly also influenced by the proficiency and skills of laboratory personnel. For certain techniques, especially the ones involving radiochemistry, it takes many years to reach the necessary level and obtain the necessary know-how. Hands on lab trainings are needed to train junior staff members or those analysts that are new in the field. Knowledge transfer from experienced professionals to junior staff already starts at undergraduate level. Another issue is how to attract, and later keep, the younger generation in this field.

Points of attention on reporting

There were some apparent blunders among the measurement results. These blunders are incorrectly reported measurement results where errors were made by placing the decimals at the incorrect place or measurement units. Since reporting is part of a PT exercise and also the analytical service, it is very important to pay attention to these details e.g. having a proper reporting review procedure or simply reading the PT reporting requirements.

Other important points to check are if counting efficiency calibration, quench correction, pulse discrimination, determination of self-absorption factor were done properly and if decay correction of the calibration source was performed. Whenever possible, the analysts should use certified/standardised materials with proper certificate from a trusted provider. Last but not least, the uncertainty budgets need to be checked and identification of all possible significant components introduced.

Policy aspect

Regarding policy impact of this PT: A minor revision of the E-DWD would be desirable to put more emphasis on promoting radionuclide specific analysis at least as part of a zero baseline study and periodical (annual or seasonal) surveys. Furthermore, considering the current trend in drinking water consumption habits in Europe, mineral water as water category should be included in the scope of the E-DWD.

7.1 Recommendations: method harmonisation and collaborations

For these aforementioned reasons, we recommend that gross alpha/beta methods are preferably not to be used as a standalone method to assess internal dose exposure or risk from radiation from water consumption. Gross measurement should be used for monitoring as a complementary or substitute method for radionuclide specific methods, once the radionuclide composition is known from radionuclide specific analysis.

We recommend (i) following strictly standardised procedures for sample preparation and measurement, (ii) to be aware of all decay and chemical processes that may affect the measurement, (iii) to test and validate methods and (iv) to build realistic uncertainty budgets.

The advantage of using liquid scintillation counting detection technique is that not only gives it quantitative information but semi-qualitative can be achieved as well from the alpha-beta spectrum.

Collaborative trials are desired to test gross methods under very rigorous truly standardised conditions to be able to conclude their accuracy, repeatability, analytical sensitivity and specificity. Furthermore detailed studies are needed to compare the cost, time effectiveness and other performance criteria of radionuclide specific- and gross alpha/beta measurement methods to be able to select fit for the purpose method(s) meeting the requirements of the different legislative/policy documents and of course the environmental radioactivity monitoring community. JRC could play a central role to coordinate such a trial involving external expert organisations in the field.

7.2 Future planning

To plan the JRC unit G.2 activity better in the course of the DG-ENER EURATOM Article 35-36 virtual-meeting in October 2020 and the JRC REM virtual workshop in May 2021 participants were requested to send their priorities on the JRC future PTs. The following PTs were mentioned by the Article 35-36 experts and participants:

- Radon-in-water PT and sampling exercise,
- Sr-90, Cs-137 in whey sample,
- U- and Ra isotopes, Po-210/Pb-210 in water,
- Repetition of gross alpha/beta activity in water PT,
- PT for PT providers as JRC proposed.

JRC-Geel in consultation with DG-ENER will take into account these requests from the Article 35 Expert group and REM PT participants when planning the upcoming proficiency tests.

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List of abbreviations, acronyms and definitions

activity concentration*	activity per unit volume
assigned value	also called reference value
BIPM	Bureau International des Poids et Mesures
cpm	counts per minute
D (%)	percentage deviation between the reported and the reference massic activity
DG ENER	European Commission's Directorate-General for Energy
EURATOM	European Atomic Energy Community
GUM	Guide to the Expression of Uncertainty in Measurement
HDPE	High Density Polyethylene
IEC	International Electrotechnical Commission
ILC	interlaboratory comparison
ISO	International Standardization Organization
JRC	Joint Research Centre (of the European Commission)
k	coverage factor according to GUM
LS	liquid scintillation
LSC	liquid scintillation counting
MAD	Median Absolute Deviation
massic activity*	activity per unit mass
MILC	Management of ILC. (JRC's online tool for reporting ILC-results)
MS	member states of the European Union
PFA	Perfluoroalkoxy alkane
PTFE	Polytetrafluoroethylene
PSA	pulse-shape analyser
PT	proficiency test
SCK CEN	Belgian nuclear research centre
SI	Système International d'Unités, International System of Units
U	expanded uncertainty according to GUM
u	standard uncertainty according to GUM
U(x _{lab})	expanded uncertainty of average laboratory result
u(x _{lab})	standard uncertainty of average laboratory result
U(X _{PT})	expanded uncertainty of reference value
u(X _{PT})	standard uncertainty of reference value
WHO	World Health Organization
X _{lab}	mean laboratory result of activity concentration
X _{PT}	reference value of activity concentration
σρτ	the standard deviation for proficiency assessment
z-score	compares each participant's deviation from the assigned value with the standard deviation

of the proficiency test assessment (σ_{PT}). Z-score

 ζ -score The zeta-score states whether a laboratory's result agrees with the assigned value considering both the reported uncertainty and the uncertainty of the reference value.

The term "test item" is used in standards; "reference material" and ""water sample" are wordings used when being more precise and less formal and less abstract/general.

* In this report, the matrix was water, which has a density very close to 1 kg/dm³. Although we clearly distinguish between massic activity (Bq/kg) and activity concentration (Bq/L), their numerical value would be almost identical.

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Annexes

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



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

Geel, 12 July 2019

Ref. Ares(2019)4523730 - 12/07/2019

Subject: Article 35-36 of the Euratom Treaty Nomination request: EC Proficiency Testing gross alpha/beta activity concentration measurements in water organised under the ICS-REM* programme

Ms Barbara Vokal Nemec Ms Carmen Rey del Castillo Ms Claudia Landstetter Ms Elena Simion Ms Iwona Matujewicz Ms Konstantina Kehagia Ms Maria José Bação Madruga Ms Marielle Lecomte Ms Monika Lepasson Ms Nathalie Reynal Ms Pia Vesterbacka Ms Rositza Kamenova-Totzeva Ms Sandra Quell Ms Sania Krca Ms Sarah Fallon Ms Sofía Luque Ms Sonia Fontani Ms María Teresa Sánchez

Mr Alar Polt Mr Andras Donaszi-Ivanov Mr Andris Abramenkovs Mr Antonis Maltezos Mr Árpád Vincze Mr Christian Katzlberger Mr Fabrice Leprieur Mr Giancarlo Torri Mr Guillaume Milot Mr Jixin Qiao Mr Josef Peter Mr Juozas Molis Mr Jurgen Claes Mr Kasper Grann Andersson Mr Kevin Kelleher Mr Lars Roobol Mr Lionel Sombré Mr Martijn van der Schaaf Mr Michalis Tzortzis Mr Michel Baudry Mr Michel Cindro Mr Ondřej Chochola Mr Pål Andersson Mr Paul Brejza Mr Pawel Lipinski Mr Pieter Kwakman Mr Reimund Stapel Mr Selwyn Runacres 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 one of the next PT exercises will be on **gross alpha/beta activity measurements in water.**

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

The tentative schedule for the PT is as follows:

- July 2019: Registration is open to participants. Announcement to Article 35/36 Experts, DG ENER and expert labs,
- 20 September 2019: on-line registration deadline,
- October-December 2019 (t.b.d.): Organizer will start shipping PT material to participants,
- 31 January 2020: deadline for results reporting,
- March 2020: preliminary report distributed,
- May 2020: Final report,
- 2020 t.b.d.: workshop and training course organised for PT participants.

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). For that purpose, please fill in your (nationally coordinated) response in the attached excel table and send it back to the functional e-mail address below by **6 September 2019**.

Please also forward the registration link with the registration instructions to your nominated laboratories so they can do the registration themselves.

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 Project Coordinator Mikael Hult Team Leader Petya Malo Logistics Assistant



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

cc: Messrs. Michael Hübel, Vesa Tanner, Alan Ryan (DG ENER.D3) Mr. Marc De Cort (JRC Ispra) Messrs. Arjan Plompen, Mikael Hult, Ms. Petya Malo (JRC Geel)





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

Geel, 12 July 2019

Subject: Invitation for participation in the JRC-REM 2019 proficiency test on gross alpha/beta activity measurements in water

Dear colleagues,

It is our pleasure to invite your laboratory to participate in the JRC-REM 2019 proficiency test on gross alpha/beta activity measurements in water (reference: JRC-REM 2019 Water 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 materials are a natural origin water sample and a spiked laboratory water sample. These water samples contain environmental level of radionuclides but the material can be transported freely and handled in the laboratory without any radiological restrictions. However, it can be expected that the gross alpha and beta activity concentrations of these samples are above the corresponding detection limits indicated in the EURATOM Drinking Water Directive-Council Directive 2013/51 (0.04 Bq/L and 0.4 Bq/L for gross alpha and gross beta activities respectively). The PT materials will be directly filled into plastic bottles with screw cap. The organizer can provide only one bottle per sample for each participant. Each bottle will contain approximately 1 L of the material, which is expected to be sufficient for the requested analyses. Additional material can be provided on request. If the bottle is damaged or significant sample loss observed, contact us immediately, in that case an extra sample can be shipped.

Registration and reporting

The registration and 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?selComparison=2321

Please be aware that the deadline for registration is 20 September 2019.

The registration for this PT is open to any organizations in Europe. However, in case of high number of participants, priority will be given to organizations nominated by the Euratom article 35/36 contacts and national authorities. Confirmation of participation will be sent shortly after the registration deadline.

You will have the opportunity to report results obtained by different gross methods (LSC, evaporation, co-precipitation etc.).

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 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. The questionnaire can be filled in from the web-based reporting interface.
- 3. Tentative timing and deadlines
 - July 2019: Registration is open to participants. Announcement to Article 35/36 Experts, DG ENER and expert labs,
- 20 September 2019: on-line registration deadline,
- October-December 2019 (t.b.d.): Organizer will start shipping PT material to participants,
- 31 January 2020: deadline for results reporting,
- March 2020: preliminary report distributed,
- May 2020: Final report,
- 2020 t.b.d.: workshop and training course organised for PT participants.

Reference values and scoring

Reference values of the comparison samples will be established by gravimetric spiking and measurements of the individual alpha and beta particle emitting radionuclides. The comparison will be evaluated with respect to these reference values using percentage difference from the reference value and in addition z-score and zeta-score where applicable. 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. Short term and long term stabilities will be also checked.

Note on reference date: in theory decay correction is not possible for gross alpha/beta parameters. Therefore, reference date will not be given.

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 <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 the Directorate-General for Energy.

In order to comply with the European regulation on the General Data Protection Regulation (GDPR), the name of contact people will remain undisclosed. We will only list the name of your organization in the final report.

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 look forward to your participation in this exercise.

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

Annex 2. Registration instructions



EUROPEAN COMMISSION DIRECTORATE-GENERAL JOINT RESEARCH CENTRE Directorate G - Nuclear Safety and Security Standards for Nuclear Safety, Security and Safeguards Ref. Ares(2019)4523730 - 12/07/2019

Geel, 12 July 2019

<u>Subject:</u> Instructions for registration in the JRC-REM 2019 Water proficiency test on gross alpha/beta 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?selComp arison=2321

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.

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rganisation details	•			
Countrys *	BELGOUM			
Organisation: *	JAC-Geel			
Department				
treet - Numberi *	Relievenung 111			
Fmail				
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response.	+12 14 123/04			
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Titles Los	1971			
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act Name: " 1.44-				
Geoder: Male	E			
Email: * wieter	where the arrows as			
elephones * +22 to	123456			
Extension				
Fax: +32				

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!

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European Commission ILC - Re	gistration	
firm Danistration		
	Companion	2321 186-864 2019 Water Perforency Test
Organisation details		
Org De Street	Country: BELGLIM arikalian: IRC God partment: Number: Religneweg 111	
	Finault Zip code: 2440 City: Geel elephone: +32 14 123456 Fax: +32	Extension:
Contact person details		
Title: Mr. First Name: Vito Gender Mia Email: Vito Telephone: 432 Foc +32	ay jobbaç∕v≊ec.europa.eu 4 123456	Extension:
ange Confrm		

Step 3: Download the registration form as 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".

E Titeles prevented this site from opening a pop-up window.	Quicos X
European ILC - Registration	
Regenerations confirmation	
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It is recommended that you print the form and keep it for your records as proof of registration but it is **NOT** required to sign it and send it to us by e-mail.

After this step, your registration is complete.

Annex 3. Reporting instructions

How to submit your results for the reporting

JRC-REM 2019 Water PT

Step 1: Click on the link to the reporting module and insert your password key and e-mail used for registration.

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

European Commission	JOINT RESEARC	H CENTRE	
Please provide your participation key			
	Password key: Contact person Email:	Login	
			version: 4.2.7 (25/04/2019) [GEELEXT 01]

Step 2-1. Report the results for JRC-GAB-1 and JRC-GAB-2 samples. You can report for both samples at once or separately. Please DO NOT report results through Excel.

	JOINT RESEARCH CENTRE
European Commission	ILC - Reporting
Deventions FIDC DEN 2010 Maters D	
T TEST	ronciency rest]
	STEP 2 Report for sample JRC-GAB1 Or Report for sample JRC-GAB2 Or Report for ALL Samples at once Report values through Docel
	Preview remotiv questionnaire Preview reported values Preview reported questionnaire Preview reported questionnaire Preview reported values and questionnaire I confirm I reported my results and answered the questionnaire

Step 2-2. Report the results for JRC-GAB-1 and JRC-GAB-2 samples. Once you filled in the table, you can validate and save your results then go back to the main page. Parameters with an asterisk are mandatory (if you do not have measurement result, you may fill in zero in number). Note: activity units in mBq/L; Uncertainty in mBq/L; indicate the coverage factor (k=1, 2, etc.). Result input for JRC-REM 2019 Water Proficiency Test

TTT TEST												
Sample Code TTT10631448 - JR	C-GAB1											
For decimal values use a dot "."	instead of a comma ",".											
Measurand		Measurement Reference Date	Result		Unil		Uncert. value	Coverage Faktor k		Technique		Clear
Gross Alpha activity-Technique 1	Specific activity [mBq/L]	Mean	= v *	*	mBq/L	٠	*	*	No technique		٠	0
Gross Alpha activity-Technique 2	Specific activity [mBq/L]	Mean	- •		mBq/L	,			No technique		•	a
Gross Alpha artivity-Technique 3	Specific activity [mBŋ/I]	Mean	= •		mBŋ/I	,			No technique		•	0
Gross Beta activity-Technique 1	Specific activity [mBq/L]	Mean	= • *	•	mBq/L	,	*	*	No technique		•	0
Gross Beta activity Technique 2	Spedfic activity [mBq/L]	Mean	- •		mBq/L	٠			No technique		•	0
Gross Beta activity-Technique 3	Specific activity [mBq/L]	Mean	- •		mBq/L	۲			No technique		٠	0
Total dissolved solids	concentration[mg/L]	Mean	- •	*	mg/L	٠	•	•	No technique		٠	a
Sample Code TTT10735330 - JR	C-GAB2											
For decimal values use a dot "."	instead of a comma ",".											
Measurand		Measurement Reference Date	Result		Unil		Uncert. value	Coverage Faktor k		Technique		Clear
Gross Alpha activity-Technique 1	Specific activity [mBq/L]	Mean	= • *	*	mBq/L	,	*	*	No technique		•	0
Gross Alpha activity-Technique 2	Specific activity [mBq/L]	Mean	- •		mBq/L	7			No technique		۲	
Gross Alpha activity-Technique 3	Specific activity [mBq/1]	Mean	= •		mBŋ/I	,			No technique		•	0
Gross Beta activity-Technique 1	Specific activity [mBq/L]	Mean	= • *	•	mBq/L	,	•	•	No technique		•	0
Gross Beta activity Technique 2	Spedfic activity [mBq/L]	Mean	- •		mBq/L	٠			No technique		•	0
Gross Beta activity-Technique 3	Specific activity [mBq/L]	Mean	- •		mBa/L	7			No technique		٠	0
Total dissolved solids	concentration/mu/L1	Mean			mu/L		•	•	No technique		Ŧ	2

Clear page results Save page results Validate and save Back to main page

1

Step 3. Fill in the questionnaire (mandatory!). It takes maximum 15 minutes. Please save and validate it.

15. What was the time delay between finishing the sample for measurements and starting gross measurements (in hours)?
16. Do you have a procedure to treat hygroscopic residues?
 Ø a) yes Ø b) no ♥ c) not applikable
17. Did you apply quenching correction? *
 Ø a) yes Ø b) no Ø c) not applicable
18. Did you correct for surface density of the prepared source? *
 Ø a) yes Ø b) no Ø c) not applicable
19. What is your gross alpha activity detection limit (in mBq/L)? *
20. What is your gross beta activity detection limit (in mBq/L)? *
21. Introduce briefly your calibration procedure including information on your calibration source. *
22. Please give your brief feedback on the PT exercise (remarks, improvements or positive feedback): *
23. Overall satisfaction score. How satisfied were you with the PT? (1: unsatisfied, 10: very satisfied) *
Clear questionnaire Save questionnaire Validation Back to main page

Step 4. Check the box next to the text "I confirm I reported my results and answered the questionnaire" and click on "Submit my results".

Step 5. 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.

UDINT RES European Eu	SEARCH CENTRE	
ILC Reporting - [JRC-REM 2019 Water Proficiency Test]		
TTT TEST		TEST TEST BELGIUM
STEP 5	Reported values and questionnaire Table Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire the submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2020 Results and questionnaire are submitted as confirmed on 20-01-2	Download the form with the submitted results and questionnaire, sign and send to JRC-Geel by e-mail

version: 4.2.7 (25/04/2019) [GEELEXT_01]

Annex 4. Accompanying letter



EUROPEAN COMMISSION JOINT RESEARCH CENTRE Directorate G - Nuclear Safety and Security

Standards for Nuclear Safety, Security and Safeguards

Geel, 8 January 2020 JRC.G.2/VJ/Ares(2020)87555

«Title» «Firstname» «Surname» «Organisation» «Department» «Address» «Zip» «Town» «Country»

Subject: JRC-REM 2019 proficiency test on gross alpha/beta activity measurements in water

Background information

EU Member States are obliged under Art. 35 and 36 of the Euratom Treaty to inform the European Commission (EC) on a regular basis on the radioactivity levels in their environment, in some food products and in drinking water. In order to obtain more information on the quality of the values reported by the Member States, the EC (through its Directorate General JRC) organises regularly a European interlaboratory comparison exercise.

Your laboratory has been either nominated by your national representative(s)/authority or the International Atomic Energy Agency and you registered to participate in the proficiency test on gross alpha/beta activity in drinking waters organised by JRC-Geel. Two different water samples were sent by courier service to your laboratory.

Water JRC-GAB1

Water JRC-GAB2

The box contains two bottles from each PT material (4 bottles in total). Each bottle contains either approximately 1 kg of the material, which is expected to be sufficient for the requested analyses.

Material information

The PT samples contain environmental levels of alpha and beta emitting radionuclides below their exemption levels. Therefore, the materials can be transported freely and handled in the laboratory without any radiological restrictions. However, it may happen that gross alpha/beta activity is above the screening levels laid down in the Euratom Drinking Water Directive (Council Directive 2013/51/Euratom of 22 October 2013). The water samples were acidified by adding concentrated HNO₃ where their acidity was set to pH < 2. Samples have to be handled accordingly respecting chemical safety regulations and following good laboratory practices.

European Commission, Retieseweg 111, 2440 Geel, BELGIUM Viktor.JOBBAGY@ec.europa.eu One of the waters was spiked gravimetrically with alpha and beta emitting isotopes from certified standard solutions, while the other contains radionuclides naturally.

Samples were bottled in polypropylene bottles at JRC-Geel. The total dissolved solid (TDS) content of the material after bottling was determined but needs to be re-measured and reported in each laboratory in order to get information on their salinity.

Reference values of the comparison samples will be established by using radionuclide specific measurements and from gravimetric spiking. The comparison will be evaluated with respect to these reference values using relative percentage deviation and when applicable zeta- and z- scores. Therefore, a well-founded estimate of the uncertainty of the reported results is required from each participating laboratory.

Upon arrival of the package, please check if the test item is undamaged after transport and send us the *Sample receipt form* by e-mail to the indicated e-mail address immediately.

Store your samples in a dark place at the temperature indicated in the *Material Information Sheet*.

Before the analysis, it is recommended to store the sample bottle at room temperature until it reaches thermal equilibrium with its environment.

Protocol in brief

- 1. Each participant should receive two bottles from each water sample (4 bottles in total).
- 2. Total dissolved solid (TDS) content is to be determined by the participant on small aliquots (e.g. 50 mL).
- 3. TDS content should be reported in mg/L as dry residue at 180 °C.
- 4. The laboratory may use several measurement procedures of its choice, which are preferably consistent with routine procedures used in the laboratory.
- 5. The gross alpha/beta activity concentration (volumetric activity) should be determined and reported as mBq/L. Also, the uncertainty should be reported in mBq/L.

Timing and deadlines

1. Material distribution of JRC-REM2019-PT samples: as of 9th January 2020

2. Deadline for reporting results: 20 March 2020

- 3. Evaluation of the individual laboratory performance will be sent by e-mail in April 2020.
- 4. The final report of this comparison exercise is planned to be available by September 2020.
- 5. Follow-up workshop: 22-25 September 2020. On 21 September 2020, there will be a half-day workshop discussing the results of a European questionnaire on methods for radioactivity monitoring in food. Further information will follow.

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 below:

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

Password key: «Part_key»

You will have the opportunity to submit results obtained by different methods. You are requested to report:

- the measurement technique you used,
- one measurement result/mean value per technique (in mBq/L),
- associated uncertainty and the coverage factor of k.

Note on reference date: in theory, decay correction is not possible for gross alpha/beta parameters. Therefore, reference date will not be given.

Questionnaire is a part of this exercise and participants should answer all relevant questions regarding the procedures that they have used. The questionnaire is available from the on-line reporting website.

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

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 DG ENER. Apart from informing these authorities, each laboratory's results will be treated anonymously and will not be given to any third parties other than the aforementioned organisations.

In case of further questions or complaints, 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

Tel: +32 (0)14 571 251 e-mail: <u>JRC-GEE-REM-COMPARISONS@ec.europa.eu</u> European Commission Joint Research Centre Directorate G - Nuclear Safety and Security Standards for Nuclear Safety, Security and Safeguards Retieseweg 111 B-2440 Geel, Belgium

Tel: +32 (0)14 571 251 e-mail: <u>JRC-GEE-REM-COMPARISONS@ec.europa.eu</u>

REM Proficiency Tests: <u>https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests</u> <u>https://ec.europa.eu/jrc/en</u>

Enclosures:

Material Information Sheet
 Sample receipt form

4





EUROPEAN COMMISSION JOINT RESEARCH CENTRE Directorate G - Nuclear Safety and Security

Standards for Nuclear Safety, Security and Safeguards

Geel, 8 January 2020

Material information sheet

The package contains two proficiency test materials a natural mineral water (JRC-GAB1) and a spiked type-II laboratory water from JRC (JRC-GAB2).

Major chemical characteristics: both water samples may contain calcium, magnesium, sodium, potassium, chlorides and nitrates. Both water samples are acidified with nitric acid to adjust $pH \cong 1-2$.

The water samples are intended for **laboratory tests only**. They are **NOT intended for human consumption**!

From radiation protection point of view processing these waters will not result in elevated external dose exposure and can be handled freely in the laboratory as such.

The activity of the PT samples is well below the exemption levels in terms of massic activity and total activity.

JRC-GAB1 water sample contain naturally occurring alpha - and beta emitting isotopes only.

JRC-GAB2 water sample may contain artificial and naturally occurring alpha - and beta emitting isotopes as well.

Take precautions to avoid cross contamination and avoid injuries from chemical risks.

Recommended storage temperature is between +4 and +20 °C.

Waste management: always follow the local rules on sorting and handling chemical waste. Do not discard directly into municipal sewage system.

European Commission, Retieseweg 111, 2440 Geel, BELGIUM Viktor.JOBBAGY@ec.europa.eu Weakly acidified water sample (aqueous nitric acid solution < 0.1 % w/w)

Provider: JRC-Geel

1 L per bottle

May cause skin and eye irritation

P260 Do not breathe dust/ fume/ gas/ mist/ vapours/ spray.

P280 Wear protective gloves/ protective clothing/ eye protection/face protection.
P303 + P361 + P353 IF ON SKIN (or hair): Take off immediately all contaminated clothing.
Rinse skin with water/shower.
P304 + P340 + P310 IF INHALED: Remove person to fresh air and keep comfortable for breathing.
Immediately call a POISON CENTER/doctor.
P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact

P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

The materials do not contain hazardous substance or mixture according to Regulation (EC) No. 1272/2008.

https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:02008R1272-20180301&from=EN

These materials do not contain substances of very high concern according to Regulation (EC) No 1907/2006 (REACH), Article 57 above the respective regulatory concentration limit of $\ge 0.1 \%$ (w/w).

https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:02006R1907-20140410&from=EN

These materials are not classified as dangerous in the meaning of transport regulations (ADR/RID/IATA).

Annex 5. Sample receipt form





EUROPEAN COMMISSION JOINT RESEARCH CENTRE

Directorate G - Nuclear Safety and Security (Karlsruhe) Standards for Nuclear Safety, Security and Safeguards

Geel, 8 January 2020

Subject: Sample receipt form

Reference: JRC-REM 2019 Water PT

Date of package arrival:

– (day/month/year):

Sample codes, bottle numbers:

JRC-GAB1	JRC-GAB2

Please return this form by e-mail 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

Organisation:	•••
Contact person:	
E-mail address:	

Thank you for returning this form to the e-mail below:

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

Retieseweg 111, B-2440 Geel - Belgium.

1

Annex 6. List of participating laboratories

ALBANIA

Institute of Applied Nuclear Physics, Tirana University Radiometry & Radiochemistry Th. Filipeu, Qesarak 1001 Tirana

ALGERIA

Centre de Recherche Nucléaire d'Alger Physique radiologique 02, Bd Frantz fanon 399 Alger R., Alger

AUSTRIA

Austrian Agency for Health and Food Safety Radiation Protection and Radiochemistry Spargelfeldstrasse 191 A-1220 Vienna

BELGIUM

IRE (Institute for radioelements) BUS Avenue de l'éspérance 1 6220 Fleurus

BELGIUM

VIVAQUA VIVAQUA LABORATOIRE TAILFER Rue des Rochers de Frêne 5170 Lustin

BOSNIA - HERZEGOVINA

Public Health Institute Radiation protection center Jovana Ducica 1 78000 Banja Luka

BOSNIA - HERZEGOVINA

Institute for Public Health of FB&H Radiation protection centre Marsala Tita 9 71000 Sarajevo

BULGARIA

Regional Health Inspectorate Radiation Control Perushtitsa 1 4002 Plovdiv

BULGARIA

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

BULGARIA

Executive Environment Agency Lab for Radiation Measurements 136, Tzar Boris III, blvd. 1618 Sofia

BULGARIA

Regional health inspectorate of Burgas Control of radiation department Aleksandrovska str. 120 8000 Burgas

CROATIA

Ruđer Bošković Institute Laboratory for radioecology Bijenička cesta 54 10000 Zagreb

CYPRUS

State General Laboratory Kimonos,44 1451 Nicosia

CZECH REPUBLIC

Water Research Institute T. G. Masaryka, Public Research Institutions Departement of Radioecology Podbabska 30/2582 160 00 Prague 6

ESTONIA

Environmental Board Radiation Safety Department Kopli 76 10416 Tallinn

FINLAND

Radiation and Nuclear Safety Authority Laippatie 4, P.O.Box 14 00881 Helsinki

FRANCE

EDF CNPE GOLFECH BP 24 82400 Valence D'Agen

FRANCE

ORANO MINING - CIME/SAN Haute Vienne 2 route de Lavaugrasse 87250 Bessines/Gartempe

FRANCE

Subatech/SMART 4 rue Alfred KASTLER 44307 Nantes

FRANCE

CNPE CHINON 37, Laboratoire Environnement BP 80 37420 Avoine

FRANCE

EICHROM EUROFINS RADIOACTIVITE Campus Ker Lann rue Maryse Bastié Parc de Lormandière, Bât. C 35170 Bruz

FRANCE

Orano Cycle La Hague 50440 Beaumont Hague

FRANCE

EDF Direction Industrielle Bat. Pierre Gilles de Gennes CNPE de CHINON 37420 Avoine

FRANCE

ANDRA DOI/CA/LAC CSA, BP 7 10200 Soulaines Dhuys

FRANCE

EDF CNPE de Flamanville Magasin relais - BP4 50340 Les Pieux

FRANCE

CEA DEN/MAR/DUSP/SPR/LMAR Centre de Marcoule Bâtiment 40 - BP 17171 30207 Bagnols-sur-ceze

FRANCE

PearL 20 Rue Atlantis 87068 Limoges

FRANCE

IRSN SAME 31 rue de l'Ecluse

78116 Le Vesinet

FRANCE

Marine Nationale Brest – Lasem 15 bis Avenue Ecole Navale 29430 Brest

GERMANY

Bundesamt für Strahlenschutz Strahlenschutz und Umwelt Koepenicker Allee 120-130 10318 Berlin

GERMANY

Karlsruhe Institute of Technology Safety and Environment (SUM) Hermann-von-Helmholtz-Platz 1 76344 Eggenstein-Leopoldsh

GERMANY

Federal Institute of Hydrology G4 Am Mainzer Tor 1 56068 Koblenz

GREECE

Greek Atomic Energy Commission Department of Environmental Ra P. Grigoriou & Neapololeos 15310 Agia Paraskevi

GREECE

Athens Analysis Laboratories Alikarnassou 31 14231 Nea Ionia Athens

GREECE

Agrolab Rds Environmental Lab Industrial Area Thessaloniki Sindos, Po Box 48 57022 Thessaloniki

HUNGARY

RadiÖko Kft. Wartha Vince 1/2 8200 Veszprém

HUNGARY

National Food Chain Safety Office Food Chain Safety Laboratory Directorate Radioanalytical Reference Lab. 13 – 15 Fogoly Street 1182 Budapest

HUNGARY

Paks NPP Enviromentalcontrol Laboratory Kurcsatov street 1/D. 7030 Paks

HUNGARY

MECSEKÉRC Zrt Radiometriai laboratórium Akna utca 2. 7673 Kővágószőlős

HUNGARY

National Public Health Center Radiobiology and Radiohygiene Anna utca 5. 1221 Budapest

HUNGARY

Baranya County Government Office Laboratory Department Szentlőrinci Street 4/1. 7634 Pécs

HUNGARY

MVMV PA Zrt. Environmental Protection Pf. 71; Hrsz.:8803/17 7031 Paks

IRELAND

EPA ORM Block 3, Clonskeagh Square, Clonskeagh Road D14 H424 Dublin 14

ITALY

ENEA Radiation Protection Institute Strada per Crescentino, 41 13040 Saluggia (VC)

ITALY

Agenzia Regionale per la Protezione dell'Ambiente della Sardegna – ARPAS DTS Servizio Agenti Fisici Viale F. Ciusa 6 09131 Cagliari

ITALY

ARPACAL Reggio Calabria Via Troncovito snc 89135 Reggio Calabria

ITALY

ARPA Lazio Sede di Viterbo Via Montezebio 17 01100 Viterbo

ITALY

ARPA LOMBARDIA CRR Milano Via Filippo Juvara 22 20129 Milano

ITALY

ARPA Valle d'Aosta Environmental Radioactivity Loc. La Maladièrerue de la Maladière, 48 11020 Saint-Christophe

ITALY

ARPA Friuli Venezia Giulia SOS CRRVia Colugna 42 33100 Udine

ITALY

A.R.P.A.C. (Agenzia Regionale per la Protezione Ambientale della Campania) Salerno (C.R.R.) via Lanzalone 54/56 84126 Salerno

ITALY

Sogin SpA Via Fermi, 5/A fraz. Zerbio 29012 Caorso

ITALY

ARPA Veneto U.O.p Anal.Spec.Lab.Ovest CRR Via Dominutti 8 37135 Verona

ITALY

ISIN- Italian Inspectorate for Nuclear Safety and Radiation Protection Radiometric Laboratories via di Castel Romano 100 00128 Rome

ITALY

ARPA Piemonte Radiation Via Jervis, 30 10015 Ivrea (TO)

ITALY

ARPA Marche Dipartimento prov.le di Ancona U.O. Radioattività Ambientale via Colombo, 106 60127 Ancona

ITALY

Environmental Protection Agency Tuscany Region UO Radioattività e Amianto via Ponte alle Mosse 211 50144 Firenze

ITALY

Arpa Piemonte (Agenzia Regionale per la Protezione Ambientale del Piemonte) Ionizzanti siti nucleari via Trino, 89 13100 Vercelli

ITALY

Agenzia provinciale per l'ambiente e la tutela del clima Lab analisi aria e radioprotez Via Amba Alagi, 5 39100 Bolzano

ITALY

ARPA SICILIA S.T. di Palermo via Nairobi, 4 90129 Palermo

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

Kaos Coop Via Montebello 13 44121 Ferrara

ITALY

ENEA Radioprotection Institute Via Anguillarese 301 00123 Rome

ITALY

Protex Italia Srl Via Cartesio 30 47122 Forlì

ITALY

Agenzia Regionale per la Protezione dell'Ambiente della Basilicata (ARPAB) Centro Regionale Radioattività Via dei Mestieri 43 75100 Matera

LATVIA

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

LITHUANIA

Radiation Protection Centre Expertise and Exposure Monitor Kalvariju 153 08352 Vilnius

LUXEMBOURG

Ministère de la Santé - Direction de la Santé Radioprotection Villa Louvigny, Allée Marconi 2120 Luxembourg

MONTENEGRO

LLC Center for Ecotoxicological Research Podgorica Radionuclide Analytics Unit Bulevar Šarla de Gola 2 81000 Podgorica

NETHERLANDS

RIVM Antonie van Leeuwenhoeklaan 9 3721 MA Bilthoven

NORTH MACEDONIA

Public Health Institute Radioecology 50 Divizija No. 6 1000 Skopje

POLAND

Central Mining Institute Silesian Centre for Envi. Radi plac Gwarkow 1 40-166 Katowice

POLAND

Central Laboratory for Radiological Protection Departmen of Radiation Hygien Konwaliowa 7 03-194 Warsaw

POLAND

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

POLAND

Technical University of Lodz Applied Radiation Chemistry Wróblewskiego 15 90-924 Łódź

POLAND

National Institute of Public Health – National Institute of Hygiene (NIPH – NIH) Radiation Hygiene& Radiobioloy 24 Chocimska street 00-791 Warsaw

PORTUGAL

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

ROMANIA

SNN-CERNAVODA NPP Environmental Laboratory Cazarmii 6a 905200 Cernavoda

ROMANIA

Directia de Sanatate Publica a Judetului Cluj Laborator Igiena Radiatiilor Nicolae Balcescu, 16 400160 Cluj Napoca

ROMANIA

Bucharest Public Health Authority Hygiene Radiation Laboratory Reconstrucției Street nr.6, sector 3 Bucharest 031 726 Bucharest

ROMANIA

Direcția de Sănătate Publică Bihor Libertății Nr.34 410042 Oradea

ROMANIA

Institute for Nuclear Research – Pitesti RadiationProtection Laboratory Campului str., No. 1 115400 Mioveni

ROMANIA

Regional Center of Public Health Cluj of National Institute of Public Health Radiation Hygiene Laboratory Louis Pasteur 6 400349 Cluj Napoca

ROMANIA

Departament of Public Health Iasi Radiation Hygiene Laboratory V. Conta, 2-4 700117 Iasi

ROMANIA

Directia de Sanatate Publica Judeteana Suceava Laborator Igiena Radiatiilor Str. Scurta Nr. 1A 720223 Suceava

ROMANIA

Directia de Sanatate Publica Caras-Severin Laboratorul de Radiatii str. Spitalului nr.36 320076 Resita

ROMANIA

Public Health District Authority Arges Radiation Hygiene Laborator Exercitiu Nr 39 Bis 110438 Pitesti

ROMANIA

Directia de Sanatate Publica a Judetului Sibiu LIRI Gh. Baritiu, Nr.3 550178 Sibiu

ROMANIA

Directorate Public Health Maramures Laboratory of Radiation Health Victiriei 132, Baia Mare, Maramures 430076 Baia Mare

ROMANIA

Public Health Authority - Dolj county Laboratory of Ionising Radiati Constantin Lecca street, no 2 200143 Craiova

ROMANIA

National Institute of RD for Physics and Nuclear Engineering - "Horia Hulubei" Life and Environmental Physics 30 Reactorului street, Magurele, jud. Ilfov, 077125 Magurele

ROMANIA

Directia de Sanatate Publica Mures Laborator Igiena Radiatiilor Gh. Doja, nr. 34 540342 Tg.-Mures

ROMANIA

Directia de Sanatate Publica Prahova Laborator Igiena Radiatiilor Transilvaniei, 2 100179 Ploiesti

ROMANIA

National Institute of Public Health Radiation Hygiene Laboratory Street Dr. Leonte Anastasievici no. 1-3, sector 5 050463 Bucharest

SERBIA

Serbian Institute of Occupational Health "Dr Dragomir Karajovic" Radioecology Department Deligradska 29 11000 Belgrade

SERBIA

Institute for Nuclear Sciences Vinča Radiation and Environmental Pr Mike Petrovića Alasa 12-14 11351 Belgrade

SERBIA

Public Company "Nuclear Facilities of Serbia" Mike Petrovića Alasa Street, 12-14 11351 Vinča, Belgrade

SERBIA

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

SLOVAKIA

Slovenské elektrárne, a. s. NPP Mochovce Komenského 6 Environmental Radiation Monitoring Laboratory 93401 Levice

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 Trnavská cesta 52 82102 Bratislava

SLOVAKIA

University Comenius in Bratislava Faculty of Natural Sciences, Nuclear Chemistry Ilkovičova 6, Mlynská dolina 84215 Bratislava

SLOVAKIA

Slovenské elektrárne a.s. Laboratória radičnej kontroly Okružná 14 91701 Trnava

SLOVAKIA

Regional Public Health Organisation Radiation protection Ipeľská 1 04001 Košice

SLOVENIA

ZVD d.o.o. Chengdujska cesta 25 1260 Ljubljana

SLOVENIA

Jozef Stefan Institute F2 - Low/Medium Energy Physics Jamova cesta 39 1000 Ljubljana

SPAIN

University of Huelva Integrated Sciences Faculty Experimental Sciences Campus El Carmen s/n 21007 Huelva

SPAIN

ENUSA Industrias Avanzadas S.A., S.M.E (Radiological Protection and Environment) Environmental Laboratory Km7 Ciudad Rodrigo- Lumbrales Road 37592 Saelices el Chico

SPAIN

Universitat Politècnica de Catalunya Institut Tècniques Energètique Edifici Etseib, Campus Sud, Planta O, Pavelló C Diagonal 647 08028 Barcelona

SPAIN

Universidad de Santiago de Compostela Fisica de Particulas C\ Xoaquin Diaz de Rabago, s/n, Edif. Monte da Condesa Campus Vida, Laboratorio LAR 15782 Santiago de Comp

SPAIN

LaRUC (University of Cantabria) Faculty of Medicine Cardenal Herrera Oria S/N 39011 Santander
Medidas Ambientales, S.l. Laboratory Barrio Villacomparada s/n 09500 Medina de Pomar

SPAIN

University of Leon Quimica Y Fisica Aplicadas Escuela de Ingenierias Industr Campus De Vegazana S/N 24007 Leon

SPAIN

University of the Balearic Islands Environmental Radioactivity La Cra. Valldemossa km 7'5, Ed. Mateu Orfila 07122 Palma De Mallorca

SPAIN

University of Granada Inorganic Chemistry, Radiochemistry Environmental Laboratory Faculty of Sciences Av. Fuentenueva, s/n 18077 Granada

SPAIN

CIEMAT Medio Ambiente Avenida de la Complutense 40, Ed. 70. P1. D11 28020 Madrid

SPAIN

University of Malaga Central Research Facilities Universidad de Málaga, SCAI Bulevar Louis Pasteur, 33 29071 Malaga

University of Extremadura LARUEX, Applied Physcis Faculty of Veterinary Avda. Universidad, s/n 10003 Cáceres

SPAIN

GEOTECNIA Y CIMIENTOS S.A. (GEOCISA) Area Nuclear c/ Los Llanos de Jerez 10-12 28823 Coslada (Madrid)

SPAIN

Universidad del Pais Vasco Escuela de Ingenieria de Bilbao; Dpt Ing Nuclear y Mec Fl Lab Medidas Baja Actividad Pz Ingeniero Torres Quevedo, 1 48013 Bilbao

SPAIN

Universidad de La Laguna Laboratorio de Física Médica Facultad de Medicina Apartado 456 38200 La Laguna

SPAIN

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

SPAIN

University of Sevilla Applied Physics II Escuela Superior Arquitectura Avda. Reina Mercedes, 2 41012 Sevilla

University of A Coruña Avenida 19 de febrero S/N Ferrol, A Coruña 15405 Ferrol

SPAIN

Universitat Politecnica De Valencia Laboratorio de 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

ENUSA Industrias Avanzadas SA SME Protección Radiológica Juzbado ctra. Salamanca-Juzbado km.26 37115 Juzbado

SPAIN

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

SPAIN

Universidad de Salamanca Física Fundamental Calle Espejo Nº 2, Edificio I+D+i 37008 Salamanca

Universidad de Valencia Laboratorio de Radiactividad Ambiental C/ Dr. Moliner, 50 Edificio de Investigación Sótano -2 46100 Burjassot

SPAIN

UPM-E.T.S.I Caminos, Canales y Puertos Laboratorio Ingenieria Nuclear Profesor Aranguren 3 28040 Madrid

SPAIN

Labs & Technological Services AGQ SL Radioactivity Laboratory Ctra. A-8013 Km. 20.8 41014 Burguillos

SPAIN

Universitat de Barcelona Lab. de Radiologia Ambiental Martí I Franqués 1-11, 3ª Planta 08023 Barcelona

SPAIN

Instituto de Salud Carlos III Radioprotección (CNSA) Ctra. Majadahonda Pozuelo km 2 28220 Majadahonda (Madrid)

SPAIN

University of Oviedo Lab. Radiactividad Ambiental c/ Independencia, 13 33004 Oviedo

SWITZERLAND

Spiez Laboratory Nuclear Chemistry Austrasse 1 3700 Spiez

TURKEY

Turkish Atomic Energy Authority Radioactivity Department Sarayköy Nükleer Araştırma Ve 65100 Kazan, Ankara

UKRAINE

The Marzeev Institute of Public Health Radiation Monitoring lab Popudrenko str., 50 02094 Kyiv

UKRAINE

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

UK

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

UK

South East WaterLaboratory 3 Columbus Drive Southwood Business Park GU14 ONZ Farnborough

UK

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

South West Water Ltd Radiochemistry Bridge Road, Countess Wear EX2 7AA Exeter, Devon

UK

Thames Water Laboratory – metals Thames Water Spencer House lab 3 Manor Park, Manor farm Rd RG2 OJN Reading

Annex 7. Questionnaire

Milc questionnaire

Comparison for JRC-REM 2019 Water Proficiency Test

Questionnaire of REM 2019 Water PT on gross alpha/beta activity in water measurements

Submission Form

1. Type of your laboratory (more than one choice is possible): *

- Monitoring of nuclear facilities
- Other
- Private commercial company
- Radioactivity in the environment monitoring laboratory
- Research and development
- □ Water supply company

2. How did you learn about this PT (more than one choice is possible)? *

- Announced by the IAEA
- Invited by JRC
- Nominated by national authorities/contact points
- from the JRC website

3. Are you a member of analytical network(s) (more than one choice is possible)? *

- European NORM network
- □ IAEA-ALMERA network
- National/regional analytical network
- Other

3.1. If other, please specify here: *

- Page 1 of 6 -

4. Is your laboratory accredited for gross activity analysis according to ISO/IEC 17025? *

0	a) yes
Ο	b) no

5. Which quality management system(s) does your laboratory follow (more than one choice is possible)? *



□ ISO 9000 series

□ ISO/IEC 17025

- Internal
- No quality management system
- Other
- 5.1. If other, please specify here: *

6. Does your National Standardisation Body involve you to comment on ISO/European standards? *



7. Would you be interested in collaborating more closely with your National Standardisation Body in order to improve the ISO/European standards? *



) b) no

⁻ Page 2 of 6 -

8. How long has gross alpha/beta analysis been performed routinely at your organization (in years)? *

(number)

9. How many measurements of this type does your laboratory perform per year (approximately)? *



10. How many individual gross alpha/beta samples can you measure in the same time? *

	(number)

11. Indicate how much time needed to provide measurement results (in hours) from the moment you start preparing your sample.



12. Did you perform the test in compliance with the following standards (more than one choice is possible)? *



- □ ISO 11704
- □ ISO 9696
- □ ISO 9697
- Other
- not applicable

12.1. If other, please specify here: *

- Page 3 of 6 -

13. Which test sample preparation method did you use? *

- Direct measurement after evaporation
- Direct measurement with liquid scintillation counting
- Thermal preconcentration for liquid scintillation counting
- coprecipitation
- other

14. Which detection technique did you use? *

- a) gas-flow proportional counter
- b) liquid scintillation counter
- c) solid-stated scintillation counter
- d) other

15. Specify the typical test sample volume needed for a single analysis (in mL). *

(number)

16. Is acidifying the sample part of your procedure? *

O a) yes

O^{b) no}

17. What was the time delay between finishing the sample for measurements and starting gross measurements (in hours)?



- Page 4 of 6 -

18. Do you have a procedure to treat hygroscopic residues?



O b) no

O c) not applicable

19. Did you apply quenching correction? *



O^{b) no}

C c) not applicable

20. Did you correct for surface density of the prepared source? *



C c) not applicable

21. What is your gross alpha activity detection limit (in mBq/L)? *

(number)

22. What is your gross beta activity detection limit (in mBq/L)? *

(number)

23. Give details on your calibration procedure: radionuclides used for calibration, alpha/beta counting efficiencies, alpha-beta spillover (%), blank preparation *

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

25. Overall satisfaction score. How satisfied were you with the PT? (1: unsatisfied, 10: very satisfied) \ast



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Annex 8. Feedback and comments from the questionnaire

Lab code	Please give your brief feedback on the PT exercise (remarks,	Overall satisfaction score.
	improvements or positive feedback):	(1: unsatisfied, 10: very satisfied)
17351	Quick email answers, support to the participants, scientifical rationalism impression	10
17353	Nothing	10
17354	PT was well planned and organised	9
17355	We have had a very positive feedback.	8
17356		8
17357	positive : time required to make the measurement long enough	7
17358	Very well run considering the COVID-19 circumstances. Thanks for the submission deadline extension. Regarding Q. 17, we prepared the sample on January 16th but due to method development and validation the sample was re-run on May 20th.	10
17359	Very good for us	10
17360	NO	7
17362	very good	10
17363	We have no comments	6
17364	The PT exercise was very positive	8
17365		8
17366	We are satisfied with the PT.	10
17367		8
17368	without recommendation	7
17369	For us there was a problem of acidification of the sample. We do not work with acidified samples. Evaporated from the acidified sample moist and badly weighs	7
17370	Positive	8
17371	It is a good PT exercise.	9
17372	It is interesting to participate in this PT exercise	10
17373	except some initial problems with the software, all great	10
17374	Necessary for ISO/IEC 17025 quality management system; Point 15. I need 10 ml for alpha (ZnS(Ag) scintillation counting) and 200 ml for beta (proportional counter)	10
17376	Everything OK	10
17377	useful and necessary to improve quality results	9
17378	It is very well organized	9
17379	We have seen some differences between 2 bottles	8
17380	Ok	8
17382	NONE	10
17383		8
17384	The PT has been well organized. It would be interesting this kind of PTs was organized more frequently	9
17385	useful PT exercise for validation of our laboratory method	10
17386	well organized - adequate range of activity	9
17387	Everything is correct	10
17389	positive feedback	9
17390	the information received is ok	8

The feedback and comments are presented here as given by the participants (typos were corrected only).

Lab code	Please give your brief feedback on the PT exercise (remarks,	Overall satisfaction score.
	improvements or positive feedback):	(1: unsatisfied, 10: very satisfied)
17391	Please, I would prefer you to send me a single bottle package.	8
17392	In general, the exercise process was satisfactory, even when some problems have presented with the platform to submit the results.	10
17393	This proficiency test is very useful to the environmental radioactivity laboratories.	9
17394	Very well organized as usual. We appreciate particularly the quality of the document provided and the reporting platform	10
17396	-	8
17397	Only positive comments.	9
17398	Positive	10
17399	The term gross beta is misleading as it is not clear if low-energy betas, K40 and H3 are included or not. Announcements for sample shipping was too immediate.	9
17400	-	8
17401	adequate PT quantity, very good organization of the PT exercise and immediate reply to all emails	10
17402	no comment	9
17408	Nothing	10
17409		10
17410	-	10
17411	All si fine	10
17412	In this PT we could not preconcentrate the samples, as we do usually, because the samples pH was already too low	7
17413	due to the covid-19 emergency, there was not enough time to perform the exercise.	9
17414	Organisation of the PT was excellent. We just have one remark about the pH value of the delivered samples: would it be possible for future PT to get untreated samples or samples with a pH value > 2.5? Since we have to concentrate samples by a factor of 15 to achieve the requested detection limits, samples with pH values < 2 will have pH values < 1 after treatment. This might lead to signal loss.	9
17415	very good exercise	9
17416	it's all right	10
17429	more information/suggestion about usage of goth the aliquots	10
17430	sample is too acid. so i need to do new calibration (ph=1.3) than sample were concentrated 1 to 5. Environmental samples are usually concentrated 1 to 10	5
17431	Well organized PT. Questionnaire suitable to the purpose	10
17432	None	8
17434	We had serious troubles with one of the bottles of GAB2 (n. 39) since gross beta gave not repeatable results, despite several tests (different samples and more counting) have been made	8
17435	no remarks	8
17436	In order to measure low level gross alpha and gross beta activity it would be better to have got a not acidified sample or a acidified sample to a pH higher level. In case of the sample we received, i t was not possible to concentrate much than twice.	8
17437	Positive	8
17439	we did not apply usual thermal preconcentration $(1:25)$ due the pH of the test samples; we used 1:10 thermal preconcentration ratio	8
17440	All was well organized	9

Lab code	Please give your brief feedback on the PT exercise (remarks,	Overall satisfaction score.
	improvements or positive feedback):	(1: unsatisfied, 10: very satisfied)
17441	Nothing	10
17442	Ok	10
17443	as usual very clearly prepared PT	10
17444	PT Remarks: 1)The PT is well organized, all the needed information is provided in advance. 2)The samples are carefully prepared, packed and marked. 3)The deadline extension for results submission was helpful. Questionnaire remarks: 6)not our team members, but some persons in NCBJ are involved 8) > 25; 15) 1000 mL-alpha, 2000 mL-beta; 21) 40-60 mBq/L; 22) 80-110 mBq/L; answers concern Tech. 1	10
17445	everything is ok	9
17448	positive feedback	10
17449	It is very useful	10
17450	Some requests from the questionnaire and reporting files were not very clear formulated	9
17451	This PT exercise is very useful for us because we can see if the method we apply conduct to values which are close to ideal parameters	10
17452	Very useful for us	10
17453	Everything went very well.	10
17454	-	10
17455	-	8
17456	Positive	10
17457	It is very useful	8
17458	This PT exercise for gross alpha/beta measurements is suitable, welcome and useful for laboratories which have to measure the radioactivity of potable water . We can compare with other laboratories which are performing the same activity	9
17459	-	10
17460	not very clear which detection limit required, we have a limit of detection for each determination	9
17461	positive feedback	8
17468	informative documentation, enough samples, good instructions, enough time, no place in questionnaire for comments, no questions about no of replicates, counting statistics	8
17470	Good information about the PT execution	9
17488	-	10
17490	Thank you for the possibility to participate in this useful and well-organized event.	10
17491	well organised, we are waiting for the reference values))))	9
17492	we have used 3 different methods: evaporation and gas flow counting, coprecipitation and gas flow counting and thermal preconcentration and LSC. questions 15-23 differ for each method but can't be entered separately for the different methods unfortunately. the answers provided are for the coprecipitation method (except question 19).	7
17493	Participation in PT exercise is a reconfirmation of the validity of the measurements and control of the accuracy of the method: it is for sure the positive feedback:	8
17494	It is our first PT exercise, It is very interesting for us to participate, I hope that through this exercise, we will establish the good bases of our measurement technique, Thanks to Jobbagy and to JRC Geel for this opportunity	10
17495	No significant remarks, since it's our first participation.	9
17496	PT fits for purpose-external control of lab procedure	10

Lab code	Please give your brief feedback on the PT exercise (remarks,	Overall satisfaction score.
	improvements or positive feedback):	(1: unsatisfied, 10: very satisfied)
17497	The concentration of the samples GAB1 and GAB2 is not typical for drinking water.	7
17498	the acidity of the sample GAB 2 is too important and we have problem with our capsule (are	5
17500	timetable was arranged due to Covid19, thanks a lot for that flexibility	10
17501		8
17503	unusual salinity of gab1	8
17504	Difficult	8
17505	Very good	9
17506	no particular remarks, satisfactory delivery and packaging, sufficient sample quantity for testing	10
17507	The high acidification required several preparations in order to obtain a uniform deposit and in accordance with our practices.	7
17510	Highly acidic samples especially GAB 2 not representative of the water analyzed routinely.	7
17511	-	8
17512	The PT is well organised, the samples arrived on time, their activities are in the range of those found in our routine samples. The complete feedback will be given after the PT is finished.	10
17513	up to now we are quite satisfied with the PT, we had no problems, information was clear and samples arrived	8
17514		9
17515	This PT was useful for our laboratory, because we can get experience how to measure gross alpha/beta from relatively small amount of water sample.	10
17516	l am very satisfied	10
17517	It was well organised and could follow the communication easily	7
17519	Good PT, too much sample quantity: according to us 2 L (1 L for GAB 1, 1 L for GAB2) were sufficient for analyses. This produces us some difficulties for waste disposal.	8
17520	good excercise to improve our techniques and measurements	10
17521	The samples are to much acidificated and so the thermal preconcentration is not completely possible, and so could be a problem in the correct alpha/beta separation. Is important in my opinion that insert in the technical data about measurements thet the candidate indicate the ROI windows and the Radionuclides used for efficiency and type of circuit separation (PSA or PLI)	θ
17522	The samples are to much acidificated and so the thermal preconcentration is not completely possible, and so could be a problem in the correct alpha/beta separation	10
17523	great communication	9
17524	Everything was perfect.	10
17525	improvements	8
17526	The PT exercise is useful to check our gross alpha/beta measurement capabilities for low radioactivity levels	10
17527		10
17528	Residue from sample JRC-GAB2 has an elastic consistency, difficult to spread in the counting tray	9
17529	Very satisfied	10
17531	We appreciate the opportunity to participate in this PT exercise in order to monitor the validity of our results and possibly improve our gross activity analysis.	10

Lab code	Please give your brief feedback on the PT exercise (remarks,	Overall satisfaction score.			
	improvements or positive feedback):	(1: unsatisfied, 10: very satisfied)			
17532	All good	10			
17533	good organization;	10			
17534	Thank you	7			
17535	Test of IAEA-ALMERA, ERA - MRAD and LG AGUACHECK commercial company are succeed	10			
17536	Very well organised PT exercise. It has been a very interesting and educating PT exercise. We hope that such PT will be organized also in future.	10			
17537	Why not to use sample which was used for radon in water measurement	8			
17538	Would have been helpful to see reporting template/questionnaire with initial paperwork rather than waiting until it was time to report.	8			
17539	Bq/L would be a better unit. Concentrations found were within range.	9			
17540	N/A	8			
17541	Due to COVID19 restrictions it would be unfair to say very much about the PT. Seems to be well organised	8			
17542	Generally well organised and straightforward internet reporting portal.	9			
17543	Ok	10			
17544	useful to demonstrate competence for gross alpha and beta activity measurement by using gas flow proportional counter	10			
17545	The quantity of sample should be larger.	8			
17546	Procedure must be improved and quality management systems must be followed	10			
17568	As mentioned before, I considered very interesting and necessary for our laboratory	10			
17569	NTD	7			
17570	probleme de lors de l'evaporation du GAB 1	5			
17571	-	10			
17572	Natural samples are more difficult to measure exactly with evaporation methods because of radon content.	8			
17808	Good organization. Relative to samples, a slight discrepancy between bottles	8			
17809	Nothing	10			

Annex 9. Communication on preliminary results (e-mails)

Participants were informed about their performance scores by sending a preliminary report via emails. It has to be noted that the initial preliminary report had to be recalled and new version was issued since the measurement units were not correct. The e-mail history is presented below in reverse chronological order.

From: JRC GEE REM COMPARISONS <JRC-GEE-REM-COMPARISONS@ec.europa.eu> Sent: Thursday, September 3, 2020 5:26 PM Cc: HULT Mikael (JRC-GEEL) <Mikael.HULT@ec.europa.eu>; MALO Petya (JRC-GEEL) <Petya.MALO@ec.europa.eu>; JOBBAGY Viktor (JRC-GEEL) <Viktor.JOBBAGY@ec.europa.eu> Subject: AMENDED Preliminary report of REM 2019 PT on gross activity measurements in water_Ares(2020)4588612 Importance: High

Dear REM2019 participants,

First of all, we would like to thank the participants for noticing errors in the preliminary report. It was also one of the <u>the main objectives with the preliminary report to give participants possibility to point to our</u> <u>errors</u> so we encourage you to review the document.

Hereby we are sending the amended version of the REM2019 preliminary report. Please **use this version Ares(2020)4588612** and DELETE the report from yesterday (2 September 2020).

Please note that the measurement units were corrected from Bq/L to mBq/L and Table 1 caption was also modified.

If you have correction requests, they will be considered for the final report except what concerns the reported results. We draw your attention again that **changing reported results is not possible** any more.

Furthermore, we would like to give extra information on the total dissolved solid content in the REM2019_TDF file as some of you already requested it. It is not in the preliminary report but we will include it in the final report of course.

Thank you for your cooperation and patience. Your contribution helps to improve our PT service.

Best regards, Viktor Jobbagy PT coordinator

From: JRC GEE REM COMPARISONS Sent: 03 September 2020 09:45:48 Cc: HULT Mikael (JRC-GEEL); MALO Petya (JRC-GEEL); JOBBAGY Viktor (JRC-GEEL) Subject: RECALLILNG: Preliminary report of REM 2019 PT on gross activity measurements in water_Ares(2020)4562657

Dear REM2019 participants,

We would like to <u>recall the report</u> we sent yesterday due to some errors with the measuerement units (they should be mBq/L correctly in Tables and Figures). Therefore, please delete it and do not use it further. However, the shape of S-plots and the numerical values of the scores are unaffected.

A new amanded version will be issued today.

We apologise for the mistakes and the inconvenience it may cause. Thank you for your patience until then.

kind regards, Viktor Jobbagy

From: JRC GEE REM COMPARISONS Sent: 02 September 2020 16:34 Cc: HULT Mikael (JRC-GEEL); MALO Petya (JRC-GEEL); JOBBAGY Viktor (JRC-GEEL) Subject: Preliminary report of REM 2019 PT on gross activity measurements in water_Ares(2020)4562657

Dear participants,

We are sending the preliminary report of the REM2019 PT on gross activity concentration measurements in water focusing on the laboratory results and performances. Please note that the evaluation is still ongoing. Therefore, this document is **for information purposes only**.

To enable you to identify your laboratory and its performance, your confidential laboratory identification number can be generated via the attached Excel application (See *LabCode assignment sheet.xlsx* file). You just have to insert your unique participation password key you used for accessing the reporting website.

We would like to ask you to review this document and check your scores. Should you notice calculation mistakes, feel free to contact us before 18 September 2020, then we will review our documents and the correct scores will be introduced in the final technical report. We would like to draw your attention that **changing reported results is not possible** any more.

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We plan to publish the final technical report before March 2021.

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 link between the laboratories and the assigned lab codes is not revealed. We remind you that the final results and performance of each <u>nominated laboratory</u> 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 would like to remind you that a workshop and training courses will be organized at JRC-Geel. The exact date we cannot say now as it depends how the coronavirus situation evolves Europe wide. You can also consult the planning of the forthcoming REM proficiency tests on this website: https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests.

We would like to express our appreciation to everyone who participated in this REM2019 PT and for your kind collaboration. Thank you for your patience you showed in these difficult times. We hope that everyone stayed healthy and we can welcome you in the coming JRC proficiency tests.

Best regards, Viktor JOBBÁGY PT Coordinator

Mikael HULT Team Leader



European Commission Joint Research Centre (JRC), JRC-Geel Unit G.2. Standards for Nuclear Safety, Security and Safeguards Retieseweg 111 B-2440 Geel/Belgium Phone: +32-14-57-12-51 JRC-GEE-REM-COMPARISONS@ec.europa.eu EU Science Hub: <u>https://ec.europa.eu/jrc</u> REM Proficiency Tests: <u>https://remon.jrc.ec.europa.eu/Services/Proficiency-Tests</u> JRC Certified Reference Materials: <u>IRMM-426</u>, <u>EURM-800</u>, <u>EURM-801</u> Disclaimer: The views expressed are purely those of the writer and may not in any circumstances be

Disclaimer: 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 21. Participants' results of gross alpha activity concentration measurements on the JRC-GAB1 sample. Reported activity
concentration values x_i and combined standard uncertainties $u(x_i)$ are expressed in mBq/L.

	JRC-GAB1 sample; Gross alpha activity concentration measurements							
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17351	Solid state scintillation counter	0.00035	0.000005	-100	-3.33	-12.83		
17353	Proportional counter	196.4	59.4	-47	-1.57	-2.66		
17354	Proportional counter	345	25.0	-7	-0.24	-0.71		
17355	Proportional counter	211	38.0	-43	-1.44	-3.37		
17356	Proportional counter	194	25.4	-48	-1.59	-4.62		
17357	Solid state scintillation counter	313.5	26.0	-16	-0.52	-1.50		
17358	Liquid-scintillation counting	273	45.0	-27	-0.89	-1.85		
17359	Proportional counter	390	70.0	5	0.16	0.24		
17360	Solid state scintillation counter	296	29.0	-20	-0.68	-1.85		
17362	Proportional counter	251	39.0	-33	-1.08	-2.49		
17363	Solid state scintillation counter	202.6	24.2	-46	-1.52	-4.48		
17363	Solid state scintillation counter	58.7	6.3	-84	-2.81	-10.56		
17363	Solid state scintillation counter	130.7	15.3	-65	-2.16	-7.36		
17364	Proportional counter	<315						
17365	Solid state scintillation counter	233	15.8	-37	-1.25	-4.21		
17366	Proportional counter	295	43.4	-21	-0.69	-1.48		
17367	Proportional counter	255	28.6	-31	-1.05	-2.87		
17367	Proportional counter	292	29.6	-22	-0.72	-1.93		
17368	Proportional counter	510	34.5	37	1.24	3.06		
17369	Proportional counter	425	9.5	14	0.47	1.74		
17370	Proportional counter	74	18.5	-80	-2.67	-8.66		
17371	Solid state scintillation counter	275	22.5	-26	-0.87	-2.64		
17372	Proportional counter	280	5.5	-25	-0.82	-3.12		
17373	Solid state scintillation counter	393	30.0	6	0.19	0.50		

	JRC-GABL sample; Gross alpha activity concentration measurements							
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17374	Solid state scintillation counter	281	17.5	-24	-0.82	-2.69		
17374	Solid state scintillation counter	279	18.5	-25	-0.83	-2.70		
17376	Proportional counter	<202.1						
17377	Solid state scintillation counter	300	43.0	-19	-0.65	-1.39		
17377	Proportional counter	317	48.0	-15	-0.49	-0.98		
17378	Proportional counter	475	30.0	28	0.92	2.47		
17379	Proportional counter	301	47.0	-19	-0.64	-1.29		
17380	Proportional counter	264	22.0	-29	-0.97	-2.97		
17382	Solid state scintillation counter	272.4	8.8	-27	-0.89	-3.29		
17383	Solid state scintillation counter	137	13.0	-63	-2.11	-7.39		
17384	Proportional counter	299	12.5	-20	-0.65	-2.31		
17384	Proportional counter	280	28.5	-25	-0.82	-2.26		
17385	Liquid-scintillation counting	370	35.0	-1	-0.02	-0.04		
17386	Proportional counter	215.66	20.3	-42	-1.40	-4.41		
17387	Proportional counter	471.6	17.5	27	0.89	2.94		
17389	Proportional counter	191.9	7.3	-48	-1.61	-6.02		
17390	Proportional counter	0.212	0.0	-100	-3.33	-12.82		
17391	Proportional counter	207.89	13.4	-44	-1.47	-5.14		
17392	Proportional counter	270	8.0	-27	-0.91	-3.39		
17393	Solid state scintillation counter	289	14.5	-22	-0.74	-2.56		
17393	Proportional counter	182	18.5	-51	-1.70	-5.52		
17394	Proportional counter	278	28.5	-25	-0.84	-2.31		
17396	Proportional counter	127.95	14.2	-66	-2.19	-7.55		
17397	Proportional counter	417	33.0	12	0.40	1.02		
17397	Liquid-scintillation counting	283	26.0	-24	-0.80	-2.29		
17398	Liquid-scintillation counting	280	32.0	-25	-0.82	-2.13		

JRC-GAB1 sample; Gross alpha activity concentration measurements							
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score	
17399	Liquid-scintillation counting	373	55.0	0	0.01	0.02	
17399	grid-ionisation chamber	323	48.0	-13	-0.44	-0.87	
17400	Proportional counter	369	8.0	-1	-0.03	-0.10	
17400	Proportional counter	367	8.0	-1	-0.04	-0.17	
17401	Liquid-scintillation counting	330	10.0	-11	-0.38	-1.37	
17402	Liquid-scintillation counting	155	13.0	-58	-1.94	-6.83	
17408	Liquid-scintillation counting	260.281	16.4	-30	-1.00	-3.35	
17409	Liquid-scintillation counting	330	30.0	-11	-0.38	-1.01	
17410	Proportional counter	102	10.0	-73	-2.42	-8.80	
17411	Liquid-scintillation counting	467	88.0	26	0.85	1.03	
17412	Liquid-scintillation counting	269	63.0	-28	-0.92	-1.49	
17413	Liquid-scintillation counting	490	92.5	32	1.06	1.22	
17414	Liquid-scintillation counting	305	50.0	-18	-0.60	-1.16	
17415	Liquid-scintillation counting	404	22.5	9	0.29	0.87	
17416	Liquid-scintillation counting	403	74.5	8	0.28	0.39	
17429	Liquid-scintillation counting	360	12.0	-3	-0.11	-0.38	
17430	Liquid-scintillation counting	384	41.5	3	0.11	0.24	
17431	Liquid-scintillation counting	423	30.0	14	0.46	1.22	
17432	Proportional counter	370	60.0	-1	-0.02	-0.03	
17434	Liquid-scintillation counting	361	27.0	-3	-0.10	-0.28	
17435	Liquid-scintillation counting	330	44.0	-11	-0.38	-0.80	
17436	Liquid-scintillation counting	0.39	0.1	-100	-3.33	-12.81	
17437	Proportional counter	165.3	26.4	-56	-1.85	-5.28	
17439	Liquid-scintillation counting	340	65.0	-9	-0.29	-0.45	
17440	Liquid-scintillation counting	372	21.0	0	0.00	0.00	
17441	Proportional counter	200	15.0	-46	-1.54	-5.27	

SRC-OADI Sample, 01055 alpha activity concentration measurements						
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17441	Proportional counter	220	15.0	-41	-1.36	-4.66
17442	Solid state scintillation counter	334	25.1	-10	-0.34	-0.99
17443	Liquid-scintillation counting	54	10.7	-85	-2.85	-10.29
17443	Liquid-scintillation counting	85.1	6.9	-77	-2.57	-9.62
17444	Proportional counter	126	32.0	-66	-2.20	-5.70
17444	Liquid-scintillation counting	153	25.0	-59	-1.96	-5.72
17445	Liquid-scintillation counting	280	50.0	-25	-0.82	-1.59
17448	Proportional counter	146	11.0	-61	-2.03	-7.29
17449	Solid state scintillation counter	331.2	9.4	-11	-0.37	-1.34
17450	Solid state scintillation counter	217	24.2	-42	-1.39	-4.10
17451	Solid state scintillation counter	132.16	19.8	-64	-2.15	-6.83
17452	Proportional counter	<135				
17453	Proportional counter	243.9	43.8	-34	-1.15	-2.44
17453	Proportional counter	266.2	48.3	-28	-0.95	-1.88
17454	Solid state scintillation counter	170.4	22.1	-54	-1.81	-5.53
17455	Solid state scintillation counter	16	3.0	-96	-3.19	-12.21
17456	Solid state scintillation counter	109	5.0	-71	-2.36	-8.94
17456	Solid state scintillation counter	109	5.0	-71	-2.36	-8.94
17456	Solid state scintillation counter	109	5.0	-71	-2.36	-8.94
17457	Solid state scintillation counter	171.599	24.6	-54	-1.80	-5.27
17458	Proportional counter	206.1	28.7	-45	-1.49	-4.07
17459	Proportional counter	134.9872	3.8	-64	-2.12	-8.10
17460	solid state scintillation counter, evaporation, sulfatation, ignition	140.07	23.0	-62	-2.08	-6.27
17461	Proportional counter	174.72	29.0	-53	-1.77	-4.81
17468	in house m, Liquid scintillation counting	356	40.0	-4	-0.14	-0.32

	JAC-GADI Sample, Gross aq	sice erbs sample, cross apria activity concentration incustrements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score				
17468	ISO11704, UG, Liquid scintillation counting	370	27.0	-1	-0.02	-0.05				
17468	ISO11704, QS400, Liquid Scintillation counting	376	29.0	1	0.04	0.10				
17470	Liquid-scintillation counting	335	30.3	-10	-0.33	-0.88				
17488	Proportional counter	243.3	85.1	-35	-1.15	-1.43				
17490	Proportional counter	85	54.0	-77	-2.57	-4.68				
17490	Proportional counter	185	50.0	-50	-1.68	-3.24				
17491	Proportional counter	280	90.0	-25	-0.82	-0.97				
17492	Coprecipitation plus proportional counter	300	20.0	-19	-0.65	-2.04				
17492	Evaporation plus proportional counter	360	40.0	-3	-0.11	-0.24				
17492	Liquid-scintillation counting	260	30.0	-30	-1.00	-2.68				
17493	Proportional counter	691.73	69.1	86	2.86	4.27				
17494	Proportional counter	302	45.0	-19	-0.63	-1.31				
17495	Proportional counter	8416.36	6862.8	2162	72.08	1.17				
17496	Proportional counter	170.6	47.8	-54	-1.80	-3.61				
17496	Proportional counter	158.3	42.3	-57	-1.91	-4.17				
17497	Solid state scintillation counter	527	47.5	42	1.39	2.79				
17497	Solid state scintillation counter	463	42.5	24	0.82	1.77				
17497	Solid state scintillation counter	480	44.5	29	0.97	2.03				
17498	Proportional counter	350	47.0	-6	-0.20	-0.40				
17498	Proportional counter	357	48.5	-4	-0.13	-0.27				
17500	Proportional counter	359	91.5	-3	-0.12	-0.14				
17501	Proportional counter	420	60.0	13	0.43	0.72				
17503	Proportional counter	411	68.0	10	0.35	0.53				
17504	Proportional counter	275	45.0	-26	-0.87	-1.81				

	JRC-GAB1 sample; Gross al	pha activity c	oncentration	measureme	ents	
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17505	Proportional counter	180	40.0	-52	-1.72	-3.89
17505	Proportional counter	210	50.0	-44	-1.45	-2.80
17506	Solid state scintillation counter	0.0006132	0.0	-100	-3.33	-12.83
17510	Proportional counter	329	61.5	-12	-0.39	-0.63
17511	Proportional counter	267	30.5	-28	-0.94	-2.49
17512	Proportional counter	164	21.5	-56	-1.86	-5.76
17513	Proportional counter	259	35.0	-30	-1.01	-2.49
17513	preparation of 200ml with H2O2 and HNO3, proportional counter	295	50.0	-21	-0.69	-1.33
17514	Liquid-scintillation counting	403.7	20.5	9	0.28	0.89
17515	Proportional counter	<318				
17515	Solid state scintillation counter	<444				
17516	PIPS detector	239	14.5	-36	-1.19	-4.10
17517	Liquid-scintillation counting	439.91	30.5	18	0.61	1.61
17519	Proportional counter	113.1	41.1	-70	-2.32	-5.15
17519	Liquid-scintillation counting	22.3	3.4	-94	-3.13	-11.98
17519	Liquid-scintillation counting	123	12.0	-67	-2.23	-7.93
17520	Proportional counter	400	100.0	8	0.25	0.27
17520	Liquid-scintillation counting	290	20.0	-22	-0.73	-2.33
17521	Liquid-scintillation counting	290.7	27.2	-22	-0.73	-2.05
17522	Liquid-scintillation counting	290.7	27.2	-22	-0.73	-2.05
17523	Proportional counter	216	15.3	-42	-1.40	-4.76
17524	Proportional counter	233.11	35.0	-37	-1.24	-3.06
17525	Solid state scintillation counter	148.6	18.5	-60	-2.00	-6.49
17525	Solid state scintillation counter	223.2	17.0	-40	-1.33	-4.43
17526	Proportional counter	656	197.0	76	2.54	1.43
17527	Solid state scintillation counter	95	25.0	-74	-2.48	-7.23

	JRC-GAB1 sample; Gross al	oha activity	concentration	measurem	ents	
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17527	Solid state scintillation counter	<100				
17528	Proportional counter	7.02	0.9	-98	-3.27	-12.58
17529	Liquid-scintillation counting	372	11.0	0	0.00	0.00
17531	Proportional counter	54	23.0	-85	-2.85	-8.59
17532	Proportional counter	<100				
17533	Proportional counter	128.6	20.3	-65	-2.18	-6.88
17534	Liquid-scintillation counting	320	100.0	-14	-0.47	-0.50
17535	Proportional counter	340	35.0	-9	-0.29	-0.70
17536	Liquid-scintillation counting	1000	250.0	169	5.63	2.50
17537	Liquid-scintillation counting	358	32.0	-4	-0.13	-0.32
17537	Liquid-scintillation counting	382	33.0	3	0.09	0.23
17538	Proportional counter	216.6	15.2	-42	-1.39	-4.74
17539	Gas proportional counting	129.5	18.0	-65	-2.17	-7.10
17540	Proportional counter	222	21.2	-40	-1.34	-4.18
17540	Proportional counter	308.1	29.4	-17	-0.57	-1.55
17541	Solid state scintillation counter	169	26.0	-55	-1.82	-5.21
17541	Proportional counter	195	29.0	-48	-1.59	-4.32
17542	Proportional counter Ref: Pu-242	336	43.5	-10	-0.32	-0.69
17542	Proportional counter Ref: Am-241	254	32.5	-32	-1.06	-2.71
17543	Liquid-scintillation counting	363	27.0	-2	-0.08	-0.23
17544	Proportional counter	324	35.0	-13	-0.43	-1.06
17545	Proportional counter	220	20.0	-41	-1.36	-4.31
17546	Proportional counter	313	24.0	-16	-0.53	-1.57
17568	Solid state scintillation counter	336	46.0	-10	-0.32	-0.66
17572	Method for total U & TRU from high salinity solutions, separation with DGA resign, measured in PIPS det.	90	10.0	-76	-2.53	-9.19

Lab code	Technique			x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i − score	zeta _i - score
17572	Evaporatio	n and PIPS		191	27.0	-49	-1.62	-4.57
17572	LB4200 V=50ml	proportional	counter	160	54.0	-57	-1.90	-3.46
17808	Proportiona	al counter		252	31.4	-32	-1.08	-2.81
17809	Proportiona	al counter		64.8	87.3	-83	-2.75	-3.34

Table 22. Participants' results of gross beta activity concentration measurements on the JRC-GAB1 sample. Reported activity
concentration values x_i and combined standard uncertainties $u(x_i)$ are expressed in mBq/L.

	JRC-GAB1 sample; Gross beta activity concentration measurements										
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score					
17351	Proportional counter	<0.00036									
17353	Proportional counter	238.7	58.1	-28	-0.94	-1.47					
17354	Proportional counter	425	37.0	28	0.92	2.01					
17355	Proportional counter	566	49.5	70	2.33	4.13					
17356	Proportional counter	286.5	28.8	-14	-0.47	-1.18					
17357	Proportional counter	416.5	58.5	25	0.84	1.30					
17358	Liquid-scintillation counting	513	104.0	54	1.80	1.68					
17359	Proportional counter	450	50.0	35	1.17	2.06					
17360	Solid state scintillation counter	458	45.0	38	1.25	2.38					
17362	Proportional counter	266	20.0	-20	-0.67	-1.99					
17363	Solid state scintillation counter	848.5	13.3	155	5.16	17.14					
17363	Solid state scintillation counter	741.3	11.6	123	4.09	13.89					
17363	Solid state scintillation counter	794.9	12.5	139	4.62	15.54					
17364	Proportional counter	342	21.2	3	0.09	0.26					
17365	Proportional counter	301	11.0	-10	-0.32	-1.10					
17365	Proportional counter	278	10.0	-17	-0.55	-1.91					
17366	Proportional counter	307	27.6	-8	-0.26	-0.67					
17367	Proportional counter	312	15.8	-6	-0.21	-0.67					
17367	Proportional counter	365	16.8	10	0.32	1.01					
17368	Proportional counter	427	19.0	28	0.94	2.85					
17369	Proportional counter	458	8.5	38	1.25	4.42					
17370	Proportional counter	350	85.0	5	0.17	0.19					
17371	Proportional counter	290	17.0	-13	-0.43	-1.35					
17372	Proportional counter	269	30.5	-19	-0.64	-1.57					
17373	Proportional counter	324	22.0	-3	-0.09	-0.26					

Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	Zi- score	zeta _i - score
17374	Proportional counter	277	12.0	-17	-0.56	-1.90
17374	Proportional counter	315	13.0	-5	-0.18	-0.60
17376	Proportional counter	493.9	90.0	48	1.61	1.71
17377	Proportional counter	282	30.0	-15	-0.51	-1.26
17377	residual beta	95	30.0	-71	-2.38	-5.90
17378	Proportional counter	312	60.0	-6	-0.21	-0.32
17379	Proportional counter	318	50.0	-5	-0.15	-0.26
17380	Proportional counter	246	14.0	-26	-0.87	-2.86
17382	Proportional counter	285.77	17.5	-14	-0.47	-1.47
17383	Proportional counter	342	47.5	3	0.09	0.16
17384	Proportional counter	338	37.5	2	0.05	0.11
17385	Liquid-scintillation counting	420	35.0	26	0.87	1.97
17386	Proportional counter	311.51	14.9	-6	-0.22	-0.70
17387	Proportional counter	238.8	7.3	-28	-0.94	-3.37
17389	Proportional counter	363.9	17.0	9	0.31	0.97
17390	Proportional counter	0.986	0.0	-100	-3.32	-12.30
17391	Proportional counter	339.18	5.2	2	0.06	0.22
17392	Proportional counter	423	8.5	27	0.90	3.18
17393	Proportional counter	316	7.5	-5	-0.17	-0.61
17394	Proportional counter	407	39.0	22	0.74	1.56
17396	Proportional counter	289.5	25.5	-13	-0.44	-1.17
17397	Proportional counter	451	35.0	35	1.18	2.67
17397	Liquid-scintillation counting	586	18.0	76	2.53	7.80
17398	Liquid-scintillation counting	403	72.0	21	0.70	0.91
17399	Liquid-scintillation counting	486	65.0	46	1.53	2.17
17401	Liquid-scintillation counting	350	10.0	5	0.17	0.59

	JRC-GAB1 sample; Gro	ss beta activity	concentration	measureme	ents	
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17402	Liquid-scintillation counting	682	62.5	105	3.49	5.13
17408	Liquid-scintillation counting	505.716	26.1	52	1.73	4.60
17409	Liquid-scintillation counting	280	45.0	-16	-0.53	-1.01
17410	Proportional counter	385	31.0	16	0.52	1.26
17411	Liquid-scintillation counting	<760				
17412	Liquid-scintillation counting	603	227.5	81	2.70	1.18
17413	Liquid-scintillation counting	<1043				
17414	Liquid-scintillation counting	485	105.0	46	1.52	1.40
17415	Liquid-scintillation counting	580	57.5	74	2.47	3.89
17416	Liquid-scintillation counting	584	291.0	75	2.51	0.86
17429	Liquid-scintillation counting	408	22.0	23	0.75	2.15
17430	Liquid-scintillation counting	<290				
17431	Liquid-scintillation counting	711	75.5	114	3.78	4.71
17432	Proportional counter	343	34.0	3	0.10	0.23
17434	Liquid-scintillation counting	465	86.0	40	1.32	1.46
17435	Liquid-scintillation counting	550	115.0	65	2.17	1.84
17436	Liquid-scintillation counting	<0.4				
17437	Proportional counter	310.1	14.0	-7	-0.23	-0.75
17439	Liquid-scintillation counting	690	230.0	107	3.57	1.54
17440	Liquid-scintillation counting	438	32.5	32	1.05	2.49
17441	Proportional counter	830	15.0	149	4.97	16.09
17441	Proportional counter	450	15.0	35	1.17	3.79
17442	Proportional counter	349	27.5	5	0.16	0.42
17443	Liquid-scintillation counting	906	39.0	172	5.74	12.08
17443	Liquid-scintillation counting	994	72.0	198	6.62	8.60
17444	Proportional counter	350	70.0	5	0.17	0.23

	JRC-GAB1 sample; Gross beta activity concentration measurements									
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score				
17444	Liquid-scintillation counting	630	55.0	89	2.97	4.85				
17445	Liquid-scintillation counting	550	110.0	65	2.17	1.92				
17448	Proportional counter	274	20.6	-18	-0.59	-1.74				
17449	Solid state scintillation counter	292.4	8.8	-12	-0.41	-1.43				
17450	Solid state scintillation counter	269	53.0	-19	-0.64	-1.08				
17451	Solid state scintillation counter	179.67	47.0	-46	-1.53	-2.83				
17452	Proportional counter	<455								
17453	Proportional counter	315.6	38.4	-5	-0.17	-0.37				
17453	Proportional counter	243.5	36.2	-27	-0.90	-1.98				
17454	Solid state scintillation counter	269.7	38.0	-19	-0.63	-1.36				
17455	Solid state scintillation counter	195.2	22.5	-41	-1.38	-3.92				
17456	Solid state scintillation counter	420	31.5	26	0.87	2.10				
17456	Solid state scintillation counter	420	31.5	26	0.87	2.10				
17456	Solid state scintillation counter	420	31.5	26	0.87	2.10				
17457	Solid state scintillation counter	268.549	33.6	-19	-0.65	-1.49				
17458	Proportional counter	345.5	11.6	4	0.13	0.43				
17459	Proportional counter	315.0164	14.1	-5	-0.18	-0.59				
17460	solid state scintillation counter, evaporation, sulfatation, ignition	315.16	58.3	-5	-0.18	-0.28				
17461	Proportional counter	267.25	40.4	-20	-0.66	-1.35				
17468	in house method, Liquid scintillation counting	431	64.0	29	0.98	1.41				
17468	ISO11704, UG, Liquid scintillation counting	448	33.0	35	1.15	2.70				
17468	ISO11704, QS400, Liquid Scintillation counting	537	91.0	61	2.04	2.15				
17470	Liquid-scintillation counting	473	43.0	42	1.40	2.76				
17488	Proportional counter	336.7	61.4	1	0.04	0.06				

Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	Zi- score	zeta _i - score
17490	Proportional counter	354	51.0	6	0.21	0.36
17490	Proportional counter	146	33.0	-56	-1.87	-4.39
17491	Proportional counter	410	85.0	23	0.77	0.86
17492	Coprecipitation plus proportional counter	311	13.0	-7	-0.22	-0.73
17492	Evaporation plus proportional counter	380	40.0	14	0.47	0.97
17492	Liquid-scintillation counting	650	70.0	95	3.17	4.23
17493	Proportional counter	1439.61	180.0	332	11.08	6.08
17494	Proportional counter	341	34.0	2	0.08	0.18
17495	Proportional counter	7200.1	4402.8	2062	68.74	1.56
17496	Proportional counter	317.7	66.5	-5	-0.15	-0.21
17496	Proportional counter	294.1	63.7	-12	-0.39	-0.56
17497	Proportional counter	281	48.0	-16	-0.52	-0.94
17497	Proportional counter	295	45.5	-11	-0.38	-0.72
17497	Proportional counter	357	50.5	7	0.24	0.42
17500	Proportional counter	329	42.5	-1	-0.04	-0.08
17501	Proportional counter	525	65.0	58	1.92	2.73
17503	Proportional counter	396	72.0	19	0.63	0.82
17504	Proportional counter	297	46.0	-11	-0.36	-0.67
17505	Proportional counter	248	21.0	-26	-0.85	-2.48
17505	Liquid-scintillation counting	270	22.0	-19	-0.63	-1.81
17507	Proportional counter	267	65.0	-20	-0.66	-0.94
17510	Proportional counter	281	38.0	-16	-0.52	-1.12
17511	Proportional counter	389	41.0	17	0.56	1.14
17512	Proportional counter	356	32.5	7	0.23	0.54
17513	Proportional counter	251	135.0	-25	-0.82	-0.60

	JRC-GAB1 sample; Gross be	eta activity o	oncentration	measureme	nts	
Lab code	Technique	<i>х_і</i> (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17513	preparation of 200ml with H2O2 and HNO3, proportional counter	<690				
17514	Liquid-scintillation counting	480	21.5	44	1.47	4.26
17515	Proportional counter	<600				
17515	Solid state scintillation counter	<343				
17516	PIPS detector	319	48.0	-4	-0.14	-0.25
17517	Liquid-scintillation counting	544.42	28.0	63	2.12	5.44
17519	Proportional counter	322.3	52.6	-3	-0.11	-0.18
17519	Liquid-scintillation counting	774	77.0	132	4.41	5.40
17519	Liquid-scintillation counting	904	90.0	171	5.72	6.08
17520	Proportional counter	800	150.0	140	4.67	3.06
17520	Liquid-scintillation counting	500	30.0	50	1.67	4.14
17521	Liquid-scintillation counting	711.2	77.3	114	3.79	4.62
17522	Liquid-scintillation counting	711.2	77.3	114	3.79	4.62
17523	Proportional counter	160	7.5	-52	-1.73	-6.18
17524	Proportional counter	438.02	65.7	32	1.05	1.48
17525	Solid state scintillation counter	400.7	23.8	20	0.68	1.88
17526	Proportional counter	272	82.0	-18	-0.61	-0.71
17527	Solid state scintillation counter	5800	1650.0	1642	54.72	3.31
17527	Solid state scintillation counter	6200	1810.0	1762	58.73	3.24
17528	Proportional counter	236.55	2.8	-29	-0.97	-3.55
17529	Liquid-scintillation counting	136	10.0	-59	-1.97	-6.84
17531	Proportional counter	214	30.0	-36	-1.19	-2.95
17532	Proportional counter	144	43.0	-57	-1.89	-3.72
17533	Proportional counter	314.76	21.0	-5	-0.18	-0.53
17534	Liquid-scintillation counting	<790				
17535	Proportional counter	345	30.0	4	0.12	0.30

	JRC-GAB1 sample; Gross be	eta activity co	oncentration n	neasuremei	nts	
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17536	Liquid-scintillation counting	1450	360.0	335	11.18	3.09
17537	Liquid-scintillation counting	407	106.0	22	0.74	0.68
17537	Liquid-scintillation counting	432	50.0	30	0.99	1.74
17538	Proportional counter	288.1	10.6	-13	-0.45	-1.55
17539	Gas proportional counting	250.75	19.8	-25	-0.82	-2.46
17540	Proportional counter	281.2	29.2	-16	-0.52	-1.30
17540	Proportional counter	<280.0				
17541	Solid state scintillation counter	914	165.0	174	5.82	3.47
17541	Proportional counter	313	36.5	-6	-0.20	-0.44
17542	Proportional counter Ref: Cs-137	408	31.0	23	0.75	1.82
17542	Proportional counter Ref: K-40	349	27.0	5	0.16	0.42
17543	Liquid-scintillation counting	384	55.0	15	0.51	0.83
17544	Proportional counter	640	28.0	92	3.07	7.89
17545	Proportional counter	470	32.5	41	1.37	3.24
17546	Proportional counter	424	49.0	27	0.91	1.63
17568	Proportional counter	322.9	15.0	-3	-0.10	-0.33
17569	Proportional counter	430	110.0	29	0.97	0.86
17571	Proportional counter	286	37.2	-14	-0.47	-1.02
17572	LB4200 proportional counter V=200ml	370	32.0	11	0.37	0.88
17572	LB4200 proportional counter V=50ml	736	139.0	121	4.03	2.85
17808	Proportional counter	365.2	47.2	10	0.32	0.59
17809	Proportional counter	359.8	31.7	8	0.27	0.64

Table 23. Participants' results of gross alpha activity concentration measurements on the JRC-GAB2 sample. Reported activity
concentration values x_i and combined standard uncertainties $u(x_i)$ are expressed in mBq/L.

	JRC-GAB2 sample; Gross a	lpha activity	concentration	measureme	ents	
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17351	Solid state scintillation counter	0.00076	0.00001	-100	-5.00	-21.50
17353	Proportional counter	594	54.4	-19	-0.94	-2.14
17354	Proportional counter	834	35.0	14	0.70	2.11
17355	Proportional counter	402	38.0	-45	-2.25	-6.45
17356	Proportional counter	620.3	44.4	-15	-0.76	-1.98
17357	Solid state scintillation counter	814	64.5	11	0.57	1.14
17358	Liquid-scintillation counting	836	102.0	14	0.72	0.98
17359	Proportional counter	950	120.0	30	1.50	1.76
17360	Solid state scintillation counter	57	5.7	-92	-4.61	-19.55
17362	Proportional counter	558	87.0	-24	-1.18	-1.85
17363	Solid state scintillation counter	457.6	25.6	-37	-1.87	-6.42
17363	Solid state scintillation counter	655.8	30.5	-10	-0.51	-1.65
17363	Solid state scintillation counter	854	35.3	17	0.84	2.51
17364	Proportional counter	639	43.9	-13	-0.63	-1.66
17365	Solid state scintillation counter	319	15.4	-56	-2.82	-11.04
17366	Proportional counter	1100	153.1	50	2.52	2.35
17367	Proportional counter	558	23.5	-24	-1.18	-4.19
17367	Proportional counter	570	23.5	-22	-1.10	-3.90
17368	Proportional counter	1620	110.0	122	6.08	7.72
17369	Proportional counter	792	15.0	8	0.42	1.64
17370	Proportional counter	360	68.5	-51	-2.54	-4.85
17371	Solid state scintillation counter	429	24.0	-41	-2.07	-7.26
17372	Proportional counter	680	19.0	-7	-0.35	-1.31
17373	Solid state scintillation counter	725	54.0	-1	-0.04	-0.09
17374	Solid state scintillation counter	681	24.5	-7	-0.34	-1.19

	JRC-GAB2 sample; Gross alpha activity concentration measurements									
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i − score	zeta _i - score				
17374	Solid state scintillation counter	689	24.5	-6	-0.29	-1.00				
17376	Proportional counter	356.7	79.4	-51	-2.56	-4.33				
17377	Solid state scintillation counter	521	70.0	-29	-1.44	-2.70				
17377	Proportional counter	611	84.0	-16	-0.82	-1.32				
17378	Proportional counter	465	35.0	-36	-1.82	-5.45				
17379	Proportional counter	691	47.5	-5	-0.27	-0.68				
17380	Proportional counter	439	31.0	-40	-2.00	-6.35				
17382	Solid state scintillation counter	707.24	22.5	-3	-0.16	-0.58				
17383	Solid state scintillation counter	338	23.5	-54	-2.69	-9.51				
17384	Proportional counter	731	27.5	0	0.00	0.00				
17385	Liquid-scintillation counting	810	70.0	11	0.54	1.02				
17386	Proportional counter	354.7	19.7	-51	-2.57	-9.58				
17387	Proportional counter	391.8	10.3	-46	-2.32	-9.55				
17389	Proportional counter	578.8	18.6	-21	-1.04	-3.93				
17390	Proportional counter	0.531	0.030	-100	-5.00	-21.48				
17391	Proportional counter	470.86	11.9	-36	-1.78	-7.22				
17392	Proportional counter	723	12.5	-1	-0.05	-0.22				
17393	Proportional counter	586	51.5	-20	-0.99	-2.35				
17393	Solid state scintillation counter	594	26.0	-19	-0.94	-3.20				
17394	Proportional counter	663	54.0	-9	-0.47	-1.07				
17396	Proportional counter	239.2	12.9	-67	-3.36	-13.53				
17397	Liquid-scintillation counting	687	13.0	-6	-0.30	-1.21				
17397	Proportional counter	794	52.0	9	0.43	1.01				
17398	Liquid-scintillation counting	682	90.0	-7	-0.34	-0.51				
17399	grid-ionisation chamber	710	110.0	-3	-0.14	-0.18				
17399	Liquid-scintillation counting	740	110.0	1	0.06	0.08				
	JRC-GAB2 sample; Gross alpha activity concentration measurements									
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Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score				
17400	Proportional counter	683	10.0	-7	-0.33	-1.35				
17400	Proportional counter	698	10.0	-5	-0.23	-0.93				
17401	Liquid-scintillation counting	620	15.0	-15	-0.76	-2.99				
17402	Liquid-scintillation counting	654	54.0	-11	-0.53	-1.21				
17408	Liquid-scintillation counting	680.842	38.2	-7	-0.34	-0.98				
17409	Liquid-scintillation counting	780	85.0	7	0.34	0.54				
17410	Proportional counter	395	31.0	-46	-2.30	-7.30				
17411		817	105.0	12	0.59	0.78				
17412	Liquid-scintillation counting	622	86.5	-15	-0.75	-1.17				
17413	Liquid-scintillation counting	653	95.0	-11	-0.53	-0.77				
17414	Liquid-scintillation counting	705	80.0	-4	-0.18	-0.30				
17415	Liquid-scintillation counting	800	42.5	9	0.47	1.27				
17416	Liquid-scintillation counting	940	87.5	29	1.43	2.23				
17429	Liquid-scintillation counting	664	16.0	-9	-0.46	-1.78				
17430	Liquid-scintillation counting	594	44.5	-19	-0.94	-2.45				
17431	Liquid-scintillation counting	774	40.5	6	0.29	0.81				
17432	Proportional counter	1110	125.0	52	2.59	2.93				
17434	Liquid-scintillation counting	827	44.0	13	0.66	1.73				
17435	Liquid-scintillation counting	740	39.5	1	0.06	0.17				
17436	Liquid-scintillation counting	0.81	0.055	-100	-4.99	-21.48				
17437	Proportional counter	767.4	70.0	5	0.25	0.47				
17439	Liquid-scintillation counting	740	50.0	1	0.06	0.15				
17440	Liquid-scintillation counting	774	40.0	6	0.29	0.82				
17441	Proportional counter	480	15.0	-34	-1.72	-6.75				
17441	Proportional counter	680	15.0	-7	-0.35	-1.37				
17442		833	61.5	14	0.70	1.45				

JRC-GAB2 sample; Gross alpha activity concentration measurements									
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score			
17443	Liquid-scintillation counting	487	38.0	-33	-1.67	-4.79			
17443	Liquid-scintillation counting	554	44.0	-24	-1.21	-3.18			
17444	Proportional counter	231	32.0	-68	-3.42	-10.71			
17444	Liquid-scintillation counting	650	70.0	-11	-0.55	-1.04			
17445	Liquid-scintillation counting	820	120.0	12	0.61	0.71			
17448	Proportional counter	670	50.3	-8	-0.42	-1.01			
17449	Solid state scintillation counter	87.69	2.5	-88	-4.40	-18.87			
17450	Solid state scintillation counter	777	19.3	6	0.31	1.18			
17451	Solid state scintillation counter	363.77	12.9	-50	-2.51	-10.10			
17452	Proportional counter	337.74	40.3	-54	-2.69	-7.46			
17453	Proportional counter	560.1	53.6	-23	-1.17	-2.69			
17453		887.6	86.8	21	1.07	1.68			
17454	Solid state scintillation counter	572.9	42.3	-22	-1.08	-2.91			
17455	Solid state scintillation counter	21.9	4.175	-97	-4.85	-20.70			
17456	Solid state scintillation counter	146	3.5	-80	-4.00	-17.12			
17456	Solid state scintillation counter	146	3.5	-80	-4.00	-17.12			
17456	Solid state scintillation counter	146	3.5	-80	-4.00	-17.12			
17457	Solid state scintillation counter	670.71	42.4	-8	-0.41	-1.11			
17458	Proportional counter	675.3	16.4	-8	-0.38	-1.48			
17459	Proportional counter	348.0516	10.2	-52	-2.62	-10.78			
17460	solid state scintillation counter, evaporation, sulfatation, ignition	635.04	31.7	-13	-0.66	-2.06			
17461	Proportional counter	637.75	39.3	-13	-0.64	-1.80			
17468	ISO11704, UG, Liquid scintillation counting	725	37.0	-1	-0.04	-0.12			
17468	ISO11704, QS400, Liquid Scintillation counting	752	54.0	3	0.14	0.33			
17468	in house m, Liquid scintillation	769	38.0	5	0.26	0.75			

JRC-GAB2 sample; Gross alpha activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
	counting							
17470	Liquid-scintillation counting	710	66.7	-3	-0.14	-0.28		
17488	Proportional counter	884	210.0	21	1.05	0.72		
17490	Proportional counter	698	53.0	-5	-0.23	-0.52		
17490	Proportional counter	846	47.0	16	0.79	1.98		
17491	Proportional counter	660	210.0	-10	-0.49	-0.33		
17492	Evaporation plus proportional counter	600	60.0	-18	-0.90	-1.90		
17492	Liquid-scintillation counting	680	70.0	-7	-0.35	-0.66		
17492	Coprecipitation plus proportional counter	880	50.0	20	1.02	2.46		
17493	Proportional counter	397.13	39.7	-46	-2.28	-6.39		
17494	Proportional counter	576	72.0	-21	-1.06	-1.95		
17495	Proportional counter	8416.36	6862.8	1051	52.57	1.12		
17496		667.8	151.4	-9	-0.43	-0.41		
17496	Proportional counter	741.6	170.7	1	0.07	0.06		
17497	Solid state scintillation counter	854	70.5	17	0.84	1.57		
17497	Solid state scintillation counter	957	78.5	31	1.55	2.64		
17497	Solid state scintillation counter	995	81.0	36	1.81	3.01		
17498	Proportional counter	807	93.0	10	0.52	0.77		
17498	Proportional counter	873	121.5	19	0.97	1.13		
17500	Proportional counter	749	120.0	2	0.12	0.14		
17501	Proportional counter	590	72.5	-19	-0.96	-1.76		
17503	Proportional counter	725	87.0	-1	-0.04	-0.06		
17504		784	59.5	7	0.36	0.77		
17505	Proportional counter	510	105.0	-30	-1.51	-2.00		
17505	Proportional counter	560	110.0	-23	-1.17	-1.49		

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JRC-GAB2 sample; Gross alpha activity concentration measurements									
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score			
17506	Solid state scintillation counter	0.000186	0.0000397	-100	-5.00	-21.50			
17510	Proportional counter	747	104.5	2	0.11	0.15			
17511	Proportional counter	510	53.5	-30	-1.51	-3.49			
17512	Proportional counter	443	58.5	-39	-1.97	-4.26			
17513	Proportional counter	706	86.0	-3	-0.17	-0.27			
17513	preparation of 200ml with H2O2 and HNO3, proportional counter	792	106.0	8	0.42	0.55			
17514	Liquid-scintillation counting	760	30.0	4	0.20	0.64			
17515	Proportional counter	856	138.0	17	0.85	0.88			
17515	Solid state scintillation counter	1247	194.0	71	3.53	2.62			
17516	PIPS detector	650	39.0	-11	-0.55	-1.57			
17517	Liquid-scintillation counting	1449.516	64.6	98	4.91	9.84			
17519	Liquid-scintillation counting	183	18.0	-75	-3.75	-14.24			
17519	Liquid-scintillation counting	317	32.0	-57	-2.83	-8.87			
17519	Proportional counter	541.3	93.2	-26	-1.30	-1.91			
17520	Proportional counter	600	150.0	-18	-0.90	-0.85			
17520	Liquid-scintillation counting	700	40.0	-4	-0.21	-0.59			
17521	Liquid-scintillation counting	740.2	37.1	1	0.06	0.18			
17522	Liquid-scintillation counting	677.7	33.6	-7	-0.36	-1.12			
17523	Proportional counter	412	16.6	-44	-2.18	-8.43			
17524	Proportional counter	839.51	125.9	15	0.74	0.83			
17525	Solid state scintillation counter	520.4	50.7	-29	-1.44	-3.45			
17525	Solid state scintillation counter	788.1	37.5	8	0.39	1.13			
17526	Proportional counter	2207	552.0	202	10.10	2.67			
17527	Solid state scintillation counter	570	160.0	-22	-1.10	-0.98			
17527	Solid state scintillation counter	815	250.0	11	0.57	0.33			
17528	Proportional counter	110	2.6	-85	-4.25	-18.21			

JRC-GAB2 sample; Gross alpha activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17529	Liquid-scintillation counting	753	15.0	3	0.15	0.59		
17531		146	14.0	-80	-4.00	-15.91		
17532	Proportional counter	350	180.0	-52	-2.61	-2.08		
17533	Proportional counter	371.82	29.2	-49	-2.46	-8.01		
17534	Liquid-scintillation counting	680	90.0	-7	-0.35	-0.53		
17535	Proportional counter	700	40.0	-4	-0.21	-0.59		
17536	Liquid-scintillation counting	290	73.0	-60	-3.02	-5.48		
17537	Liquid-scintillation counting	753	60.0	3	0.15	0.32		
17537	Liquid-scintillation counting	762	53.0	4	0.21	0.49		
17538	Proportional counter	891.4	15.2	22	1.10	4.30		
17539	Gas proportional counting	484.25	69.1	-34	-1.69	-3.20		
17540	Proportional counter	818.2	78.0	12	0.60	1.03		
17540	Proportional counter	830.4	79.2	14	0.68	1.15		
17541	Solid state scintillation counter	329	28.5	-55	-2.75	-9.06		
17541	Proportional counter	403	38.0	-45	-2.24	-6.43		
17542	Proportional counter Ref: Am-241	700	55.0	-4	-0.21	-0.48		
17542	Proportional counter Ref: Pu-242	1030	85.0	41	2.05	3.27		
17543	Liquid-scintillation counting	760	50.5	4	0.20	0.48		
17544	Proportional counter	1531	65.0	109	5.47	10.91		
17545	Proportional counter	365	15.0	-50	-2.50	-9.85		
17546	Proportional counter	573	22.0	-22	-1.08	-3.90		
17568	Solid state scintillation counter	788	93.5	8	0.39	0.57		
17572	Evaporation and PIPS	521	40.0	-29	-1.44	-4.00		
17572	LB4200 proportional counter V=50ml	810	120.0	11	0.54	0.63		
17572	Method for total U & TRU from high salinityc solutions, separation with DGA resign, measured in PIPS det.	927	47.0	27	1.34	3.38		

JRC-GAB2 sample; Gross alpha activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17808	Proportional counter	543.8	19.8	-26	-1.28	-4.76		
17809	Proportional counter	505.5	35.4	-31	-1.54	-4.60		

JRC-GAB2 sample; Gross beta activity concentration measurements									
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score			
17351	Proportional counter	0.00141	0.00002	-100	-5.00	-30.38			
17353	Proportional counter	1151.1	66.1	-29	-1.43	-5.42			
17354	Proportional counter	1757	54.0	9	0.46	1.94			
17355	Proportional counter	2124	119.0	32	1.60	3.95			
17356	Proportional counter	1247.3	50.7	-23	-1.13	-4.95			
17357	Proportional counter	1560	176.0	-3	-0.16	-0.27			
17358	Liquid-scintillation counting	1592	222.0	-1	-0.06	-0.08			
17359	Proportional counter	1640	120.0	2	0.09	0.23			
17360	Solid state scintillation counter	820	82.0	-49	-2.45	-8.09			
17362	Proportional counter	1245	94.0	-23	-1.13	-3.38			
17363	Solid state scintillation counter	924.4	14.5	-43	-2.13	-12.48			
17363	Solid state scintillation counter	1074.2	16.4	-33	-1.66	-9.66			
17363	Solid state scintillation counter	1223.9	18.3	-24	-1.20	-6.89			
17364	Proportional counter	1310	30.4	-19	-0.93	-4.91			
17365	Proportional counter	1221	19.0	-24	-1.21	-6.91			
17365	Proportional counter	1286	20.0	-20	-1.01	-5.72			
17366	Proportional counter	1200	102.0	-25	-1.27	-3.57			
17367	Proportional counter	1119	21.9	-30	-1.52	-8.56			
17367	Proportional counter	1199	23.5	-26	-1.28	-7.09			
17368	Proportional counter	1210	55.0	-25	-1.24	-5.24			
17369	Proportional counter	1360	25.0	-16	-0.78	-4.27			
17370	Proportional counter	2000	268.0	24	1.21	1.43			
17371	Proportional counter	1148	56.5	-29	-1.43	-5.96			
17372	Proportional counter	1273	46.0	-21	-1.05	-4.80			

Table 24. Participants' results of gross beta activity concentration measurements on the JRC-GAB2 sample. Reported activity concentration values x_i and combined standard uncertainties $u(x_i)$ are expressed in mBq/L.

JRC-GAB2 sample; Gross beta activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17373	Proportional counter	1563	34.0	-3	-0.15	-0.75		
17374	Proportional counter	1237	23.0	-23	-1.16	-6.46		
17374	Proportional counter	1239	23.0	-23	-1.15	-6.42		
17376	Proportional counter	1392	205.0	-14	-0.68	-1.03		
17377	residual beta	927	83.0	-42	-2.12	-6.94		
17377	Proportional counter	1225	83.0	-24	-1.20	-3.91		
17378	Proportional counter	1234	250.0	-23	-1.17	-1.47		
17379	Proportional counter	1590	47.5	-1	-0.06	-0.28		
17380	Proportional counter	1230	32.0	-24	-1.18	-6.14		
17382	Proportional counter	1283.63	16.6	-20	-1.01	-5.88		
17383	Proportional counter	1281	73.0	-20	-1.02	-3.65		
17384	Proportional counter	1480	57.5	-8	-0.40	-1.66		
17385	Liquid-scintillation counting	1460	120.0	-9	-0.47	-1.14		
17386	Proportional counter	1294.22	22.4	-20	-0.98	-5.49		
17387	Proportional counter	1437	20.3	-11	-0.54	-3.05		
17389	Proportional counter	1408	53.2	-13	-0.63	-2.69		
17390	Proportional counter	1.721	0.0235	-100	-4.99	-30.34		
17391	Proportional counter	1363.89	7.4	-15	-0.76	-4.60		
17392	Proportional counter	1228	11.0	-24	-1.19	-7.06		
17393	Proportional counter	1397	16.5	-13	-0.66	-3.84		
17394	Proportional counter	1570	120.0	-2	-0.12	-0.30		
17396	Proportional counter	1181	33.2	-27	-1.33	-6.86		
17397	Proportional counter	1288	58.0	-20	-1.00	-4.10		
17397	Liquid-scintillation counting	1593	23.0	-1	-0.05	-0.29		
17398		1600	155.0	-1	-0.03	-0.06		
17399	Liquid-scintillation counting	1620	160.0	1	0.03	0.06		

JRC-GABZ Sample; Gross bela activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17401	Liquid-scintillation counting	1290	23.0	-20	-0.99	-5.54		
17402	Liquid-scintillation counting	1858	168.0	15	0.77	1.41		
17408	Liquid-scintillation counting	1459.318	74.8	-9	-0.47	-1.64		
17409	Liquid-scintillation counting	1490	125.0	-7	-0.37	-0.88		
17410	Proportional counter	1583	126.0	-2	-0.08	-0.20		
17411		1312	350.0	-19	-0.93	-0.84		
17412	Liquid-scintillation counting	1850	278.5	15	0.75	0.85		
17413	Liquid-scintillation counting	1608	309.5	0	-0.01	-0.01		
17414	Liquid-scintillation counting	1690	290.0	5	0.25	0.27		
17415	Liquid-scintillation counting	1470	98.0	-9	-0.43	-1.26		
17416	Liquid-scintillation counting	1605	298.5	0	-0.02	-0.02		
17429	Liquid-scintillation counting	1493	30.0	-7	-0.36	-1.92		
17430	Liquid-scintillation counting	930	115.0	-42	-2.11	-5.37		
17431	Liquid-scintillation counting	1522	114.0	-5	-0.27	-0.70		
17432	Proportional counter	1600	90.0	-1	-0.03	-0.10		
17434	Liquid-scintillation counting	1560	117.0	-3	-0.16	-0.39		
17435	Liquid-scintillation counting	1466	31.5	-9	-0.45	-2.34		
17436	Liquid-scintillation counting	1.2	0.2000	-100	-5.00	-30.35		
17437	Proportional counter	1181	51.5	-27	-1.33	-5.81		
17439	Liquid-scintillation counting	1700	365.0	6	0.28	0.24		
17440	Liquid-scintillation counting	1522	55.0	-5	-0.27	-1.15		
17441	Proportional counter	1520	10.0	-6	-0.28	-1.67		
17441	Proportional counter	1770	10.0	10	0.50	2.97		
17442	Proportional counter	975	55.0	-39	-1.97	-8.31		
17443	Liquid-scintillation counting	1941	84.0	21	1.03	3.33		
17443	Liquid-scintillation counting	2084	76.0	29	1.47	5.12		

SRC-GABZ sample; Gross beta activity concentration measurements								
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score		
17444	Proportional counter	1360	100.0	-16	-0.78	-2.21		
17444	Liquid-scintillation counting	1650	130.0	2	0.12	0.28		
17445	Liquid-scintillation counting	1670	260.0	4	0.19	0.23		
17448	Proportional counter	1214	91.1	-25	-1.23	-3.76		
17449	Solid state scintillation counter	1246.4	3.5	-23	-1.13	-6.85		
17450	Solid state scintillation counter	1206	19.0	-25	-1.25	-7.18		
17451	Solid state scintillation counter	1243.92	18.3	-23	-1.14	-6.53		
17452	Proportional counter	1270.8	90.7	-21	-1.05	-3.23		
17453	Proportional counter	1207.9	165.3	-25	-1.25	-2.32		
17453	Proportional counter	1282.5	70.5	-20	-1.02	-3.71		
17454	Solid state scintillation counter	1198.9	65.4	-26	-1.28	-4.89		
17455	Solid state scintillation counter	920.2	30.2	-43	-2.14	-11.31		
17456	Solid state scintillation counter	125.8	17.0	-92	-4.61	-26.67		
17456	Solid state scintillation counter	125.8	17.0	-92	-4.61	-26.67		
17456	Solid state scintillation counter	125.8	17.0	-92	-4.61	-26.67		
17457	Solid state scintillation counter	1215.498	71.9	-25	-1.23	-4.42		
17458	Proportional counter	1299.5	8.7	-19	-0.96	-5.78		
17459	Proportional counter	1181.668	49.4	-27	-1.33	-5.91		
17460	solid state scintillation counter, evaporation, sulfatation, ignition	1175.83	25.7	-27	-1.35	-7.37		
17461	Proportional counter	1175.45	61.4	-27	-1.35	-5.36		
17468	in house m, Liquid scintillation counting	1560	120.0	-3	-0.16	-0.38		
17468	ISO11704, UG, Liquid scintillation counting	1563	87.0	-3	-0.15	-0.46		
17468	ISO11704, QS400, Liquid Scintillation counting	1749	141.0	9	0.43	0.92		
17470	Liquid-scintillation counting	1510	139.4	-6	-0.31	-0.67		

Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17488	Proportional counter	1448	130.0	-10	-0.50	-1.15
17490	Proportional counter	1050	53.0	-35	-1.74	-7.47
17490	Proportional counter	1180	56.0	-27	-1.34	-5.58
17491	Proportional counter	1410	290.0	-12	-0.62	-0.68
17492	Coprecipitation plus proportional counter	1220	50.0	-24	-1.21	-5.35
17492	Evaporation plus proportional counter	1520	70.0	-6	-0.28	-1.03
17492	Liquid-scintillation counting	1800	200.0	12	0.59	0.92
17493	Proportional counter	1270.31	158.6	-21	-1.05	-2.03
17494	Proportional counter	1082	81.0	-33	-1.64	-5.45
17495	Proportional counter	7200.1	4402.8	347	17.36	1.27
17496	Proportional counter	1377.1	284.8	-14	-0.72	-0.80
17496	Proportional counter	1390	288.5	-14	-0.68	-0.75
17497	Proportional counter	1065	60.5	-34	-1.69	-6.78
17497	Proportional counter	1079	78.0	-33	-1.65	-5.63
17497	Proportional counter	1121	83.0	-30	-1.52	-4.97
17500	Proportional counter	1409	121.5	-12	-0.62	-1.52
17501	Proportional counter	1450	145.0	-10	-0.50	-1.04
17503	Proportional counter	1589	87.5	-1	-0.07	-0.21
17504	Proportional counter	1337	65.0	-17	-0.85	-3.26
17505	Proportional counter	1110	65.0	-31	-1.55	-5.96
17505	Proportional counter	1150	65.0	-29	-1.43	-5.48
17507	Proportional counter	1360	108.0	-16	-0.78	-2.08
17510	Proportional counter	1338	91.5	-17	-0.84	-2.57
17511	Proportional counter	1263	126.5	-22	-1.08	-2.53
17512	Proportional counter	1367	125.0	-15	-0.75	-1.79

JRC-GADZ sample; gross beta activity concentration measurements							
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score	
17513	preparation of 200ml with H2O2 and HNO3, proportional counter	1339	274.0	-17	-0.84	-0.97	
17513	Proportional counter	1406	188.0	-13	-0.63	-1.04	
17514	Liquid-scintillation counting	1520	65.0	-6	-0.28	-1.07	
17515	Solid state scintillation counter	1120	176.0	-30	-1.52	-2.67	
17515	Proportional counter	1301	214.0	-19	-0.96	-1.40	
17516	PIPS detector	1366	205.0	-15	-0.76	-1.15	
17517	Liquid-scintillation counting	1912.04	44.5	19	0.94	4.36	
17519	Liquid-scintillation counting	1331	133.0	-17	-0.87	-1.95	
17519	Proportional counter	1415	127.0	-12	-0.61	-1.42	
17519	Liquid-scintillation counting	1896	190.0	18	0.89	1.45	
17520	Liquid-scintillation counting	680	40.0	-58	-2.89	-14.01	
17520	Proportional counter	850	150.0	-47	-2.36	-4.78	
17521	Liquid-scintillation counting	1940	150.0	20	1.02	2.07	
17522	Liquid-scintillation counting	1831.3	139.6	14	0.69	1.48	
17523	Proportional counter	960	30.6	-40	-2.02	-10.62	
17524	Proportional counter	1378.42	206.8	-14	-0.72	-1.08	
17525	Solid state scintillation counter	1303.8	73.9	-19	-0.95	-3.37	
17526	Proportional counter	1348	337.0	-16	-0.81	-0.77	
17527	Solid state scintillation counter	4610	1170.0	186	9.32	2.56	
17527	Solid state scintillation counter	5600	1700.0	248	12.39	2.35	
17528	Proportional counter	1070	7.9	-34	-1.68	-10.08	
17529	Liquid-scintillation counting	640	16.0	-60	-3.01	-17.52	
17531		1411	40.0	-12	-0.62	-3.00	
17532	Proportional counter	960	290.0	-40	-2.02	-2.20	
17533	Proportional counter	1280.95	32.9	-20	-1.02	-5.28	

JRC-GAB2 sample; Gross beta activity concentration measurements						
Lab code	Technique	x _i (mBq/L)	u(x _i) (mBq/L)	D _{<i>i</i>,%}	z _i - score	zeta _i - score
17534	Liquid-scintillation counting	1270	270.0	-21	-1.06	-1.24
17535	Proportional counter	1430	43.0	-11	-0.56	-2.64
17536	Liquid-scintillation counting	500	125.0	-69	-3.45	-8.18
17537	Liquid-scintillation counting	1312	150.0	-19	-0.93	-1.87
17537	Liquid-scintillation counting	1352	79.0	-16	-0.80	-2.71
17538	Proportional counter	1246.4	10.6	-23	-1.13	-6.73
17539	Gas proportional counting	1171.75	93.1	-27	-1.36	-4.09
17540	Proportional counter	1147	119.2	-29	-1.44	-3.55
17540	Proportional counter	1259	130.8	-22	-1.09	-2.49
17541	Proportional counter	1070	50.0	-34	-1.68	-7.41
17541	Solid state scintillation counter	1094	152.0	-32	-1.60	-3.21
17542	Proportional counter Ref: K-40	1490	95.0	-7	-0.37	-1.10
17542	Proportional counter Ref: Cs-137	1890	120.0	17	0.87	2.13
17543	Liquid-scintillation counting	1506	108.0	-6	-0.32	-0.86
17544	Proportional counter	2503	98.0	55	2.77	8.02
17545	Proportional counter	1360	40.0	-16	-0.78	-3.77
17546	Proportional counter	1661	44.0	3	0.16	0.74
17568	Proportional counter	1369	47.6	-15	-0.75	-3.38
17569	Proportional counter	1550	120.0	-4	-0.19	-0.46
17570	Proportional counter	1292	105.5	-20	-0.99	-2.69
17571	Proportional counter	1017	68.1	-37	-1.84	-6.87
17572	LB4200 proportional counter V=200ml	1346	41.0	-16	-0.82	-3.94
17572	LB4200 proportional counter V=50ml	2373	212.0	47	2.37	3.49
17808	Proportional counter	1608	46.6	0	-0.01	-0.03
17809	Proportional counter	1369.4	24.6	-15	-0.75	-4.12

Annex 11. Calculation of performance evaluation scores

Percentage difference (D%)

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

$$D_{i,\%} = 100 \times \frac{x_i - x_{\rm PT}}{x_{\rm PT}}$$
 (5)

z-score and ζ (zeta)-score

$$z_i = \frac{x_i - x_{\rm PT}}{\sigma_{\rm PT}} \tag{6}$$

$$zeta_i = \frac{x_i - x_{\rm PT}}{\sqrt{u(x_i)^2 + u(x_{\rm PT})^2}}$$
 (7)

With:

- x_i the measurement result reported by a participant;
- $u(x_i)$ the standard measurement uncertainty reported by a participant;
- $x_{\rm PT}$ the assigned reference value;
- $u(x_{PT})$ the standard measurement uncertainty of the assigned value;
- $\sigma_{
 m PT}$ the standard deviation for proficiency test assessment.

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 38.**). 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

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

$$u_i^2 = u_c^2(A_i) + u_c^2(A_0)$$
(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 38. Interpretation of a PomPlot.



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 $\zeta=\pm 3$ lines are probably inconsistent with the reference value.

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