

Almera Proficiency Test Determination of Naturally Occurring Radionuclides in Phosphogypsum and Water

IAEA-CU-2008-04



IAEA

International Atomic Energy Agency

ALMERA PROFICIENCY TEST
DETERMINATION OF NATURALLY
OCCURRING RADIONUCLIDES IN
PHOSPHOGYPSUM AND WATER

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Naturally Occurring Radionuclides in
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FOREWORD

The Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) network, established by the IAEA in 1995, makes available to Member States a world-wide network of analytical laboratories capable of providing reliable and timely analysis of environmental samples in the event of an accidental or intentional release of radioactivity. The network is a technical collaboration of existing institutions. It provides an operational framework to link expertise and resources, in particular when a boundary-transgressing contamination is expected or when an event is of international significance. The Chemistry Unit of the IAEA Terrestrial Environment Laboratory in Austria is the central coordinator of the ALMERA network's activities.

The IAEA organizes proficiency tests for ALMERA members on a regular basis. These proficiency tests are designed to monitor and demonstrate the analytical performance and technical capabilities of the network members, and to identify gaps and challenging areas where further development is needed.

Continued membership in ALMERA network has benefits in training and educational opportunities, enhanced mutual trust in results and methodology and objective evidence for accreditation purposes.

The performance evaluation results of the proficiency tests performed in the frame of the ALMERA network are not anonymous for those laboratories nominated to participate as ALMERA members.

This report describes the methodology employed and the results obtained in the IAEA-CU-2008-04 proficiency test on determination of naturally occurring radionuclides in phosphogypsum and water.

The IAEA officer responsible for this publication was A. Shakhashiro of the IAEA Environment Laboratories.

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1. INTRODUCTION

Phosphogypsum is generated as a by-product of the phosphoric acid based fertilizer industry. The discharge of phosphogypsum on earth surface deposits is a potential source of enhanced natural radiation and heavy metals, and the resulting environmental impact should be considered carefully to ensure safety and compliance with environmental regulations.

A reliable determination of technologically enhanced naturally occurring radioactive materials in phosphogypsum is necessary to comply with the radiation protection and environmental regulations.

This proficiency test (PT) is one of the series of the ALMERA network proficiency tests organised on a regular basis by the Chemistry Unit of the IAEA Terrestrial Environment Laboratory. These proficiency tests are designed to identify analytical problems, to support Member States laboratories to maintain their preparedness and to provide rapid and reliable analytical results.

In this PT, the test item set consisted of six samples: one phosphogypsum (the IAEA-434 reference material) and five water samples spiked with natural radionuclides. The main task of the participating laboratories was to identify and quantify the activity levels of radionuclides present in these matrices. The tasks of IAEA were to prepare and distribute the samples to the participating laboratories, to collect and interpret analysis results and to compile a comprehensive report.

The certified massic activity values of all radionuclides used in this PT were fulfilling the requirements of metrological traceability to international standards of radioactivity.

In this PT, 306 test items (reference materials) were prepared and distributed to 52 participants from 40 countries in November 2008. The deadline for receiving the results from the participants was set to 15 May 2009. For gross alpha/beta results the deadline was one working day from the date of sample delivery.

The participating laboratories were requested to analyse Ra-226, U-234 and U-238 in water samples 01 and 02, and gross alpha/beta in water samples 03, 04 and 05. In the phosphogypsum sample number 06 the participants were asked to analyse Pb-210, Ra-226, Th-230, U-234 and U-238. Fifty two laboratories registered to take part in the proficiency test. Of these, 49 laboratories reported their results to the IAEA. The analytical results of the participating laboratories were compared with the reference values assigned to the reference materials, and a rating system was applied.

The laboratories which responded to this proficiency test and contributed their efforts to the present work are highly appreciated and acknowledged.

Three National Metrology Institutes (NMI) and six expert laboratories took part in the characterization campaign of the IAEA-434 phosphogypsum reference material; namely: ERISS, Australia (A. Bollhöfer), PTB, Germany (H. Wershofen), HAA, Hungary (S. Tarjan), KINS, Republic of Korea (S. Kim and Y. J. Kim), KRISS, Republic of Korea (S. H. Lee), LNE-LNHB, France (M. Moune), IJS, Slovenia (M. Korun), AECS, Syria (M. S. Al-Masri), and the Agency's Laboratories in Seibersdorf.

The IAEA is appreciative of the contribution of these institutes, which was at no cost to the IAEA.

The summary evaluation of this PT has shown that 67% of the overall reported analytical results fulfilled the evaluation criteria applied in this PT.

2. MATERIALS AND METHODS

2.1. Proficiency test objectives

The measurement of phosphogypsum and water samples containing a mixture of radionuclides with an unknown (to the participants) composition was aiming at (i) assessment of the analytical performance of the participating ALMERA network laboratories in analyzing natural radionuclides, (ii) testing the analytical system response in case of emergency and (iii) encouraging the participating laboratories in finding remedial actions where shortcoming in analytical performance are detected.

2.2. Participants

A total of 49 laboratories reported their results to the IAEA. Figure 1 shows the geographical distribution of the countries of the participants. A full listing of participants is given in Appendix III.

2.3. Preparation of the proficiency test materials

The following proficiency test materials were considered:

- Two water samples (100 mL) spiked with Sr-90 and Th-230;
- One blank water sample for determination of gross alpha/beta;
- Two water samples (500 mL) spiked with Ra-226, U-234 and U-238;
- One phosphogypsum sample (250 g) for determination of Pb-210, Ra-226, Th-230, U-234 and U-238.

Figure 2 and Figure 3 show the PT materials sets.



Figure 2: Combining the PT materials of each set.



Figure 3: A set of the PT materials.

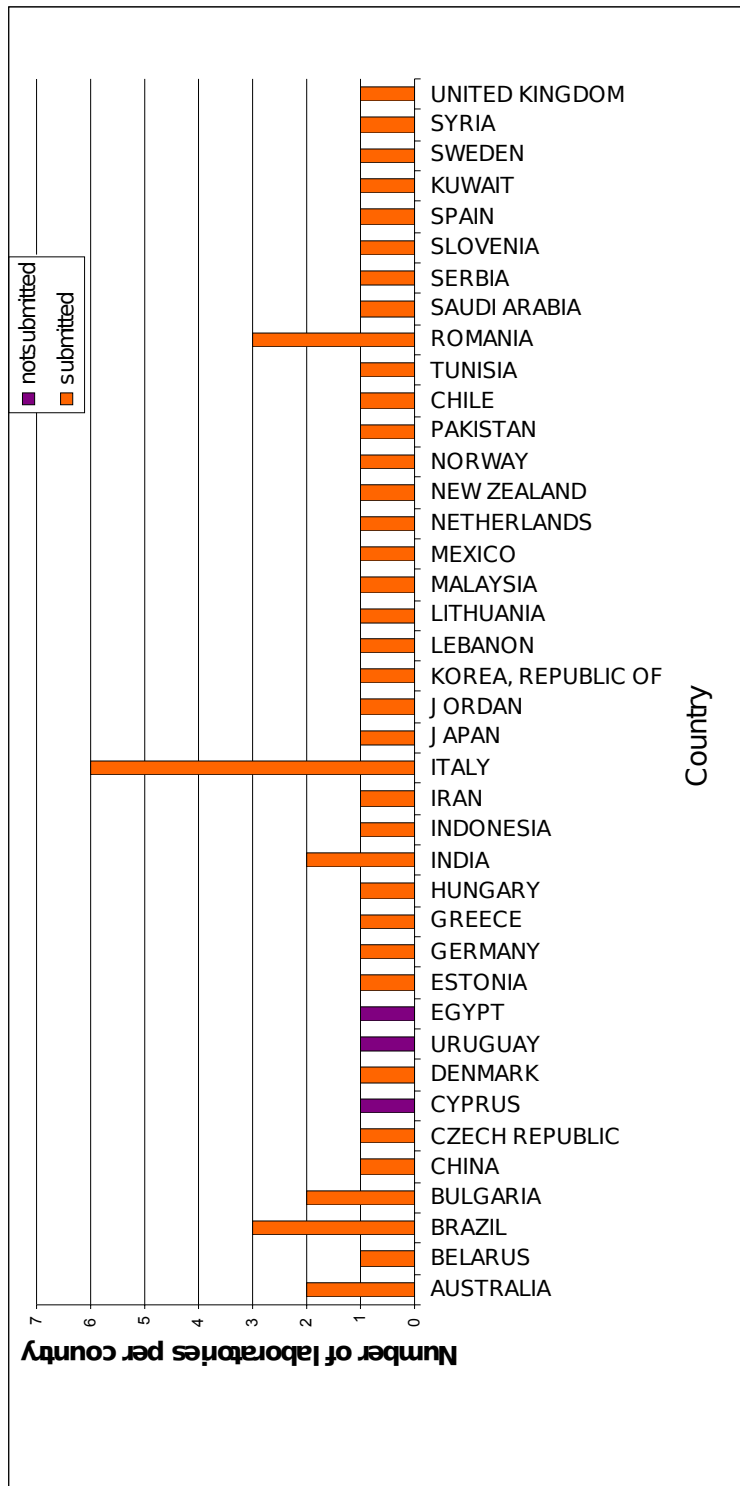


Figure 1: Participation distribution per country.

2.3.1. Preparation of the spiked water samples 01 and 02

Three hundreds kg of tap water outsourced in one batch from Seibersdorf laboratories were used to prepare all spiked water samples. The blank water was acidified and then analysed for the radionuclides of interest. It was found that the activity value of each measurand of interest in this PT was below the detection limit of the analytical method which is far below the spiked activity value in the spiked samples.

The water samples 01 and 02 were gravimetrically prepared in two batches. A portion of 170 kg of the blank water prepared above was spiked with an appropriate amount of a mixture of certified single radionuclide solutions of Ra-226, U-234 and U-238 traceable to the international standard of radioactivity. Table 1 shows the identification of the certified solutions of each radionuclide used in spiking the water samples in this PT.

For homogenising the spiked water a pump with multiple outlets was used to mix the water in a tank of 600 l. The homogenised spiked water was bottled in 500 mL portions in appropriate plastic bottles. The total mass of the bottle with the label was registered for further control.

Table 2 lists the target values and the associated combined uncertainty in the water samples.

TABLE 1: IDENTIFICATION OF THE CERTIFIED SOLUTIONS OF EACH RADIONUCLIDE USED IN SPIKING THE SOIL AND WATER SAMPLES IN THIS PT

Nuclide	Source manufacturer
Ra-226	AMERSHAM - RAY 44
U-234	NIST 4321C and ISOTRAK UEP 10020
U-238	NIST 4321C
Th-230	NIST 4342A
Sr-90	NIST 4234A

2.3.2. Preparation of the spiked water samples 03, 04 and 05

Sample 03 was the blank acidified water which was used for preparation of the spiked water samples.

Water samples 04 and 05 for gross alpha/beta measurements were gravimetrically prepared in two batches. Two portions of 40 kg of the blank water were spiked with an appropriate amount of a mixture of certified single radionuclide solutions of Th-230 and Sr-90 traceable to the international standard of radioactivity.

The homogeneity test results provided experimental evidence that satisfactory levels of homogeneity within and between bottles were attained.

The water samples 03, 04 and 05 were filled in bottles of 100 mL and weighed on an appropriate balance, and the mass was recorded for further control.

TABLE 2. TARGET VALUES AND RESPECTIVE STANDARD COMBINED UNCERTAINTIES OF THE MEASURANDS OF INTEREST IN WATER SAMPLES.

	Radionuclides	Activity Concentration [Bq.kg ⁻¹]	Standard Uncertainty [Bq.kg ⁻¹]	LAP [%]	MAB [%]
Sample 01	U-234	0.56	0.02	15	15
	U-238	0.36	0.01	15	15
	Ra-226	0.69	0.04	20	20
Sample 02	U-234	1.20	0.04	15	15
	U-238	1.25	0.04	15	15
	Ra-226	1.93	0.09	20	20
Sample 03	Gross alpha	<0.2			
	Gross beta	<0.2			
Sample 04	Gross alpha	3.93	0.08	Not applicable	
	Gross beta	15.7	0.3		
Sample 05	Gross alpha	7.68	0.15		
	Gross beta	30.7	0.6		

The reference date is 01 December 2008; the combined standard measurement result uncertainty is expressed at 1σ level.

2.3.3. Verification of the target activity values and homogeneity test

The final target activity value in water samples for each radionuclide was calculated from the certified activity values assigned to each radionuclide, taking into account the successive gravimetric dilution steps, the mass of spiking mixture and the amount of water being spiked as determined from weighing. The combined standard uncertainty includes two major components: uncertainty of the certified solution and weighing uncertainty.

To confirm the assigned target values and to test the homogeneity of the test items two bottles from each water sample were analyzed in ARPA Lombardia, Department of Milano laboratory, Italy. The measurement results obtained by the laboratory were in good agreement with the assigned target values and demonstrated that the homogeneity of the water samples fits the purpose of this PT.

2.3.4. Analytical procedure applied in analysis of water samples

The ARPA Lombardia laboratory reported the following summary of the applied analytical procedure for the determination of gross alpha/beta, Ra-226, U-234 and U-238 in water samples.

2.3.4.1. Analytical procedure for gross alpha/beta measurements

A weighed amount of sample (8 g) was transferred in a scintillation vial. Quicksafe 400 (Zinsser) scintillation cocktail (12 mL) was added and the sample was measured by liquid scintillation counting. The instrument calibration was performed using U-236 and Sr-90/Y-90 sources.

The standard method had to be modified as the sample acidity was out of the acceptable range. New calibrations were performed in similar matrix to take into account the increase of spill-over.

2.3.4.2. Analytical procedure of Ra-226, U-234 and U-238

Uranium isotopes were extracted from a weighed amount of sample (100 g), previously acidified by 14 M nitric acid (5 mL), by a selective scintillation cocktail (double extraction, 10 + 10 mL of cocktail).

The cocktail was then degassed with Argon and measured by liquid scintillation counting.

The scintillation cocktail was homemade and prepared by dissolving bis-ethylhexyl orthophosphoric acid (HDEHP), naphthalene and fluorescent substance (PBBO) in p-xylene.

Calibration was performed using U-236 source.

Radium-226 was measured through Rn-222 and short live daughters at secular equilibrium. A weighed amount of sample (10 g) was transferred in a scintillation vial. Optiscint (Perkin Elmer) scintillation cocktail (10 mL) was added to the sample (this cocktail is not miscible with aqueous samples).

2.3.4.3. Counting method

Samples (20 mL Teflon coated vials) were measured by a Quantulus 1220 LS counter applying alpha-beta discrimination optimized for the specific sample-cocktail mixture. Quench was kept under strict control by SQP(E) measurement.

Radium-226 measurement was performed after the secular equilibrium between Ra-226 and Rn-222 and its short live daughters was attained (1 month). The target values of the water samples with the respective uncertainties are presented in Table 2.

2.3.5. Preparation and characterization of the IAEA-434 phosphogypsum (sample 06)

The IAEA-434 RM [1] was collected from a processing plant located in Gdansk (Poland) in 2003. The matrix composition is: $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (96%), P_2O_5 (1-2%), F Total (1.2%), SiO_2 (1%), Al_2O_3 (0.2%).

The milled material was homogenised in one lot in a clean atmosphere at a temperature of 20 ± 2 °C and a relative humidity of 55%. The used homogeniser was a clone mixer of 1000 litre capacity.

Bottling of IAEA-434 was done under normal laboratory conditions; 1000 bottles was filled in one day taking all precautions to avoid segregation. The bottles were labeled arranged into

plastic boxes and sterilized using gamma ray irradiation with a total dose of 25 kGy using a Co-60 source.

The bottle size was 900 mL with wide secure-sealed cover to preserve the integrity of the reference material in the bottle. The amount of the material in each bottle was 250 g.

2.3.5.1. Homogeneity study of the IAEA-434

10 bottles covering the whole bottling range were randomly selected; three independent sample portions at 12.5 g from each bottle were analyzed using gamma spectrometry for Pb-210, Ra-226, Th-230, U-234 and U-238. The homogeneity of Ra-226 was also tested by analyzing three sample portions of one gram from five bottles using alpha spectrometry technique. The analysis of homogeneity study was performed under repeatability conditions to minimize variations.

The outcome of the homogeneity study demonstrated that the uncertainties due to between and within bottles heterogeneity u_{bb} were generally very small and the material could be considered sufficiently homogeneous for the tested radionuclides at the range of mass used.

The calculated parameters of the homogeneity study are listed in Table 3. It can be noticed that the uncertainty associated with the between-bottles heterogeneity is reasonably small.

TABLE 3: BETWEEN-BOTTLES UNCERTAINTIES ASSOCIATED WITH THE MATERIAL'S HETEROGENEITY OF THE IAEA-434

Nuclide	Standard uncertainty associated with the between-bottles heterogeneity
	[%]
Pb-210	0.76
Ra-226	0.45
Th-230	0.36
U-234	0.41
U-238	0.33

2.3.5.2. *Characterization and assignment of target values of the measurands of the IAEA-434*

The material was initially characterized at the Agency's Laboratories in Seibersdorf during the feasibility study phase. Then the characterization of the material took place within the interlaboratory comparison piloted by the Agency's Laboratories in Seibersdorf in cooperation with the Consultative Committee on Ionization Radiation (CCRI) of the International Bureau des Poids et Mesure (BIPM). The results reported in this comparison were used to derive the property values of the measurands of interest.

The test portion mass for the analysis was proposed to be at least 1 gram for radiochemical analysis and 50 grams for gamma spectroscopy analysis. To assess the digestion difficulty of the phosphogypsum, and to assist users of the IAEA-434 in selecting the dissolution technique, the IAEA laboratories in Seibersdorf performed several dissolution experiments, the most effective one was based on using of HNO₃ and HF acids.

As an example for the measurement of gamma emitting nuclides in the IAEA-434 the gamma-spectrum of phosphogypsum is shown on the Fig 3. The low energy part is shown in Fig 4. The sample was closed in a 250 cm³ metal (radon tight) sample container. The spectrum was collected after 30 days when the Ra-226 Rn-222 equilibrium was reached. A special low background n-type HPGe detector 30% relative efficiency with CARBON-EPOXI window was used for the spectrum collection. The shielding was made of 7 cm low background lead and 3 mm of copper.

The property values of all radionuclides of the IAEA-434 were established on the basis of a robust approach proposed by David L. Duewer [2] and the Mixture Model Median (MM-median) of the analytical results reported by the expert laboratories was calculated. The MM-median is a direct analogue of the median. It is the location which divides the Mixture Model Probability Density Function (MM-PDF) into two sections of equal area. The MM-median is closely related to the median. It is robust to outliers and also accounts for the reported uncertainty of each measurement result.

To estimate the standard uncertainty associated with the property value the MM-median based Standard Deviation S(MM-median) was calculated from the span of the central 50% of the MM-PDF density function [2].

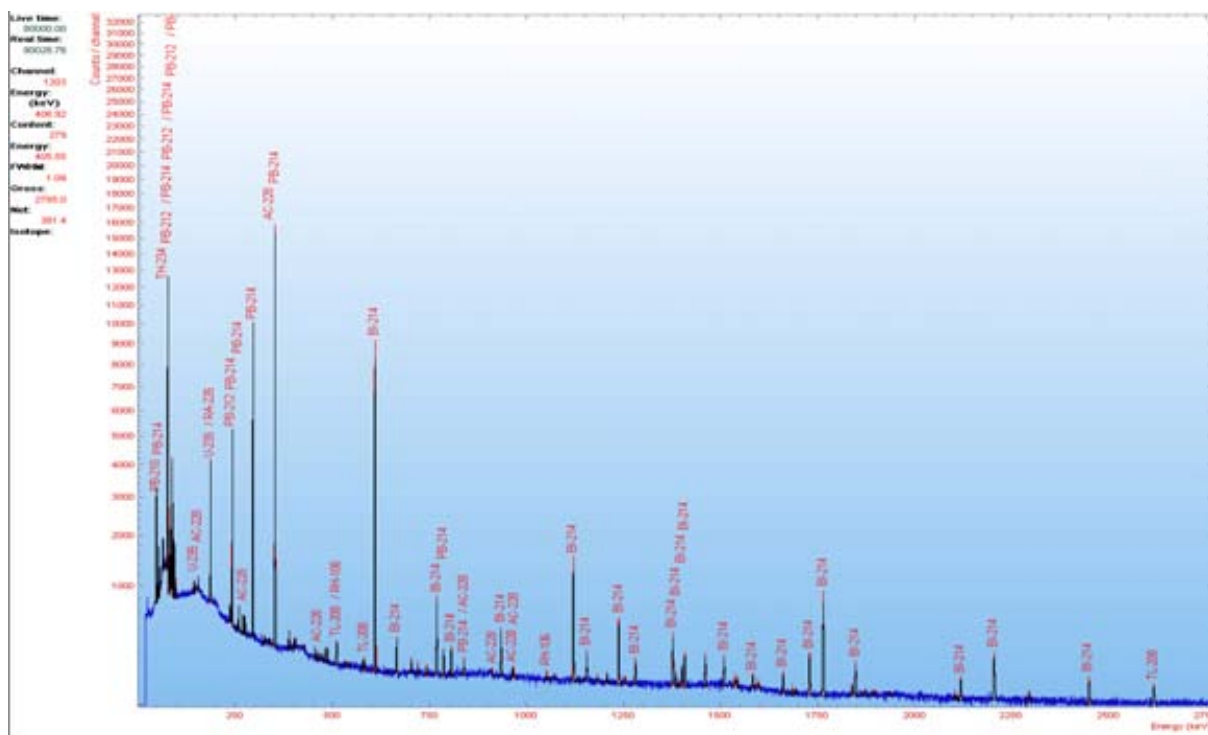


Figure 3. Gamma spectrum of the phosphogypsum.

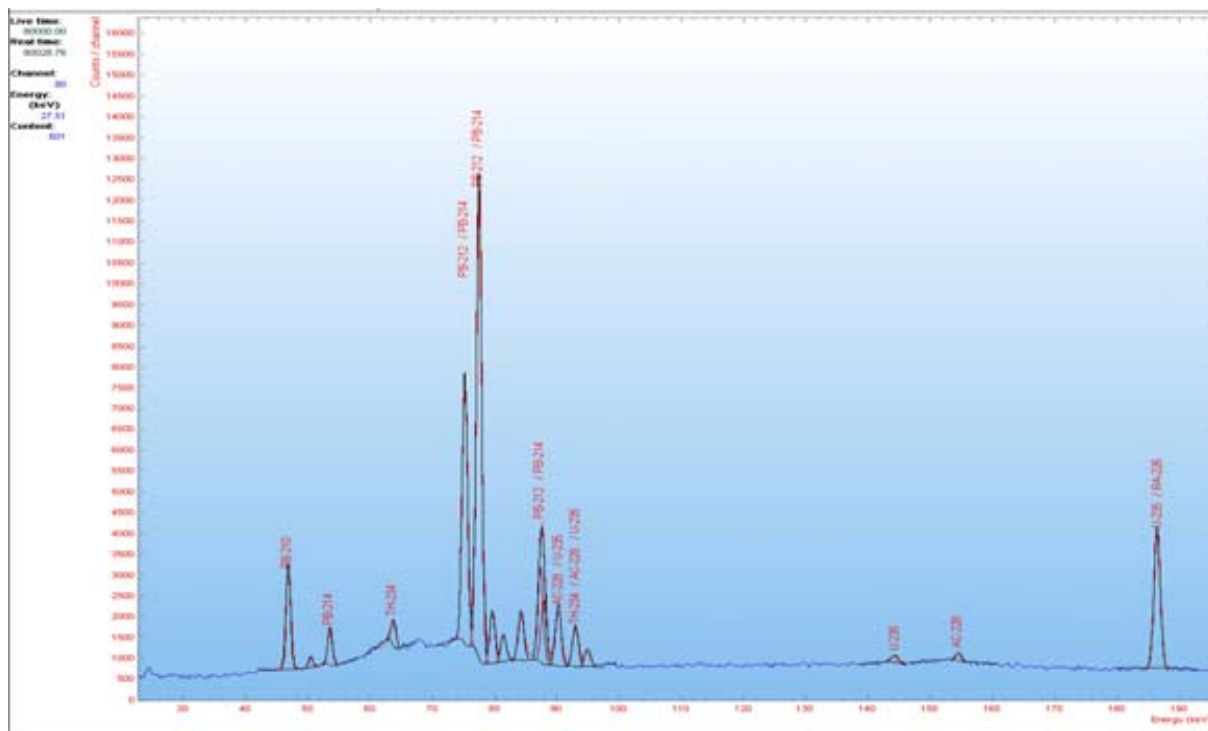


Figure 4. Low energy part of the gamma spectrum.

2.3.5.3. Determination of Pb-210, U and Th isotopes in phosphogypsum

Ten bottles of phosphogypsum were selected, and then 5 sample portions at around 0.5 g were randomly taken from each bottle and weighed into microwave containers. For checking chemical recovery, Pb²⁺ carrier (30 mg), U-232 and Th-229 (its daughter Ra-225) were added. The samples were digested in a microwave oven using the following program: 150°C for 15 min and at 190°C for 25 min. The sample solutions were then transferred to plastic centrifuge tubes and centrifuged for 10 min. The supernatants were transferred to Teflon beakers. The residues were transferred back into the microwave containers with 3 mL HNO₃, and 2 mL of 40% HF were added. The residue was digested in a microwave oven using the same program as mentioned above. The solutions were combined with supernatants and 0.1g H₃BO₃ was added and then evaporated with three portions of 5 mL of 65% HNO₃ to remove HF. The residue was dissolved in 30 mL of 2 M HCl.

2.3.5.4. Sequential separation of Pb-210, U, Th and Ra-226 using Sr resin, TEVA and UTEVA

After sample digestion, the solutions were loaded on Strontium Resin columns, preconditioned in advance with 100 mL 2M HCl. The columns were rinsed with 100 mL of 2 M HCl and 25 mL 6 M HNO₃ to remove the non-retained ions. The effluent and washing solutions were combined into a beaker and used for analysis of uranium and thorium. Lead was eluted with 60 mL 6 M HCl. A Pb-210 source was prepared as lead oxalate and measured by liquid scintillation counting [3].

The effluents and the washing solutions from Strontium Resin columns were combined and then evaporated to dryness. The residue was dissolved in 20 mL 3 M HNO₃ and then loaded on TEVA columns which were preconditioned in advance with 20 mL of 3 M HNO₃. The columns were washed with 10 mL of 3 M HNO₃. The washing solution was combined with the effluent from the TEVA column for analysis of uranium. After additional washing with 20 mL of 3 M HNO₃, 20 mL 8 M HCl was used to elute Th. The effluent and the first washing solution obtained from the TEVA column were directly loaded onto UTEVA column which was preconditioned in advance with 20 mL of 3 M HNO₃. The column was washed with 20 mL of 3 M HNO₃, 5mL 9 M HCl and followed by 15 mL 6 M HCl to remove traces of Po and Th remaining on the column. Finally, the uranium on the UTEVA column was eluted with 6 mL H₂O.

The effluent (30 mL of 3 M HNO₃) and the washing solution (20 mL of 3 M HNO₃) from UTEVA column were combined in a 200 mL volume of beaker. 4 mL of concentrated H₂SO₄ and 20 mL of 20% Na₂SO₄ were added, and then 5 mL of Pb²⁺ carrier (10 mg mL⁻¹) was slowly added while stirring to form Pb(Ra)SO₄ co-precipitation. The precipitation was allowed to settle for 3-4 hours and centrifuged at 3000 rpm for 10 min. The precipitation was dissolved in 10 mL of 0.2 M DTPA (diethylenetriaminepentaacetic acid).

2.3.5.5. Source preparation and measurement

The Pb fraction was evaporated 3 times with 2 mL of HNO₃. The residue was dissolved in 20 mL 1 M HNO₃, 0.400 g oxalic acid was added to warm the solution and adjust the pH to 3-5 with NH_{3(aq)} to precipitate Pb-oxalate. The Pb-oxalate precipitate was filtered through a pre-weighed filter paper (Ø 24 mm). The filter was washed three times with 1 mL water and 2 mL of ethanol, dried in oven at 40-50°C, cooled in a desiccator and weighted to determine the mass of lead-oxalate and the chemical recovery gravimetrically. The lead-oxalate

precipitate was transferred together with the filter into liquid scintillation vial, dissolved in 1 mL 6 M HNO₃ and mixed it with 14 mL 'INSTA-GEL PLUS' liquid scintillation cocktail. Pb-210 was determined by liquid scintillation spectrometry.

U and Th fractions were three times evaporated with few mL of 65% HNO₃, respectively. The residues were dissolved in 10 mL of 10 (NH₄)₂SO₄ plating solution with pH 2 and transferred into electro-deposition cell. U and Th were electrodeposited onto stainless steel discs at 0.97A for 90 min, respectively and then measured by alpha-spectrometry. An example of alpha-spectrum for uranium isotopes is given in Figure 5.

Four mL mixed solution of 5 parts of 20% Na₂SO₄ with 1 part of acetic acid was added to 10 mL of 0.2 M DTPA dissolved Pb(Ra)SO₄ precipitation and 0.4 mL the Ba seeding solution was added, and then allowed to sit for 30min. The colloidal suspension of Ba(Ra)SO₄ was filtered through a pre-wetted polypropylene filter (Pall, 0.1µm pore size, 25mm dia.) and measured by alpha-spectrometry Figure 5.

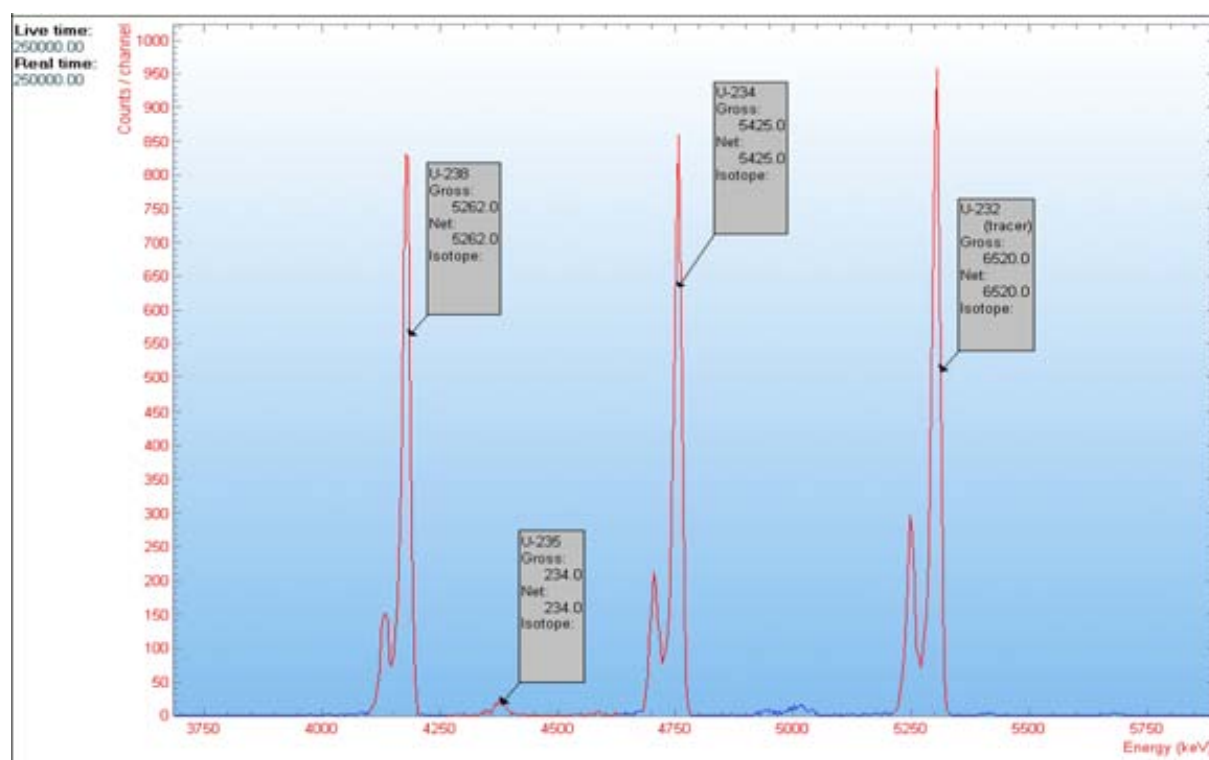


Figure 5: The alpha spectra of uranium isotopes.

Table 4 shows the target values of the massic activities and associated standard combined uncertainties of the measurands of interest.

TABLE 4. THE TARGET VALUES AND THE RESPECTIVE STANDARD COMBINED UNCERTAINTIES OF THE SAMPLE 06 THE IAEA-434 PHOSPHOGYPSUM REFERENCE MATERIAL.

Nuclide	Recommended massic activities [Bq.kg ⁻¹ dry mass]	Standard combined uncertainty* [Bq.kg ⁻¹ dry mass]	LAP (%)	MAB (%)
Pb-210	680	58	15	15
Ra-226	780	62	20	20
Th-230	211	9	15	15
U-234	120	9	15	15
U-238	120	11	15	15

*Uncertainty is expressed as a Mixture model median based standard deviation S(MM-median). Reference date: 01 December 2008.

3. PERFORMANCE CRITERIA

Several rating systems have been developed for determining a laboratory's performance and the meaning of the results of the different scoring systems are not always comparable. Among various statistics, z-scores and u-scores are most often used. The drawback of z-scores is that the uncertainty of the participant's measurement result is not taken into account for the evaluation of performance. In the case of u-scores, the evaluation includes uncertainties of the participant measurements and the uncertainty of the assigned value. Laboratories performing well in classical proficiency testing (z-scores) will not necessarily exhibit the same level of performance when their analytical uncertainties are considered in the evaluation.

The proficiency testing scoring system applied by the Chemistry Unit of the IAEA Terrestrial Environment laboratory takes into consideration the trueness and the precision of the reported data and it includes in the evaluation both the total combined uncertainty associated with the target value of proficiency testing samples and the total uncertainty reported by the participating laboratories. According to the newly adopted approach, the reported results are evaluated against the acceptance criteria for accuracy and precision and assigned the status "acceptable" or "not acceptable" accordingly. A result must pass both criteria to be assigned the final status of "acceptable". The advantage of this approach is that it checks the credibility of uncertainty statement given by the participating laboratories, and results are no longer compared against fixed criteria but participants establish their individual acceptance range on the basis of the uncertainties assigned to the values. Such an approach highlights not only methodological problems affecting the accuracy of the reported data but also identifies shortcomings in uncertainty estimation.

In addition, three other statistical parameters namely: z-score, IAEA/Laboratory result ratio and relative bias are calculated as complementary information for the participating laboratories.

3.1. Relative bias

The first stage in producing a score for a result $Value_{reported}$ (a single measurement of analyte concentration in a test material) is obtaining the estimate of the bias. To evaluate the bias of the reported results, the relative bias between the reported value and the target value is calculated and expressed as a percentage:

$$Bias_{relative} = \frac{Value_{reported} - Value_{target}}{Value_{target}} \times 100\% \quad (1)$$

3.2. Evaluation procedure for samples 01, 02 and 06

The proficiency test results were evaluated against the acceptance criteria for trueness and precision and assigned the status "Acceptable", "Warning" or "Not Acceptable" accordingly [4].

3.2.1. Trueness

The participant result is assigned "Acceptable" status for trueness if:

$$A1 \leq A2$$

where:

$$A1 = |\text{Value}_{\text{target}} - \text{Value}_{\text{reported}}|$$

$$A2 = 2.58 \times \sqrt{u_{\text{target}}^2 + u_{\text{reported}}^2}$$

3.2.2. Assessment of the claimed uncertainty

To evaluate the claimed measurement result uncertainty an estimator P is calculated for each reported uncertainty, according to the following formula:

$$P = \sqrt{\left(\frac{u_{\text{target}}}{\text{Value}_{\text{target}}}\right)^2 + \left(\frac{u_{\text{reported}}}{\text{Value}_{\text{reported}}}\right)^2} \times 100\%$$

P directly depends on the measurement result uncertainty claimed by the participant. The Limit of Acceptable Precision (LAP) for each analyte respectively is defined for the respective proficiency test in advance, including any adjustment due to the concentration or activity level of the analytes concerned and the complexity of the analytical problem. Participants' results are scored as "acceptable" for the claimed uncertainty when $P \leq \text{LAP}$. The LAP values used in the evaluation of all radionuclides are listed in Table 2.

In the final evaluation, both scores for trueness and precision are combined. A result must obtain an "acceptable" score in both criteria to be assigned the final score "acceptable". Obviously, if a score of "not acceptable" was obtained for both trueness and precision, the final score will also be "not acceptable". In cases where either precision or trueness is "not acceptable", a further check is applied. The reported result relative bias (R. Bias) is compared with the maximum acceptable bias (MAB). If $\text{R. Bias} \leq \text{MAB}$, the final score will be "acceptable with warning". "Warning" will reflect mainly two situations. The first situation will be a result with small measurement uncertainty; however its bias is still within MAB. The second situation will appear when result close to the assigned property value is reported, but the associated uncertainty is large. If $\text{R. Bias} > \text{MAB}$, the result will be "not acceptable". The MAB values used in the evaluation of all radionuclides are listed in Table 2.

3.3. Evaluation procedure for gross alpha/beta results in water sample 03

For evaluation of the analytical measurement results of gross alpha/beta in the blank water sample 03 a factor F was calculated as the following:

$$F = \text{Value}_{\text{reported}} - 2 * \text{uncertainty}_{\text{reported}}$$

The participant result was assigned "acceptable" score if:

$$F \leq 0.2$$

The participant result was assigned "warning" score if:

$$0.2 < F \leq 0.3$$

The participant result was assigned "not acceptable" score if:

$$F > 0.3$$

3.4. Evaluation procedure for gross alpha/beta results in water samples 04 and 05

For evaluation of the analytical results of gross alpha/beta in the spiked water samples 04 and 05 the relative bias $Bias_{Relative}$ was calculated according to 3.1 and a factor R was calculated as the following:

$$R = \text{Value}_{reported} / \text{uncertainty}_{reported}$$

The participant result was assigned “acceptable” score if:

$$R > 2 \text{ and } Bias_{Relative} \leq 50\%, \text{ for gross alpha}$$

$$R > 2 \text{ and } Bias_{Relative} \leq 30\%, \text{ for gross beta}$$

The participant result was assigned “acceptable with warning” score if:

$$R > 2 \text{ and } 50\% < Bias_{Relative} \leq 75\%, \text{ for gross alpha}$$

$$R > 2 \text{ and } 50\% < Bias_{Relative} \leq 60\%, \text{ for gross beta}$$

The participant result was assigned “not acceptable” score if:

$$R \leq 2 \text{ or } Bias_{Relative} > 75\%, \text{ for gross alpha}$$

$$R \leq 2 \text{ or } Bias_{Relative} > 60\%, \text{ for gross beta}$$

If the evaluation approach and/or acceptance criteria applied in this PT are not appropriate for the types of analyses and application performed in one of the participating laboratories, it is suggested to apply a self- scoring evaluation system which could fits specific requirements.

4. RESULTS AND DISCUSSION

4.1. General

Six hundreds and twenty five measurement results were reported to the IAEA in this PT from 49 laboratories in 39 Member States.

One month after the deadline for results reporting each participant was able to download an individual evaluation report using a dedicated on-line application to obtain a rapid feed back on the results evaluation.

The participants' data along with the statistical performance evaluation were compiled and presented in two types of tables: tables of summary of the reported technical information in the PT questionnaire regarding the applied analytical technique sorted by laboratory code as shown in Appendix I, and the tables of the performance evaluation results sorted by analyte are reported in Appendix II.

To have an overview of the participants' performance for each specific radionuclide, each radionuclide was studied individually; Table 5 shows the distribution of results scores for each evaluated radionuclide and for each sample.

The number of reported results and obtained evaluation as acceptable/acceptable with warning/not acceptable of each laboratory is presented graphically in Figure 7 and Table 6. This way of results evaluation presentation allows the participating laboratories to compare their results to those reported by other laboratories and to benchmark their performance level with peers. In addition, all participating laboratories could share the technical information provided in the Appendix I to identify the commonly applied analytical procedures.

The overall evaluation showed that 23% of all reported results failed to meet the PT criteria. It was noticed that the percentage of acceptable results for natural radionuclides is slightly lower (around 10%) than the observed level for the artificial ones. Figure 6 shows the result of the summary evaluation of the analytical performance of the participating laboratories.

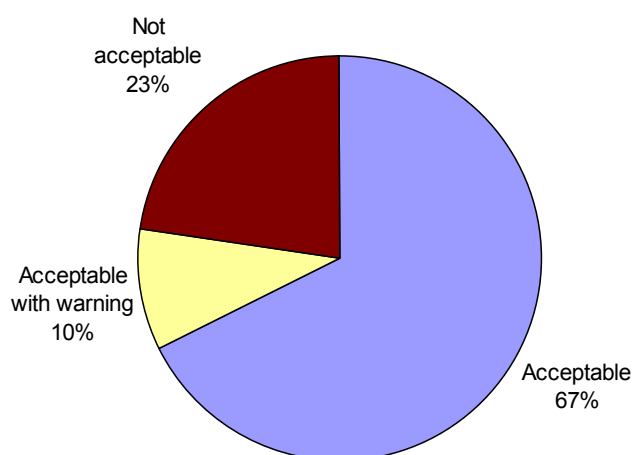


Figure 6. Summary evaluation of the analytical performance of the participating laboratories.

To compare the performance level of each laboratory a normalized mean performance score and the percentage of “Not Acceptable” scores were calculated as the following:

$$\text{Normalized mean performance score (\%)} = ((A+W)/ \text{NRR}) * 100$$

$$\text{Percentage of “Not Acceptable scores” (\%)} = (N/\text{NRR}) * 100$$

Where: A: number of obtained “Acceptable” scores,

W: number of obtained “Acceptable with warning” scores.

N: number of obtained “Not Acceptable” scores.

NRR: Total number of reported results.

The results of the calculation are shown in Table 7.

TABLE 5. SUMMARY EVALUATION OF ALL NUCLIDES SORTED BY SAMPLE.

	Radionuclide	No. of reported results	Percentage of “Acceptable” results (%)	Percentage of “Acceptable with warning” results (%)	Percentage of “Not Acceptable” results (%)
Water Sample 01	Ra-226	35	51	6	43
	U-234	33	82	3	15
	U-238	37	65	11	24
Water Sample 02	Ra-226	35	60	9	31
	U-234	32	75	6	19
	U-238	35	71	6	23
Water Sample 03	Gross alpha	32	81	0	19
	Gross beta	38	71	5	24
Water Sample 04	Gross alpha	38	47	18	34
	Gross beta	41	66	20	15
Water Sample 05	Gross alpha	38	47	21	32
	Gross beta	41	76	9	15
Phosphogypsum Sample 06	Pb-210	48	71	10	19
	Ra-226	37	73	8	19
	Th-230	28	54	14	32
	U-234	34	79	6	15
	U-238	43	77	7	16

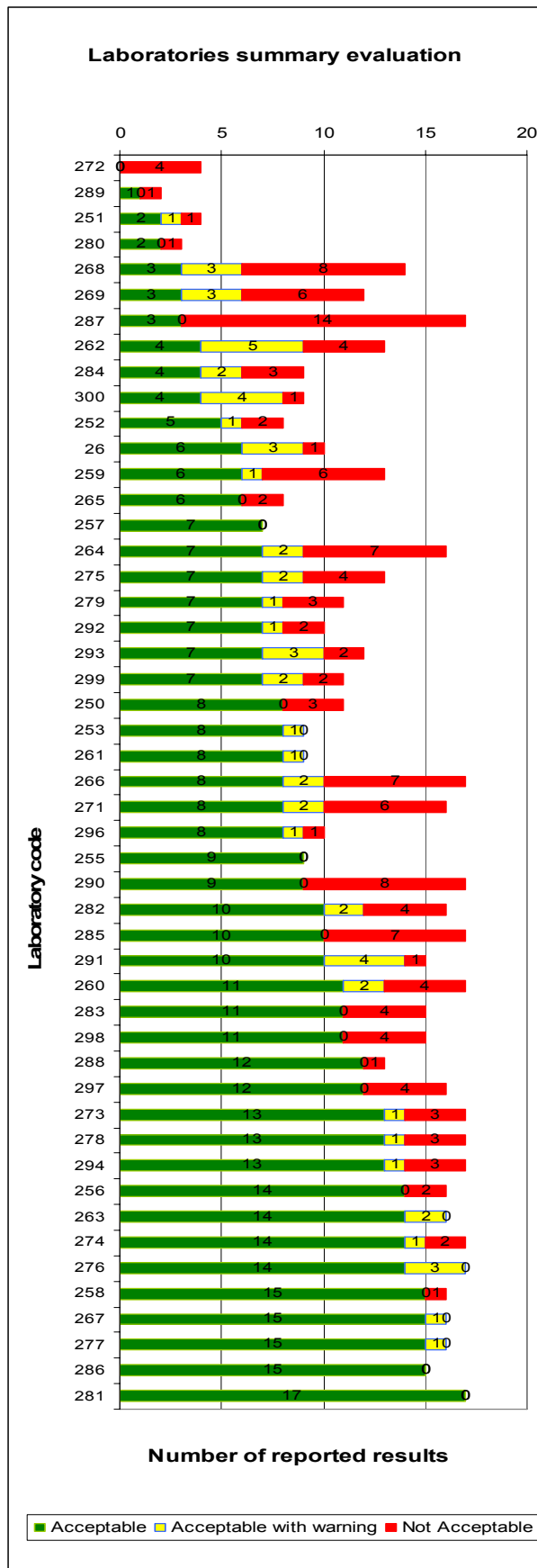


Figure 7: Performance evaluation results sorted by the number of acceptable results obtained.

TABLE 6. SUMMARY EVALUATION OF THE REPORTED RESULTS OF THE IAEA-CU-2008-04 ALMERA PROFICIENCY TEST

Laboratory code	Sample 01		Sample 02		Sample 03		Sample 04		Sample 05		Phosphogypsum sample						Number of not reported results	Acceptable	Acceptable with warning	Not Acceptable	Number of reported results
	Ra-226	U-234	U-238	Ra-226	U-234	U-238	Gross Alpha	Gross Beta	Gross Alpha	Gross Beta	Ra-226	Pb-210	Th-130	U-234	U-238						
26	NR	NR	NR	NR	NR	NR	A	A	W	N	W	W	A	A	NR	A	A	3	6	7	10
250	N	A	A	N	A	A	NR	NR	NR	NR	NR	NR	A	A	N	A	A	0	8	6	11
251	N	NR	NR	A	NR	NR	NR	NR	NR	NR	NR	NR	W	A	NR	NR	NR	1	2	13	4
252	NR	NR	NR	NR	NR	NR	A	A	W	A	N	A	A	A	NR	NR	NR	1	5	9	8
253	A	NR	NR	A	NR	NR	A	W	A	A	A	A	A	A	NR	NR	NR	1	8	8	9
255	NR	A	A	NR	A	NR	NR	NR	NR	NR	NR	NR	A	A	NR	NR	NR	0	9	8	9
256	A	A	A	A	A	N	N	A	A	A	A	A	A	A	NR	A	A	0	14	1	16
257	NR	A	A	NR	A	NR	NR	NR	NR	NR	NR	NR	A	A	NR	NR	A	0	7	10	7
258	A	A	A	A	A	A	A	A	A	A	A	A	A	A	NR	A	A	0	15	1	16
259	N	A	A	N	A	NR	NR	N	A	N	A	W	N	NR	N	NR	NR	1	6	4	13
260	A	A	A	A	N	N	W	A	A	A	A	A	N	A	A	W	A	2	11	0	17
261	NR	NR	NR	NR	NR	NR	NR	A	A	A	A	A	A	A	NR	NR	A	1	8	8	9
262	N	NR	N	W	NR	N	A	W	W	W	A	N	W	NR	NR	A	A	5	4	4	13
263	A	W	W	A	A	NR	A	A	A	A	A	A	A	A	NR	A	A	2	14	1	16
264	N	N	N	N	N	N	A	W	A	W	A	NR	N	A	NR	N	A	2	7	1	16
265	NR	NR	NR	NR	NR	NR	A	N	A	N	A	A	A	A	NR	NR	NR	0	6	9	8
266	A	A	N	N	N	N	A	W	A	W	A	A	A	A	N	N	N	2	8	0	17
267	A	A	A	A	A	A	A	A	A	A	A	A	A	A	NR	W	A	1	15	1	16
268	N	NR	N	W	NR	NR	A	N	W	N	A	A	W	N	N	N	N	3	3	3	14

Laboratory code	Sample 01		Sample 02		Sample 03		Sample 04		Sample 05		Phosphogypsum sample						Number of not reported results	Acceptable	Acceptable with warning	Not Acceptable	Number of reported results			
	Ra-226	U-234	U-238	Ra-226	U-234	U-238	Gross Alpha	Gross Beta	Gross Alpha	Gross Beta	Ra-226	Pb-210	Th-130	U-234	U-238									
269	NR	A	W	N	NR	NR	NR	A	N	A	N	W	N	NR	N	NR	N	NR	W	5	3	3	6	12
271	N	N	N	N	A	W	A	A	W	N	A	N	A	NR	A	A	A	A	A	1	8	2	6	16
272	N	NR	N	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	13	0	0	4	4
273	A	A	W	A	N	A	N	N	A	A	A	A	A	A	A	A	A	A	A	0	13	1	3	17
274	A	A	A	A	A	A	A	N	W	N	A	A	A	A	A	A	A	A	A	0	14	1	2	17
275	A	A	A	A	NR	NR	N	NR	NR	N	NR	W	NR	NR	A	NR	A	A	A	4	7	2	4	13
276	A	A	A	A	A	A	A	A	A	A	A	W	W	W	A	W	A	A	A	0	14	3	0	17
277	A	A	A	A	NR	A	A	W	A	A	A	A	A	A	A	A	A	A	A	1	15	1	0	16
278	N	A	A	N	A	A	A	A	A	W	N	NR	NR	A	A	N	A	A	A	0	13	1	3	17
279	A	A	A	NR	NR	A	NR	NR	NR	N	NR	N	NR	NR	NR	NR	W	A	A	6	7	1	3	11
280	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	A	A	NR	NR	14	2	0	1	3
281	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	0	17	0	0	17
282	A	A	A	A	A	N	N	W	W	A	A	N	NR	NR	A	N	NR	A	A	1	10	2	4	16
283	A	N	N	NR	NR	A	A	A	A	A	A	A	A	A	A	A	A	NR	A	2	11	0	4	15
284	NR	NR	NR	NR	NR	A	A	W	N	W	N	W	N	A	A	N	NR	NR	A	8	4	2	3	9
285	N	A	A	A	A	A	A	N	N	N	N	N	N	N	N	N	N	N	N	0	10	0	7	17
286	A	A	A	A	A	A	A	A	A	A	A	A	A	NR	NR	A	A	A	A	2	15	0	0	15
287	N	N	N	N	N	N	N	N	N	N	N	N	N	N	N	N	N	N	N	0	3	0	14	17
288	A	NR	NR	A	NR	N	A	A	A	A	A	A	A	A	A	A	A	NR	A	4	12	0	1	13
289	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	A	NR	NR	NR	15	1	0	1	2

Laboratory code	Sample 01		Sample 02		Sample 03		Sample 04		Sample 05		Phosphogypsum sample						Number of not reported results	Acceptable	Acceptable with warning	Not Acceptable	Number of reported results
	Ra-226	U-234	U-238	Ra-226	U-234	U-238	Gross Alpha	Gross Beta	Gross Alpha	Gross Beta	Ra-226	Pb-210	Th-130	U-234	U-238						
290	N	A	A	A	A	N	N	N	N	N	A	N	A	A	A	A	0	9	0	8	17
291	N	A	W	NR	W	A	W	A	A	A	A	A	NR	A	A	A	2	10	4	1	15
292	W	A	A	A	A	NR	NR	NR	NR	NR	NR	N	N	A	A	A	7	7	1	2	10
293	N	NR	NR	N	NR	A	W	W	A	A	A	A	NR	A	A	A	5	7	3	2	12
294	A	A	A	N	A	A	A	A	A	A	A	A	W	N	N	N	0	13	1	3	17
296	W	A	A	N	A	NR	NR	NR	NR	NR	A	A	NR	A	A	A	7	8	1	1	10
297	NR	N	A	A	A	A	N	A	A	A	N	A	A	A	A	A	1	12	0	4	16
298	NR	A	A	A	A	N	N	N	N	N	A	A	A	NR	A	A	2	11	0	4	15
299	NR	NR	NR	NR	NR	A	A	A	A	A	A	N	N	W	W	W	6	7	2	2	11
300	NR	NR	N	W	NR	NR	NR	W	A	W	A	W	NR	NR	NR	NR	8	4	4	1	9
Total															208	422	60	143	625		

TABLE 7. SUMMARY INDIVIDUAL EVALUATION SORTED BY THE NORMALIZED MEAN OF PERFORMANCE SCORE.

Laboratory code	Number of not reported results	Number. of “Acceptable” results	Number of “Warning” results	Number of “Not Acceptable” results	Normalized Mean of performance (%)	Percentage of “Not Acceptable” (%)
26	7	6	3	1	90	10
250	6	8	0	3	73	27
251	13	2	1	1	75	25
252	9	5	1	2	75	25
253	8	8	1	0	100	0
255	8	9	0	0	100	0
256	1	14	0	2	88	13
257	10	7	0	0	100	0
258	1	15	0	1	94	6
259	4	6	1	6	54	46
260	0	11	2	4	76	24
261	8	8	1	0	100	0
262	4	4	5	4	69	31
263	1	14	2	0	100	0
264	1	7	2	7	56	44
265	9	6	0	2	75	25
266	0	8	2	7	59	41
267	1	15	1	0	100	0
268	3	3	3	8	43	57
269	5	3	3	6	50	50
271	1	8	2	6	63	38
272	13	0	0	4	0	100
273	0	13	1	3	82	18
274	0	14	1	2	88	12
275	4	7	2	4	69	31
276	0	14	3	0	100	0
277	1	15	1	0	100	0
278	0	13	1	3	82	18
279	6	7	1	3	73	27
280	14	2	0	1	67	33
281	0	17	0	0	100	0
282	1	10	2	4	75	25
283	2	11	0	4	73	27
284	8	4	2	3	67	33
285	0	10	0	7	59	41
286	2	15	0	0	100	0
287	0	3	0	14	18	82
288	4	12	0	1	92	8
289	15	1	0	1	50	50
290	0	9	0	8	53	47
291	2	10	4	1	93	7
292	7	7	1	2	80	20
293	5	7	3	2	83	17

Laboratory code	Number of not reported results	Number. of “Acceptable” results	Number of “Warning” results	Number of “Not Acceptable” results	Normalized Mean of performance (%)	Percentage of “Not Acceptable” (%)
294	0	13	1	3	82	18
296	7	8	1	1	90	10
297	1	12	0	4	75	25
298	2	11	0	4	73	27
299	6	7	2	2	82	18
300	8	4	4	1	89	11

4.2. Recommendations and lessons learned

Based on the performance evaluation results certain recommendations to enhance the analytical performance of the participating laboratories could be suggested. It was not possible to find a clear correlation between the years of experience and the level of performance. There were many cases where the laboratory declared an insufficient experience but its reported results were acceptable. It is evident that the skills and experience of the analyst performing the analysis at the time of the PT sample analysis is the crucial factor in obtaining acceptable results. Proper equipment, human and material resources are of course important factors in obtaining reliable and good quality results.

Method validation and robustness studies of the analytical procedure could assist the analyst to know more in depth its strong and weak points, where more attention should be given and what are the critical points in the procedure. When a laboratory reports 5-10 unacceptable analytical results it means that it has not the appropriate tools to verify the quality of the outcome of the analytical system, there should be a dedicated mechanism and tools which help the analyst to assess the quality of the results produced. If the analyst does not have the control on the analytical system, erroneous results could be reported without being detected.

It is quite normal to make mistakes in an analytical laboratory, the analytical system is a very complex one, it even interacts extensively with human factors and even with the specific conditions of the laboratory. However, the main objective of each laboratory should focus, after detection of the shortcoming, on finding out the root cause of the analytical problem and to implement the necessary actions to remediate the problem and obtain satisfactory results.

The Agency’s Laboratories in Seibersdorf would appreciate to get a feedback from the participants who were able to benefit from this PT in finding out the root cause of the analytical problem and were able to improve their performance in analysing natural radionuclides.

4.2.1. Recommendations regarding Ra-226 determination

48 laboratories reported their results for Ra-226 in phosphogypsum only 19% of which did not fulfil the PT performance criteria. This is considered a good level of performance taking into consideration the difficulty of the matrix and non-availability of appropriate reference material.

Most of the laboratories used gamma spectrometry with HPGe detector; different efficiency calibration procedures were applied. Analysts were innovative in adopting the appropriate method for calibration of gamma spectrometer and in performing the needed corrections.

The PT participants reported in the questionnaire a wide range of analytical methods taken from the open literature such as ASTM E 181-98(2003), CIEMAT PR-X2-04, DOE RA-06-RC, IAEA-TRS-295, IAEA-TECDOC-1401, ISO 10703:2007, NEN5623 (Dutch), NF M 60-803, NF M 60-805-5 and DOE EML HASL-300. Many laboratories used computational calibration such as Monte Carlo for efficiency calibration. Few laboratories used spiked milk powder for calibration; others used some matrix reference materials such as IAEA-Soil-6 and IAEA-RGU ore. One laboratory reporting the used of well detector where a sample portion of only 2 grams was used in the analysis of the phosphogypsum and reported acceptable result which indicates the suitability of the material for measurement using small sample portion in well detectors.

Many laboratories applied radiochemical separation and alpha spectrometry measurement. Technical details of successful application of digestion procedure, radiochemical separation and auto-plating including were reported by the participating laboratories and summarized in Appendix I.

The shortcomings shown in the performance level of the participating laboratories in gamma measurements of Ra-226 could be attributed to several factors. These factors are related to the gamma spectrometry procedure utilised by each laboratory; some of these factors are the use of inappropriate calibration standards or procedure, sample geometry, interference of gamma lines and their densities. Furthermore, the radionuclides of interest in this specific sample are interrelated. Therefore, the decay and ingrowth of daughter radionuclides and their secular equilibrium with their parents should be reached.

Many laboratories reported the use of plastic container for as measurement geometry. The analysts in this case assumed the tightness of such type of containers. However, in practice most of plastic containers are not Rn-222 tight and they allow the emanation of the noble gas even through the walls of the container. Special containers should be used and validated for Ra-222 tightness and not only air tightness.

Certain laboratories reported not acceptable results for Ra-226 due to negative bias. The main reason could be due to the fact that most of the laboratories have used different gamma lines instead of the main gamma energy line 186 keV for Ra-226. In this case one issue should be considered which is the time required to establish secular equilibrium between Ra-226 and its daughters and to avoid the escape of Rn-222 from the container. This is always conducted by storing the sample in Ra-222-tight containers for at least 3 weeks. Air tight container does not mean that the same container is Rn-222 tight; this should be verified and demonstrated. The negative bias could be related to the equilibrium situation and could be caused either by using a sample container which is not Rn-222-tight, or due to a wrong calibration standard or calibration procedure.

A smaller group of laboratories reported not acceptable results for Ra-226 due to a positive bias which could come from inappropriate correction factor for gamma attenuation in the sample or due to the use of inappropriate gamma energy line which suffers from spectral interference. This could be overcome by using a calibration standard with a density similar to the test sample applying a correct calibration procedure.

In water samples the performance level was lower than in phosphogypsum due to the low activity level and more complex analytical procedure needed comparing to phosphogypsum.

A more detailed examination and study of the reported technical information related to the applied analytical procedures will take place in a forthcoming IAEA publication to give more in-depth information and conclusions about the effect of different procedures on the level of analytical performance based on the results of this PT.

4.2.2. Recommendations regarding Pb-210 determination

37 laboratories reported their results for Pb-210 in sample 06 phosphogypsum, 19% of them where not acceptable. This level of performance is comparable to the level of performance in the 2007 PT for the determination of Pb-210 in soil samples [5]. In the contrary, the results evaluation of 2005 PTs resulted in 59% of unacceptable reported results for the same analyte in soil [6]. This might suggests a general improvement in the analytical performance of the population of laboratories interested in Pb-210 determination in solid matrix.

In this PT the majority of laboratories measured the Pb-210 at 46.52 keV using n type wide energy range HPGe detector and applying a calibration procedure and corrections.

Equally successful determinations were reported by several laboratories using radiochemical procedures such as the separation of Pb-210 after the addition of a stable carrier according to the method of chromate precipitation followed by lead chloride precipitation. Then mixing the source with scintillation cocktail and perform measurement using Liquid Scintillation Spectrometer.

Many laboratories that used radiochemical procedure failed in proper implementation of the procedure which resulted in unacceptable results, the exact technical reason of the shortcoming should be investigated. Little information was reported in the questionnaire on the chemical recovery correction. The main reason for unacceptable results could be attributed to a non validated radiochemical procedure or to incomplete digestion procedure.

Some laboratories stated that they used gamma detectors with energy range from 200 keV which might be not suitable for Pb-210 measurement; also some laboratories extrapolated the efficiency calibration to cover the Pb-210 energy which resulted in unacceptable results. The most frequent reasons for failure in Pb-210 are discussed in more details in [7].

4.2.3. Recommendations regarding U-234, U-238 determination

34 and 43 laboratories reported their results for U -234 and U-238 respectively. Different methods were used successfully in this PT. Alpha spectrometry with various extraction and purification procedures was used successfully; more details on applied procedures could be found in Appendix I.

The uranium content of the water samples was a few Bq/kg only. In this case the low activity level was the main cause of analytical difficulty during the analysis.

To obtain a good quality of gamma spectrum it would have required a special low background detector and a well controlled quality of the laboratory air to avoid the effect of Rn-222 on the analysis, "Rn-free" laboratory air would be the best option. The enrichment of the radon progeny in the detector shielding could lead to an uncontrolled (random) positive bias which is comparable to the activity level in the sample.

Using a chemical separation and alpha spectrometry the radiochemical purity of the applied reagent and tools may be the crucial and critical point for both of uranium and radium determinations. The analysis and statistical monitoring of the “blank” sample could help to recognize false positive result or positive bias. In particular, this factor is important due to the similarity of the chemical behaviour of Ba compounds which contains a traces amount of Ra.

4.2.4. Recommendations regarding gross alph/beta determination

The gross alpha/beta measurements are considered to be primarily screening determinations, therefore, this PT was aimed at testing the ability of a laboratory to determine if significant radioactivity level is present or not. Also the PT aimed at testing if a laboratory could by an erroneous analytical result trigger a false alarm which might cause wrong decisions and waste of resources. For this reason the acceptance criteria were fairly wide for alpha/beta measurements.

The participants were not informed in advance about the nuclides used in spiking the water samples. This was in the design of the PT to check the effect of calibration with different nuclides on the final results. Also to simulate the real situation in a laboratory where in a real sample the radionuclides present will not be known, and hence a calibrated instrument used successfully in this PT cannot be assumed to give similarly accurate and precise measurement results on real and routine gross alpha/beta determination.

Different radionuclides were successfully used in calibration such as: K-40, Sr-90/Y-90, Cs-137, U-236, U-238, Am-241 and Pu-242.

4.2.5. Recommendations regarding Ra-226, U-234 and U-238 determination

Gamma spectrometry is not the best suited method for the measurement of Ra-226 and Uranium isotopes in water as the expected concentration is usually low (less than 1 Bq/kg in drinking waters) and the background contribution quite high when compared to the expected values. Furthermore, U-238 lacks direct gamma emissions and can be measured only assuming radioactive equilibrium with daughter nuclide Th-234 (useful gamma emissions at 63.3 keV and 92.4-92.8 keV); U-234 lacks gamma emissions and no reliable assumptions can usually be made on its equilibrium with parent and daughter nuclides (it is well known that U-234 is not even in secular equilibrium with U-238, due to different chemical-physical behaviour of the two Uranium isotopes in aquifers [8] [9] [10]); Ra-226 has a direct gamma emission at 186.2 keV strongly interfering with both U-235 gamma emission and background contribution and, as outlined in par. 4.3.1, its measurements by Rn-222 and daughter nuclides needs specific care in order to avoid radon losses and assure radioactive equilibrium.

However, gamma spectrometry is often used as it is a well established and widely diffused technique (furthermore, high efficiency detectors with low background are more and more often available); in such a case, special care has to be taken to assess background contribution e.g. by repeated background measurements and a thorough statistical analysis of results (in low level measurements the variability of repeated measurements can be higher than the counting - Poisson - uncertainty of single measurement, and the background contribution and uncertainty can be best assessed as average and standard deviation of repeated measurements). Sample containers must be filled to the top as any air headspace can cause uneven radon dispersion (and consequently affect the actual geometrical efficiency). The use of updated and internationally agreed Nuclear Data Tables is strongly supported (e.g. [11] [12]) in order to avoid mistakes due to the use of out-of-date gamma ray intensities.

Measurements by liquid scintillation counting, both in homogeneous and in non homogeneous phase, rely on daughter nuclides at equilibrium and must be performed using radon tight containers (e.g. polytetrafluoroethylene (PTFE), polyethylene-PTFE coated or high density polyethylene vials); furthermore, radon extraction yield cannot usually be directly measured and must be assessed on the basis of the results of method validation, that should include a thorough evaluation of both repeatability and robustness. Counting can be performed considering alpha or alpha + beta window: in the last case, blank contribution has to be carefully checked to account for possible reagent contamination (e.g. K-40 is often present at detectable levels in commercial liquid scintillation cocktails) and for fluctuations well beyond the variability expected on the basis of counting (Poisson) statistic.

Measurements based on emanometry are widely used; in this case a critical point is the evaluation of Rn-222 extraction yield from the radium solution since internal yield tracers are not available and the recovery strongly depends on degassing procedure (gas flow, bubble size, etc.); a particular effort must be put in method validation, with special regard to repeatability assessment.

Ra-226 can be measured by direct alpha counting of Ba(Ra)SO₄, precipitated and deposited on filter; in this case Rn-222 and short live daughters contribution to alpha counting is a critical point as radon can be trapped in sulphate crystals in variable amounts thus affecting actual counting rate. Again, method validation and repeatability assessment are key points.

The radium co-precipitation with barium sulphate is often used as a preliminary step for concentration and purification, and can be applied in all the above reported methods. In such cases, the assessment of chemical yield is necessary and it is usually performed gravimetrically by weighing the recovered Ba(Ra)SO₄; yield overestimates can occur when similar elements (e.g. calcium) are co-precipitated. The use of radioactive tracer (e.g. Ba-133 that can be measured by gamma spectrometry) overcomes this problem.

The determination of chemical yield is as well the limiting factor for the determination of radium isotopes by alpha spectrometry, as the use of the most commonly employed yield tracer Ra-225 relies on multiple assumptions on its equilibrium both with parent (Th-229) and daughter (Ac-225) nuclides. Furthermore, the whole procedure is cumbersome and requires operator high skill.

Uranium measurement by alpha spectrometry is a well established procedure but not the easiest one. Special care should be taken in order to avoid cross contamination that can occur when the same laboratory materials and deposition device (beakers, deposition cells, etc.) are used for the analysis of waters, soils etc. Use of disposable materials is strongly recommended as far as possible.

Uranium can be measured by liquid scintillation counting following extraction by homemade scintillation cocktail containing specific complexing agents (e.g. HDEHP) or by specifically designed commercial cocktails (e.g. URAEX® by Ordela, Inc.). As internal yield tracers can hardly be used due to poor spectral resolution, the evaluation of overall efficiency and repeatability are key points of method validation.

For all the above reported methods, traceability of radioactive sources used for calibration or as internal yield tracers must be assured at any time. Liquid solutions of tracer nuclides are often provided without any indication about expiry date, which (if present) actually refers to the sealed, untouched source. Once opened (and diluted), internal procedures should be

applied for their management (dilution included) possibly including periodical check of actual concentration

4.3. Technical information provided by the participants

Appendix I contains the summary of the reported technical information. For each laboratory, the years of claimed experience in analysis and summary of analytical procedure and counting method is summarized.

5. CONCLUSIONS

The IAEA-CU-2008-04 PT was successfully completed with high level of reporting-back the analytical results, whereas 94% of the registered laboratories reported their results to the IAEA. Most of the participants were able to quantify certain number of radionuclides of interest in phosphogypsum and water. 23% of all reported results did not pass the proficiency test acceptance criteria.

This PT provides the possibility to improve the ALMERA laboratories comparability and reliability of the determination of natural radionuclides in environmental matrices.

This PT shows the need for further improvement of the analytical performance for determination of natural radionuclides in aqueous samples at low level of radioactivity. The performance level of such determinations was the lowest in this proficiency test.

A second PT on the same radionuclides is recommended to assess any improvement in the analytical performance based on the lessons learned in this PT.

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APPENDIX I
SUMMARY OF THE REPORTED TECHNICAL INFORMATION
SORTED BY LABORATORY CODE

The summary technical information on the analytical procedures applied in the laboratories of the PT participants is presented as reported by the participants without editing.

Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the IAEA or any of its staff members thereof.

In these tables, laboratory information is presented in ascending order of the laboratory code. The blank cells indicate that no technical information was provided. The listed years of experience is as reported by the participants according to their judgment.

The abbreviation “NR” in the Table of Appendix I denotes “Not reported”.

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
26	> 3 years	NR	NR
250	1 - 3 years	Reference materials Verified methods	HPGe gamma spectrometry Self-absorption corrections
251		NR	NR
252	> 3 years	The efficiency calibration was performed using a cocktail provided by the Metrology Laboratory consisting of several isotopes to span the energy range of interest. The system was calibrated for the same geometry of the sample. The efficiency calibration was performed using a cocktail provided by the Metrology Laboratory consisting of several isotopes to span the energy range of interest. The system was calibrated for the same geometry of the sample. The counting system consist of a 25% GX Canberra HPGe. The Pb-210 was determined in the low energy range and corrected for the sample self-absorption.	The sample (175 g) was weighed in a 7 cm plastic vial routinely used at our laboratory as gamma counting geometry. The plastic vial was wrapped with Aluminium foil to wait for Radon ingrowth. We waited more than 30 days before gamma counting. The plastic vial containing the sample was placed directly on the 25% efficiency GX Canberra HPGe detector and counted for 60000 seconds.
253	> 3 years	Gamma-spectrometry, HPGe detector. Software: Gamma Trac. Efficiency calibration: multinuclide standard.	Sample preparation: 0,5 l Marinelli container.
255	> 3 years	Measurements were performed with 40% HPGe gamma spectrometer "Canberra". Spectra were evaluated with Canberra Genie 2000 v3.1. Gamma spectrometer was calibrated with cocktail mixture (241Am, 109Cd, 139Ce, 57Co, 60Co, 137Cs, 113Sn, 85Sr, 88Y, 203Hg) with 0.46 g/cm ³ density.	25 g of dried sample were transferred to 50 mL plastic box. The cover of the box was tied to the box with silicone glue. After 28 days activity was measured.
256	> 3 years	Kim et al, App. Radiation. & Isotope. (2001) 275-2	Radium in water sample was co-precipitated with Barium sulphate, which was purified with EDTA solution. Water sample was dried in stainless dish under IR lamp. PG added with Ba carrier was decomposed by HF and Nitric acid, and radium was coprecipitated with Barium sulphate ppt., which was purified with EDTA solution. 100 mL, 200 mL, 300 mL, and 500 mL of ground water spiked with standard Sr-90 and Am-241, and then dried into planchette, which used for counting efficiencies of gross alpha and gross beta. Purified Barium(Ra) sulfate was suspended in Instagel XF and UltimaGold AB, and then measured using LSC after 21 days. Radium in standard Ra solution was coprecipitated

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
			with Barium sulphate, and then measured with the same cocktail composition for calibration.
257	> 3 years	<p>In-house method.</p> <p>Efficiency calibration is performed using a certified multi-radionuclide solution in 4M HCl(QCY.48), with energies ranging from 59.54 keV to 1836.06 keV. Energy calibration is performed with several point and volume sources in the same energy range. During gamma spectroscopic analysis the sample were put on top of the detector used. For the sample analysis 4 detectors were used, namely: a GeLi (Model 7229), a HPGe (Coaxial), a LEGe (Model GL2020-7500) and an XtRa (Model GX10021). An in-house experimental-numerical method is applied to correct for differences in self-absorption between the calibration source and sample matrix for energies < 200 keV.</p>	<p>The samples were enclosed in plastic cylindrical containers of 1mm thickness (volume 285mL, height 69mm), sealed with an epoxy resin against the escape of Radon. The sample completely filled the container. The net mass of the sample was determined using a balance (accuracy 0.1g). Dry mass was determined using a separate portion of the sample, after drying overnight.</p>
258	> 3 years	<p>Direct measure of the sample with germanium detector. Calibration of proportional gas counter with Sr-90 and U-236 certified standard solution</p>	<p>ISO 9696 - ISO 9697. The laboratory for the determination of Ra226, and U-238 in water did not use gamma spectrometry, but liquid scintillation and alpha spectrometry respectively.</p>
259	NR	<p>HPGe: With liquid solutions containing multiple radionuclides. PIPS: Energy calibration with four different radionuclides. A Petri dish was filled with the sample and set aside for 3-4 weeks to allow re equilibrium between Ra-226 and the short-lived Rn-222 daughters. Ra-226 and Pb-210 were determined with HPGe. Ra-226 via Bi-214 and Pb-214. Self-absorption in the sample was corrected for using a point source</p>	<p>U and Th: 1.024 gram sample was dissolved in 1.5 litre of slightly acidic water and Th-229 and U-232 added. The organics in the sample were destroyed with H2O2 and MnO4- and U and Th co-precipitated with MnO2. MnO2 was collected by decantation and centrifugation, and dissolved with HCl and H2O2. The silicates in the sample were dissolved with HNO3 + HF. U and Th were then separated using UTEVA and their activities determined with alpha spectrometry.</p>
260	> 3 years	<p>Counting on High Purity Germanium, N-type (40%). Counting time 60.000 seconds. Analysis with Gamma Vision. Calibration is performed with a nuclide mixture (QCY-48 from PTB (Germany)in the same geometry. Afterwards correction for density and composition difference was done.</p>	<p>A 250 mL counting box was filled with 135.68 grams of Phosphogypsum. Dry weight was 93.7 %.</p>

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
261	> 3 years	The sample was analyzed by gamma spectrometry HPGE; the calibration of the gamma spectrometry detector was carried out with a multinuclide standard source.	The moisture content was calculated by determination of loss of weight, at 85 degree Celsius in oven, overnight; then a quantity of sample was sifted; then a net quantity of 30.46 grams of sample was poured in a little cylindrical container (with the same geometry of the calibration standard source) and analyzed by gamma spectrometry HPGE, twice, with a counting time of 120000 seconds and 200000 seconds.
262		For gamma spectrometric analysis, water samples are acidified to pH 2 and preconcentrated by evaporation under an infrared light to 900 mL. After preconcentration, all water samples were sealed into Marinelli beakers and left for 28 days to reach the radioactive equilibrium between 226Ra and daughters (214Bi and 214Pb). Gamma spectrometric measurement were performed using a HPGe detector (AMETEK) with relative efficiency of 25% and 40% and energy resolution of 1,85 keV (1332.5 keV 60Co). The analysis of each measured γ -ray spectrum has been carried out by a software program GAMMA VISION-32. The radium isotope 226Ra was determined from its daughters 214Pb (295.1 keV and 351.9 keV) and 214Bi (609.3 keV, 1120.3 keV and 1764.5 keV). The uranium isotope 238U was determined from its daughters 234Th (63.29 keV). We use the international standard ISO 10703. Sample from proficiency test with similar density and known activities were used for calibration. Gamma spectrometric measurement were performed using a HP Ge detector (AMETEK) with relative efficiency of 25% and energy resolution of 1,85 keV (1332.5 keV 60Co). The analysis of each measured γ -ray spectrum has been carried out by a software program GAMMA VISION-32.	From open literature, ISO, National standard, ISO 9696 and ISO 9697, ASTM International, Designation: E 181-98. The sample was acidified to stabilize it, evaporated almost to dryness converted to the sulphate form and then ignited at 3500C. A portion of the residue is transferred to a planchette and the alpha-beta activity measured. The measurements of gross alpha and beta activity are carried out by α - β -proportional gas counter PIC-WPC-9550, Protean Instrument Corporation. We are used the international standard ISO 9696 and ISO 9697.
263	1 - 3 years	Gamma-spectrometry HPGe, peaked background correction, decay correction, multiplet deconvolution; calibration with multiplex source. Alpha spectrometry with PIPs detectors and inductively coupled plasma-mass spectrometry (ICP-MS).	Rapid measurement with no treatments; only to determinate Ra-226, Pb-210 activities. The other nuclides were determined by alpha spectrometry and ICP-MS systems.

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
264	> 3 years	-Energy calibration with eu-152 standard source; efficiency calibration with mix qy44-amersham in Marinelli 0.5l; -counting method: high-resolution gamma spectrometer with hpge detector with be window, counting time=24h; background subtract; bottle 0.5l geometry.	direct measurement in Marinelli 0.5l geometry dry-to-wet ratio determination for an aliquot of 5 g at 85 degree Celsius overnight
265	> 3 years	NR	NR
266	> 3 years	System calibrated with certified mixed standard radionuclides calibration checked using IAEA 326 and Soil-6	Sample prepared in a 200mL Marinelli Beaker Sealed and wait >1 month for secular equilibrium Sample counted using gamma spectrometry Spectra peak area marked, subtracted background, calculated manually
267	1 - 3 years	The same for Gamma emitting RN in water plus material self attenuation correction.	Geometrical configuration used: cylinder (diam.65 mm)100 mL.
268	> 3 years	calibration was made with an extended gamma source containing a mixture of radionuclides, traceable to the national standard of activity. Counting between 20000 and 50000 s. Background measured for 200000s was subtracted. ISO 10703:2007 Water quality.	APHA-AWWA-WEF - Standard Methods. ISO 18589-3:2007 Measurement of radioactivity. Sample was evaporated to near dryness, redissolved and poured into a standard counting container. 10 mL of sample was evaporated onto a 7 sq. cm planchette. Phosphogypsum was poured into a 100 cc standard counting container and pressed to obtain apparent density of 1g/cc. Alpha calibration with Am-241 beta calibration with Sr/Y-90 Sources traceable to national standard Counting time 30 min Background counted 30 min.
269		From open literature, ISO, National standard. Gamma emitting RN in Water geometry of Marinelli beaker using HpGe 40% eff. calibrated With standard reference material with the same geometry. Geometry of 65.0gm pettery dish using HpGe 40% eff calibrated With standard reference material with the same geometry.	using liquid scintilator counter TRI-CARP_3170 Cocktail Instagel plus solution Calibration Source Sr-90,Am-241(standard)
271	> 3 years	efficiency calibration(Mixed source) chemical preparation Alpha PIPS detector chemical yield	Ra-226: 500 mL in MB, HPGe detector, counting time 80000 s Activity Ra-226 calculated from Pb-214 and Bi-214. U-238,U-234 Alpha spectrometry, electrodeposited source using U-232 tracer
272	> 3 years	We use a Ortec 25% GeHp detector, Multiuclide certified solution Eu152, Cs137, Co60 geometry plastic flask 250 mL For counting 6 measurements were made each one of 60000 seconds.	NR

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
273	> 3 years	Gamma Spectrometer, Canberra Model, HPGe Detector, 45% Efficiency.	Mixed Gamma as calibration source
274		U alpha spectrometry Ra alpha spectrometry calibration with internal tracer Th-alpha spectrometry calibration with internal tracer Pb-gross beta calibration with reference source	U – Fe coprecipitation, TOPO-column separation, electrodeposition Ra- BaSO4 coprecipitation, TOPO and cation column separation, electrodeposition Th- Fe coprecipitation, TOPO-column separation, electrodeposition Pb Leaching, PbS precipitation, anion column separation, PbSO4 deposition
275	> 3 years	Gamma spectrometry (Ra-226) Alpha spectrometry (Th-230) ICP-MS (U-isotopes)	Dissolution in weak HNO3 + HF on residue. Th-230: Ion-exchange and electrodeposition. Th-229 spike. Ra-226: MnO2+PbSO4 ppt + LSC U-isotopes: ICP-MS directly on dissolved sample.
276	> 3 years	Spectrometers are calibrated using a calibration standard produced in the same way that the measured sample from a liquid QCY48 certified solution. Calibration spectra is collected until the number of counts in each peak is higher than 10000. Spectra are analyzed by Gamma Vision software.	The method used is the direct introduction of phosphogypsum (or sediments, soil, ash...)in a plastic container which is sealed and stored 25 days to reach radioactive equilibrium, if natural radionuclides are searched. And then, the container is directly measured on the top of a Ge(Hp)spectrometer from Canberra with EG&G Ortec electronics. Measuring time depends on the activity contents; in routine measurements 24 hours. The same time is chosen for background measurement done with blank samples.
277	> 3 years	original method beta: Determination of Gross beta, Radioactivity Measurement Series No.1,Ministry of Education, Culture, Sports, Science and Technology of Japan,Tokyo,Japan.(1976)	For alpha and beta: Sample solution was heated and evaporated to dryness. calibration source: evaporated Al(NO3)3 solution added Uranium.(alpha),U3O8(beta) counting system: ZnS scintillation counter(alpha),Low-background counter(beta).
278	> 3 years	The measurement technique is based on the use of germanium detectors coupled to a computerized analytical system. The detectors are calibrated for efficiency using a mixed radionuclide standard that covers an energy range of approximately 120-2000 keV. Efficiencies at lower energies are determined on an individual basis. Stored spectra are analysed using the software FITZPEAKS for photopeak identification and subsequent quantification.	An acidified water sample was concentrated by evaporation and sulphuric acid (specific gravity 1.84) added, the solution was then evaporated to dryness. The resulting solid material was ashed in a muffle furnace. An aliquot of the ground residue was used to prepare a uniform thickness source which was counted on a Berthold LB770 low-level proportional counter for 1000 minutes.
279	< 1 year	The PSA is adjusted by Am-241 reference standard and Sr-90 reference standard.	Acidification, concentration by evaporation , 3 mL sample + 17 mL cocktail (Ultima Gold LLT),Counting by LSC Tricarb 3180.

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
281	> 3 years	<p>from open literature, ISO, National standard, Lowson.R., Short.S. (1986), Australian Std method AS3550.5(1990) .</p> <p>Final dried residue masses were recorded and the planchettes counted on a Protean Gas Proportional Counter (WPC 9550) for 4 cycles of 100 minutes per cycle. (400 minutes counting time per sample in total) Matched standards of similar mass were used to ascertain alpha and beta counting efficiencies. The planchette standards consisted of traceable americium-241 for alpha efficiency and dried analytical grade potassium sulphate (Assay 99.99%) for the beta efficiency via potassium-40. Two background planchettes were counted with each cycle to provide instrument background performance. Errors are calculated as counting errors only.</p>	<p>Ra-226 in water - add Ba-133 tracer, manganese dioxide coprecipitation, lead sulphate coprecipitation, Ba & Ra precipitate collected on a filter paper. U-234, U-238 in water - add U-236 tracer, ion exchange resin to isolate uranium, electrodeposit uranium.</p> <p>5mL sample aliquots were weighed onto stainless steel planchettes and evaporated via gentle hotplate heat. Ten aliquots in total were evaporated for each spiked sample, resulting in residual masses of ~ 50 milligrams per sample.</p> <p>Ra-226, U-234, U-238 sources were counted by alpha spectrometry. Ra-226 recovery was calculated by counting Ba-133 activity by gamma spectrometry.</p>
282	> 3 years	Alpha spectrometry calibration by Pu-239 standard and Th recovery was assumed.	<p>Ra-226: Gamma counting in 35% P-type HPGe detector in pre-calibrated 130 g cylindrical geometry plastic bottle. Th & U isotopes: 20-25 g sample added U-232 tracer. Acid leaching. Hydroxide precipitation. Anion exchange chromatography with 8N HNO3. Elute with 8 N HCL for Th. Washing of 8N HNO3 for U and again anion ex. separation for U with 8 N HCL. elute with 0.1 N HCL. Electroplating of U and Th separately and counted in PIPS alpha spectrometer.</p>
283	> 3 years	gamma spectrometer with mix standard sources alpha spectrometry using U-232 tracer and standard calibration of 4 radionuclides.	for Ra-226 and Pb-226 by gamma spectrometer for U-234 and 238 by radiochemical analysis
285	1 - 3 years	<p>Measurement Technique and Methods: In this work an ultra low level liquid scintillation counting system has been used for determine gross alpha and beta activity. This technique involves mixing the water sample with a proper detection cocktail to be counted in a liquid scintillator. Under these conditions, problems relating to sample self-absorption and attenuation of particles by detector windows are completely avoided. Furthermore, alpha and beta events can be simultaneously recorded in separated counting channels by pulse shape analysis (PSA), which is based on the difference between the delayed components of their fluorescent decay . Instrumental setup: An ultra-low level Perkin Elmer Quantulus 1220</p>	<p>Ra-226 Determination in samples 01 and 02: 100g of each sample was added specified amount of Ba-133 tracer solution. After drying the sample, the residue was dissolved in HCl solution and by addition of Pb carrier and H2SO4, Pb(Ra,Ba)SO4 was precipitated. The precipitate was dissolved in EDTA and transferred to a bubbler. Ra-226 was measured by emanation method of Radon gas. Uranium-234,238 Determination in samples 01and 02: Water samples were added U-232 tracer solution and they were dried and changed to HCl form. Uranium separation was performed by an anionic Bio-Rad (1X8) (100-200mesh) resin. In order to purify the uranium fraction (0.1 M HCl), the eluate was extracted by Diiso-butyl ketene.</p>

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
		<p>liquid scintillation counter has been used for all gross alpha and beta and ²²⁶Ra activity measurements. This instrument is specifically designed for the determination of very low level activities using both an anticoincidence active and a passive shield and low background Teflon-copper vials. It also includes a pulse shape analyzer which separates pulses produced by alpha and beta radiations into different spectra, MCA1; SP11(Beta type events) and SP12(Alpha type events). An automatic sample changer allows to measure up to 60 samples. A computer is used to operate the system and to store and manipulate the resulting spectra. For each selected procedure, LS counter PSA parameter, background; efficiency and minimum detectable activity (MDA) were measured as follows: ?PSA parameter: an alpha (²⁴¹Am) and a beta emitter (⁹⁰Sr/⁹⁰Y) are separately measured and the interference between both spectra was determined for different PSA values; the best PSA parameter is determined by minimizing the sum of alpha and beta interference. Because quenching can significantly alter the pulse shape and therefore affect the pulse discrimination effectiveness .the quench was monitored by the SQP (E) instrument parameter which corresponds to pH of the sample and presence of dissolved oxygen must be controlled; ?Background: background count rate determined by measuring dead water samples and scintillation cocktail in the same proportions and conditions as real samples. Background measurements should be repeated in order to take account of its statistical and seasonal fluctuations; mean count rate and standard deviation are then used in calculations; ?Calibration: to maintain the lower possible detection limits, analytical grade reagent and high-purity deionized water were used throughout this work. Ideally, reference radioisotopes used for calculation of system efficiency should be similar to those expected to measure. In the case of natural radionuclides this is difficult to achieve due to the complex nature of the numerous decay chain and source equilibrium system. An alternative is to chose artificial sources with a similar emission energies range. In this work, calibration surrogates for alpha and beta radionuclides of ²⁴¹Am (α; energy ~ 5.64 MeV) and ⁹⁰Sr (β; mean energy ~ 0.2 MeV) + ⁹⁰Y progeny (β; mean energy ~ 0.93 MeV),</p>	<p>The uranium source was prepared as a micro-precipitate according to the HASL procedure. The sources were measured 20000s.</p> <p>250 mg of the sample was added U-232, Th-229 and Po-208 tracer solutions and was leached by HF and HNO₃ in T<85°C. Po, U and Th were precipitated with Fe(OH)₃. Po-210 alpha spectrometry source was prepared by spontaneously plating the polonium isotopes from 0.1M HCl solution onto a copper disk. The source was measured 10000s. We assumed that Po-210 and Pb-210 are in radioactivity equilibrium in phosphogypsum, and then we used the activity concentration of Po-210 for pb-210. The remaining solution from Polonium plating was dried and dissolved in HCl 9M. U and Th separation were performed by a Bio Rad (1X8) (100-200mesh). Di iso-butyl kenene was used for uranium purification. Thorium purification was performed by the second column in HNO₃ form. U and Th alpha spectrometry source were prepared as micro precipitates according to HASL procedure. The sources were measured 20000s.</p> <p>Analytical Procedure: Samples 03, 04 and 05 were concentrated 10 times. After cooling the samples in room temperature, 5 ml of the concentrated water samples were transferred in 20 mL Polyethylene vial and 15 mL of optiphase Hisafe3 (Perkin Elmer) cocktail were added and samples were vigorously shaken. Sample were measured after a 3 hours rest in dark to allow a decrease of photo and chemi-luminescence and decay of short lived radon progeny with water samples, background and standard samples (²⁴¹Am, ⁹⁰Sr/⁹⁰Y) were counted for 500 minutes in the same procedure.</p>

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
		<p>where respectively, where chosen. The radionuclides were prepared from standard solutions traceable to the US National Institute of science and technology (NIST). A spiking standard counting 6.42 Bq/sample for ²⁴¹Am and 5.095 Bq/sample for ⁹⁰Sr/⁹⁰Y was prepared in the same proportions and condition as real sample.</p>	
286	> 3 years	<p>High resolution gamma spectrometer was used for the determination of Ra-226. Alpha spectrometry was used for the determination of uranium.</p>	<p>Samples are mixed with a epoxy resin and set in a calibrated geometry. The samples are allowed to sit for 30 days to allow for ingrowth of radon decay products (for Ra-226). Samples for uranium determined by digesting the sample (microwave digestion/hotplate), separation using UTEVA resin.</p>
287		<p>In-house method: PO-LRA-0907 IPEN-CNEN/SP, Sao Paulo, 2008 and Nuc Instr Met Phys Res A278 (1989) 541-549.</p> <p>The gross alpha and beta determination in water samples was performed by using a low-background gas-flow proportional counter from EG&G Berthold, model LB 770. The low-background gas-flow proportional counter efficiency calibration for gross-alpha measurement was determined using an Am-241 reference source; for gross beta measurement using a Sr-90 reference source.</p> <p>For Ra-226 determination in water samples a low-background gas-flow proportional counter from EG&G Berthold, model LB 770 was used. The low-background gas-flow proportional counter efficiency calibration for gross-alpha measurement of a Ba(Ra)SO₄ is determined using a Am-241 reference source, 2 inches diameter nickel disk, were the source was electro-plated. These sources especially prepared have approximately 500 pCi (18.5 Bq) of Am-241 isotope (half-life=432.6 y). The self-absorption factor was determined by measurement of precipitates prepared with the addition of Ra-226 standard solution, with activity concentrations ranging between 8.7 and 20.3 Bq. The value of the self-absorption factor obtained was 0.3001+/-0.0065. Chemical yield was calculated gravimetrically considering the initial addition of a 20 mg/sample Ba²⁺ carrier solution. The gravimetric recovery of the precipitates obtained for these tested water samples was (95+/-3)%. The LLD for this method was 2.2 mBq/kg for Ra-226 for a 200</p>	<p>An aliquot of 100 g of water sample was taken for this essay. Sample was analysed in triplicate. Each aliquot was diluted to 1,000 g with Mill-Q purified water. The Ra was co-precipitated as Ba,Pb(Ra)SO₄ by adding 50 mL of 3M H₂SO₄. The precipitate was dissolved with alkaline EDTA. When pH was adjusted to 4.5 with glacial acetic acid, Ba(Ra)SO₄ was re-precipitated, while interfering elements remained in the solution. The precipitate was filtered and the chemical yield determined gravimetrically. The measurement of Ra-226 activity concentration was performed 21 days after the precipitation.</p> <p>100mL-capacity polyethylene flasks were loaded with an amount of each sample as received. Sample was analysed in duplicate. The flasks - cylindrical with 4cm height - were weighted using an analytical balance, before and after filling. After counting, the dry-to-wet mass ratios were determined by drying to constant mass.</p>

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
		minutes counting time at the 95% confidence level. Detector-Type and Model: HPGe XTRa Canberra GX2518(25% relative efficiency). Method of efficiency calibration: multinuclide standards using reference materials, prepared in house in the same geometry of the samples. Self-attenuation correction was applied for the measurement of Pb-210 (gamma energy 46keV).	
288	> 3 years	Canberra gamma-spectrometry system, HPGe detector, the system is calibrated with multinuclide standard in standard Marinelli 0.45 L, 1.0 g/cm ³ density.	Measuring time: 259200 seconds.
289	> 3 years	From open literature, ISO, National standard, IAEA Technical Report 295.	Sample was shifted to a Marinelli beaker having capacity 200 gm and a reference material IAEA soil 327 was also counted in the same geometry on a HPGe detector.
290	NR	NR	NR
291	NR	NR	NR
292	NR	NR	NR
293	> 3 years	In-house method. The calibration was made with a multinuclide standard solution of Isotope Products Laboratories it was spilt in Marinelli Beaker of 0.5 L, and taken to volume of with solution carrier of the stable isotopes of the different RN in the Metrology Laboratory of our Institute. The calibration was made with Po-210 and Sr-90 in solution and evaporated like the samples. 250 grams of the sample were spilt in Marinelli Beaker of 0.5 L, it was sealed and rested for 30 days. Later they were counted during 60 000 seconds three times, the Pb-210, Ra-226 and U-238 were evaluated in the photopeak of 46.5, 609 and 63.3 keV respectively.	450 mL of the samples were spilt in Marinelli Beaker of 0.5 L, these were sealed and rested for 30 days. Later they were counted during 100 000 seconds three times, the Ra-226 was evaluated in the photopeak of 609 keV. 100 grams of samples were evaporated, the evaporation residues were spilt directly in planchette and dried to constant weight and counted in a alpha and beta counting system
294		Calibration source is prepared with spiking of artificial soil (mostly SiO ₂) as method is usually used for sediments or soils.	embedding of sample in epoxy resin in standard size container and waiting for ingrowth radon daughters.
296	1 - 3 years	The spectrometers are calibrated with traceable radionuclide activity standards supplied by PTB. Direct gamma measurements on two different spectrometers from January to April 2009, but needed to be interrupted for routine measurements several times. Finally, sum spectra from each	Ca. 55 g of the sample was gravimetrically transferred into a polypropylene container.

Lab code	Years of experience	Counting method	Summary of the applied analytical procedure
		spectrometer were formed and evaluated for the final results. Self-attenuation correction was applied by use of the GESPECOR code.	
297	> 3 years	Gamma-spectrometer DSA-1000 "Canberra", detector GX5019 with Be window (0,5mm thick. Calibration on CRM CBSS_2 (Cert.No:9031-OL-06/08)	Th-230 and U-234 on ICP-MS 1. mineralization 2. Selective allocation on anion-exchange columns 3. Measuring on ICP-MS Ra-226, Pb-210 and U-238 on gamma-spectrometry 1. 3 aliquots the dried up sample is transferred to hermetic closed measuring vessel. 2. Vessels are stored within 14 days for achievement of balance Ra-226 with daughter radionuclides. After that measurements on gamma-spectrometer are carried out.
298	> 3 years	The sample was measured in eight germanium detectors; the reported result is obtained as a non weighted average over the results individual results. The detectors are calibrated biannually with calibrated solution.	The sample material was pressed in a plastic canister with the diameter of 90 mm to the height of 35 mm. The sample was sealed to minimize the exhalation of radon. The activity of U-238, Th-230 and Pb-210 were obtained from the count rate in the 63 keV, 68 keV and 46 keV peaks respectively. The Ra-226 concentration was obtained from the count rates of the 242, 295, 352, 609 1120 and 1765 keV peak, belonging to radon daughters. The radon exhalation was taken into account on the basis of the calculated disequilibrium between Ra-226 and Rn-222.
299	> 3 years	We analyzed the sample by gamma-spectrum and have achieved our results on the basis of radiochemistry: we know that the production of phosphogypsum separate U-234, U-238 and Th-238 from Ra-226; therefore we suppose the equilibrium of the U-238 decay chain before Ra-226, and attribute to all these nuclides the activity found for Pa-234m. The other values have been performed directly by gamma spectrum. We used a 500 cc Marinelli geometry, with reference to a 0.5 g/cm ³ density. The counting method is gamma spectrometry HPGE.	A 50 cc sample has been evaporated and dried on a 48 mm planchette, and then measured by a gas proportional counter. Since the aim is to find natural nuclides, the Th-230 energy is chosen as reference. The efficiency to self-absorption curve has been fitted counting planchette where we put the same Th-230 activities and different aliquots of solids. All measurements are performed by Berthold technology.
300	> 3 years	ISO 10703 and in-house method. Calibration sources Am-241, C-136 and Ra-226; Efficiency determination for alpha, beta and Ra-226; Opti Phase Hisafe 3; LSC Quantulus 1220 Wallac.	Sample was concentrated to 60 mL, Freeze drying 38 mL. Reference material QCY48 from Amersham; Dual efficiency calibration; 24h measurement.

APPENDIX II PERFORMANCE EVALUATION TABLES SORTED BY ANALYTE

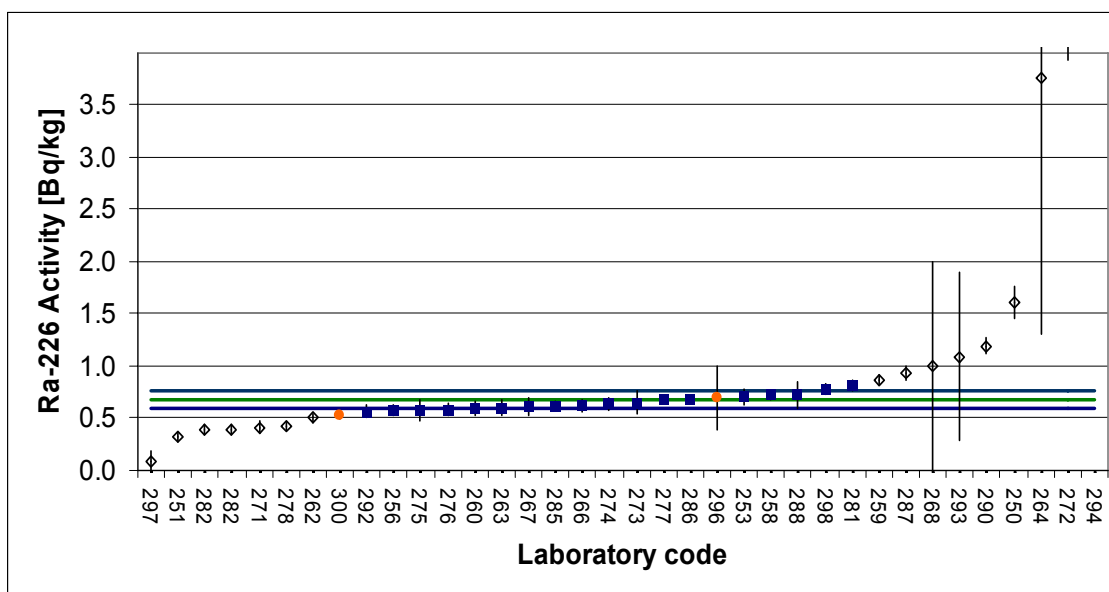
All results listed in this Appendix are expressed in Bq/kg units at a reference date set to 01 December 2008.

The abbreviations used in the Table header are explained below:

Rep. Value:	Reported measurement result value for the analyte of interest in Bq/kg.
Rep. Unc.	Reported standard measurement result uncertainty in Bq/kg.
Unc. [%]	Reported standard measurement result uncertainty in percentage.
Rel. Bias	Relative bias calculated according to formula (1) in paragraph 3.1.
A1 and A2	Evaluation estimators for trueness please see 3.4 in the report body.
True	Evaluation score for Trueness please see 3.4 in the report body.
P	Evaluation estimator for precision please see 3.4 in the report body.
Prec.	Evaluation score for Precision, please see 3.4 in the report body.
F	Evaluation estimator for evaluation of the blank sample 03.
A	Acceptable: The reported measurement result fulfils the PT criteria. Acceptable evaluation is denoted on the S-shape graphs with a bold-blue square.
W	Acceptable with Warning: Although the relative bias of the reported measurement result meets the PT criteria, but a flag was raised due to either underestimated or overestimated measurement result uncertainty. Acceptable with Warning evaluation is denoted on the S-shape graphs with a bold-orange circle.
N	Not Acceptable: The reported measurement result did not fulfil the PT criteria. Not Acceptable evaluation is denoted on the S-shape graphs with a black rhombus.

The evaluation results are presented in ascending order of the laboratory code.

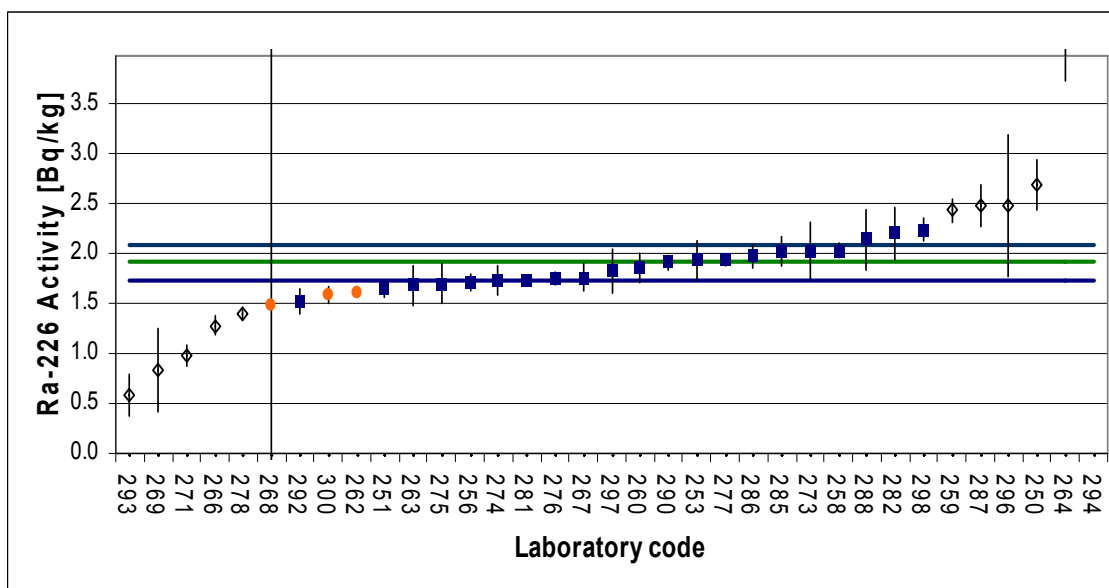
Performance evaluation of Ra-226 measurement results
Spiked water sample 01
Target Value: 0.69 ± 0.04 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	1.62	0.15	9.26	134.8	0.93	0.40	N	10.9	A	N
251	0.33	0.04	12.80	-52.5	0.36	0.15	N	14.1	A	N
253	0.72	0.07	10.35	3.6	0.03	0.22	A	11.9	A	A
256	0.59	0.05	7.84	-14.9	0.10	0.16	A	9.7	A	A
258	0.73	0.04	5.07	5.8	0.04	0.14	A	7.7	A	A
259	0.88	0.04	4.55	27.5	0.19	0.15	N	7.4	A	N
260	0.60	0.07	11.67	-13.0	0.09	0.21	A	13.0	A	A
262	0.51	0.04	7.84	-26.1	0.18	0.15	N	9.8	A	N
263	0.61	0.07	11.48	-11.6	0.08	0.21	A	12.9	A	A
264	3.76	2.44	64.95	444.6	3.07	6.30	A	65.2	N	N
266	0.63	0.06	9.52	-8.7	0.06	0.19	A	11.1	A	A
267	0.62	0.08	13.04	-10.0	0.07	0.23	A	14.27	A	A
268	1.00	1.00	100.00	44.9	0.31	2.58	A	100.2	N	N
271	0.42	0.06	14.42	-38.7	0.27	0.19	N	15.54	A	N
272	5.85	1.92	32.82	747.8	5.16	4.95	N	33.3	N	N
273	0.66	0.11	15.91	-4.3	0.03	0.29	A	16.93	A	A
274	0.65	0.06	8.94	-5.9	0.04	0.18	A	10.7	A	A
275	0.59	0.10	16.95	-14.5	0.10	0.28	A	17.91	A	A
276	0.59	0.06	10.37	-14.5	0.10	0.19	A	11.9	A	A
277	0.69	0.02	3.07	-0.7	0.00	0.12	A	6.56	A	A
278	0.43	0.03	6.98	-37.7	0.26	0.13	N	9.1	A	N

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
281	0.82	0.05	6.10	18.8	0.13	0.17	A	8.41	A	A
282	0.40	0.05	12.72	-41.9	0.29	0.17	N	13.98	A	N
285	0.62	0.04	7.06	-9.7	0.07	0.15	A	9.1	A	A
286	0.69	0.04	5.97	-0.4	0.00	0.15	A	8.32	A	A
287	0.94	0.06	6.38	36.2	0.25	0.19	N	8.6	A	N
288	0.73	0.12	16.44	5.8	0.04	0.33	A	17.43	A	A
290	1.20	0.07	5.83	73.9	0.51	0.21	N	8.2	A	N
292	0.57	0.06	9.76	-16.8	0.12	0.18	A	11.35	A	A
293	1.10	0.80	72.73	59.4	0.41	2.07	A	73.0	N	N
294	775	59	7.61	11221	774.3	152.2	N	9.6	A	N
296	0.70	0.30	42.86	1.4	0.01	0.78	A	43.25	N	W
297	0.10	0.10	100.00	-85.5	0.59	0.28	N	100.2	N	N
298	0.79	0.05	6.33	14.5	0.10	0.17	A	8.58	A	A
300	0.53	0.03	5.66	-23.2	0.16	0.13	N	8.1	A	W

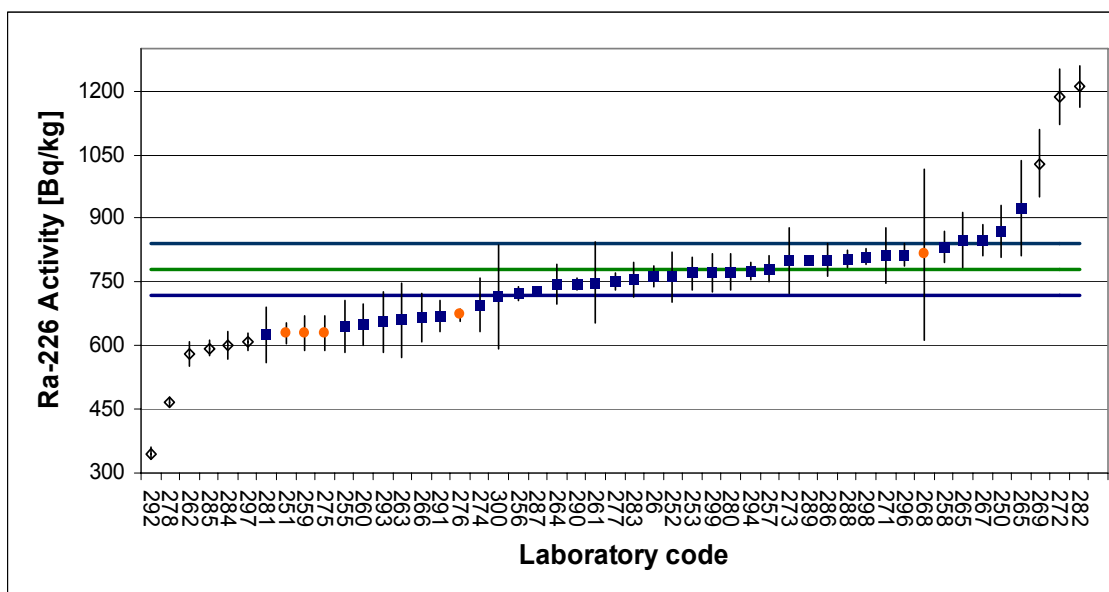
Performance evaluation of Ra-226 measurement results
Spiked water sample 02
Target Value: 1.93 ± 0.09 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	2.70	0.25	9.26	39.9	0.77	0.69	N	10.4	A	N
251	1.66	0.07	4.22	-14.0	0.27	0.29	A	6.3	A	A
253	1.95	0.20	10.00	1.0	0.02	0.55	A	11.0	A	A
256	1.73	0.09	5.20	-10.4	0.20	0.33	A	7.0	A	A
258	2.05	0.07	3.17	6.2	0.12	0.29	A	5.6	A	A
259	2.45	0.12	4.90	26.9	0.52	0.39	N	6.8	A	N
260	1.87	0.14	7.49	-3.1	0.06	0.43	A	8.8	A	A
262	1.62	0.04	2.47	-16.1	0.31	0.25	N	5.3	A	W
263	1.70	0.20	11.76	-11.9	0.23	0.57	A	12.7	A	A
264	6.04	2.30	38.08	213.1	4.11	5.94	A	38.4	N	N
266	1.30	0.10	7.69	-32.6	0.63	0.35	N	9.0	A	N
267	1.77	0.13	7.50	-8.1	0.16	0.41	A	8.8	A	A
268	1.50	3.00	200.00	-22.3	0.43	7.74	A	200.1	N	W
269	0.85	0.41	48.24	-56.0	1.08	1.08	A	48.5	N	N
271	1.00	0.10	10.00	-48.2	0.93	0.35	N	11.0	A	N
273	2.04	0.30	14.46	5.7	0.11	0.80	A	15.2	A	A
274	1.75	0.15	8.57	-9.3	0.18	0.45	A	9.8	A	A
275	1.71	0.20	11.70	-11.4	0.22	0.57	A	12.6	A	A
276	1.76	0.06	3.56	-8.7	0.17	0.28	A	5.9	A	A
277	1.95	0.06	2.92	1.0	0.02	0.27	A	5.5	A	A
278	1.41	0.06	4.26	-26.9	0.52	0.28	N	6.3	A	N

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
281	1.75	0.05	2.86	-9.3	0.18	0.27	A	5.5	A	A
282	2.22	0.26	11.71	15.0	0.29	0.71	A	12.6	A	A
285	2.03	0.15	7.18	5.4	0.10	0.44	A	8.6	A	A
286	1.99	0.11	5.53	3.1	0.06	0.37	A	7.2	A	A
287	2.50	0.20	8.00	29.5	0.57	0.57	N	9.3	A	N
288	2.16	0.30	13.89	11.9	0.23	0.81	A	14.7	A	A
290	1.93	0.08	4.15	0.0	0.00	0.31	A	6.2	A	A
292	1.54	0.13	8.44	-20.2	0.39	0.41	A	9.6	A	A
293	0.60	0.21	35.00	-68.9	1.33	0.59	N	35.3	N	N
294	2400.0	120.00	5.00	12425	2398.0	309.60	N	6.8	A	N
296	2.50	0.70	28.00	29.5	0.57	1.82	A	28.4	N	N
297	1.85	0.22	11.89	-4.1	0.08	0.61	A	12.8	A	A
298	2.26	0.12	5.31	17.1	0.33	0.39	A	7.1	A	A
300	1.6	0.08	5.00	-17.1	0.33	0.31	N	6.8	A	W

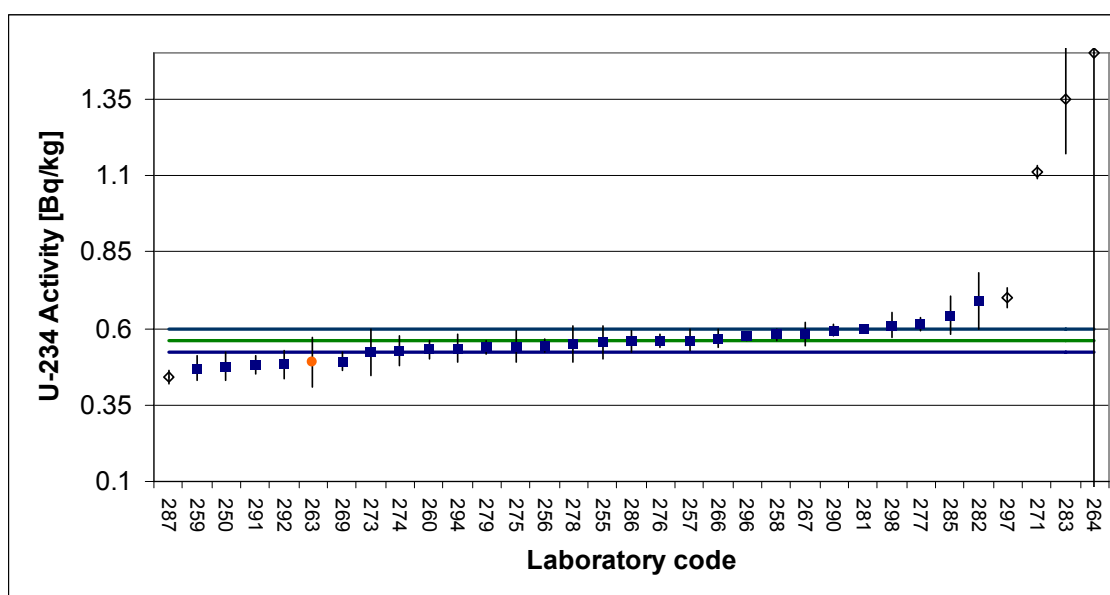
Performance evaluation of Ra-226 measurement results
Phosphogypsum IAEA-434 sample 6
Target Value: 780.0 ± 31.0 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
26	762	25	3.28	-2.3	18	103	A	5.2	A	A
250	870	62	7.13	11.5	90	179	A	8.2	A	A
251	630	24	3.81	-19.2	150	101	N	5.5	A	W
252	762	58	7.61	-2.3	18	170	A	8.6	A	A
253	770	40	5.19	-1.3	10	131	A	6.5	A	A
255	645	60	9.31	-17.4	135	174	A	10.1	A	A
256	722	17	2.35	-7.4	58	91	A	4.6	A	A
257	781	31	3.97	0.1	1	113	A	5.6	A	A
258	832	36	4.34	6.7	52	123	A	5.9	A	A
259	630	40	6.35	-19.2	150	131	N	7.5	A	W
260	650	50	7.69	-16.7	130	152	A	8.7	A	A
261	748	95	12.70	-4.1	32	258	A	13.3	A	A
262	580	27	4.66	-25.6	200	106	N	6.1	A	N
263	660	88	13.33	-15.4	120	241	A	13.9	A	A
264	745	45	6.04	-4.5	35	141	A	7.2	A	A
265	847	65	7.67	8.6	67	186	A	8.6	A	A
266	666	58	8.71	-14.6	114	170	A	9.6	A	A
267	849	35	4.12	8.8	69	121	A	5.7	A	A
268	815	200	24.54	4.5	35	522	A	24.9	N	W
269	1029	78	7.58	31.9	249	217	N	8.6	A	N
271	812	63	7.80	4.1	32	182	A	8.8	A	A
272	1186	64	5.35	52.1	406	182	N	6.7	A	N

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
273	800	75	9.42	2.6	20	210	A	10.2	A	A
274	696	63	9.05	-10.8	84	181	A	9.9	A	A
275	630	40	6.35	-19.2	150	131	N	7.5	A	W
276	672	14	2.14	-13.8	108	88	N	4.5	A	W
277	751	22	2.93	-3.7	29	98	A	4.9	A	A
278	467	10	2.14	-40.1	313	84	N	4.5	A	N
280	773	41	5.33	-0.8	7	133	A	6.6	A	A
281	625	65	10.40	-19.9	155	186	A	11.1	A	A
282	1211	50	4.13	55.3	431	152	N	5.7	A	N
283	755	41	5.43	-3.2	25	133	A	6.7	A	A
284	601	32	5.33	-23.0	179	115	N	6.6	A	N
285	595	18	3.08	-23.8	185	93	N	5.0	A	N
286	802	40	4.99	2.8	22	131	A	6.4	A	A
287	727	8	1.10	-6.8	53	83	A	4.1	A	A
288	803	20	2.49	2.9	23	95	A	4.7	A	A
289	802	7	0.82	2.8	22	82	A	4.1	A	A
290	745	15	2.01	-4.5	35	89	A	4.5	A	A
291	669	37	5.53	-14.2	111	125	A	6.8	A	A
292	346	16	4.49	-55.6	434	89	N	6.0	A	N
293	657	71	10.77	-15.7	123	199	A	11.5	A	A
294	776	20	2.58	-0.5	4	95	A	4.7	A	A
296	814	25	3.07	4.4	34	103	A	5.0	A	A
297	610	20	3.28	-21.8	170	95	N	5.2	A	N
298	810	20	2.47	3.8	30	95	A	4.7	A	A
299	771	45	5.82	-1.2	9	141	A	7.0	A	A
300	715	122	17.06	-8.3	65	325	A	17.5	A	A

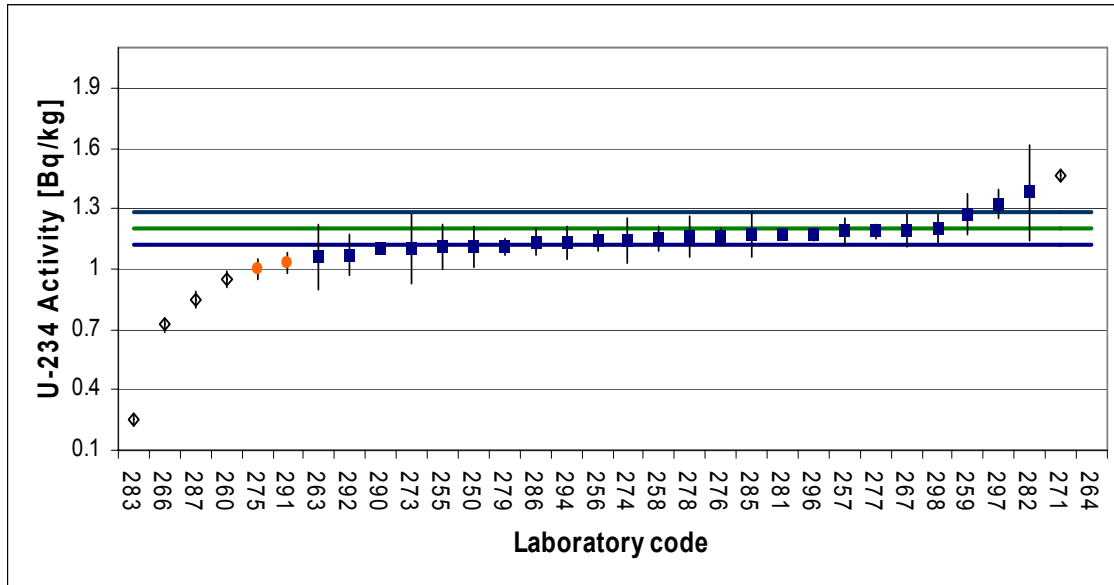
Performance evaluation of U-234 measurement results
Spiked water sample 1
Target Value: 0.56 ± 0.02 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	0.48	0.045	9.47	-15.2	0.09	0.13	A	10.1	A	A
255	0.55	0.052	9.39	-1.1	0.01	0.14	A	10.0	A	A
256	0.54	0.02	4.42	-3.0	0.02	0.08	A	5.7	A	A
257	0.56	0.03	6.06	0.2	0.00	0.10	A	7.0	A	A
258	0.58	0.02	3.45	3.6	0.02	0.07	A	5.0	A	A
259	0.47	0.04	8.51	-16.1	0.09	0.12	A	9.2	A	A
260	0.53	0.03	5.66	-5.4	0.03	0.09	A	6.7	A	A
263	0.49	0.08	16.33	-12.5	0.07	0.21	A	16.7	N	W
264	124.5	86.9	69.82	22132.1	123.94	224.28	A	69.9	N	N
266	0.57	0.03	5.29	1.2	0.01	0.09	A	6.4	A	A
267	0.58	0.038	6.55	3.6	0.02	0.11	A	7.5	A	A
269	0.49	0.03	6.12	-12.5	0.07	0.09	A	7.1	A	A
271	1.11	0.02	1.80	98.2	0.55	0.07	N	4.0	A	N
273	0.52	0.075	14.42	-7.1	0.04	0.20	A	14.9	A	A
274	0.53	0.048	9.09	-5.7	0.03	0.13	A	9.8	A	A
275	0.54	0.05	9.26	-3.6	0.02	0.14	A	9.9	A	A
276	0.56	0.02	3.66	-0.3	0.00	0.07	A	5.1	A	A
277	0.61	0.02	3.26	9.5	0.05	0.07	A	4.8	A	A
278	0.55	0.06	10.91	-1.8	0.01	0.16	A	11.5	A	A
279	0.54	0.02	4.10	-4.3	0.02	0.08	A	5.4	A	A
281	0.60	0.01	1.67	7.1	0.04	0.06	A	3.9	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
282	0.69	0.09	13.04	23.2	0.13	0.24	A	13.5	A	A
283	1.35	0.18	13.33	141.1	0.79	0.47	N	13.8	A	N
285	0.64	0.06	9.81	14.6	0.08	0.17	A	10.4	A	A
286	0.56	0.034	6.10	-0.5	0.00	0.10	A	7.1	A	A
287	0.44	0.02	4.55	-21.4	0.12	0.07	N	5.8	A	N
290	0.59	0.02	3.04	5.9	0.03	0.07	A	4.7	A	A
291	0.48	0.03	6.25	-14.3	0.08	0.09	A	7.2	A	A
292	0.48	0.05	9.54	-13.9	0.08	0.13	A	10.2	A	A
294	0.53	0.046	8.61	-4.6	0.03	0.13	A	9.3	A	A
296	0.57	0.012	2.09	2.3	0.01	0.06	A	4.1	A	A
297	0.70	0.03	4.29	25.0	0.14	0.09	N	5.6	A	N
298	0.61	0.04	6.56	8.9	0.05	0.12	A	7.5	A	A

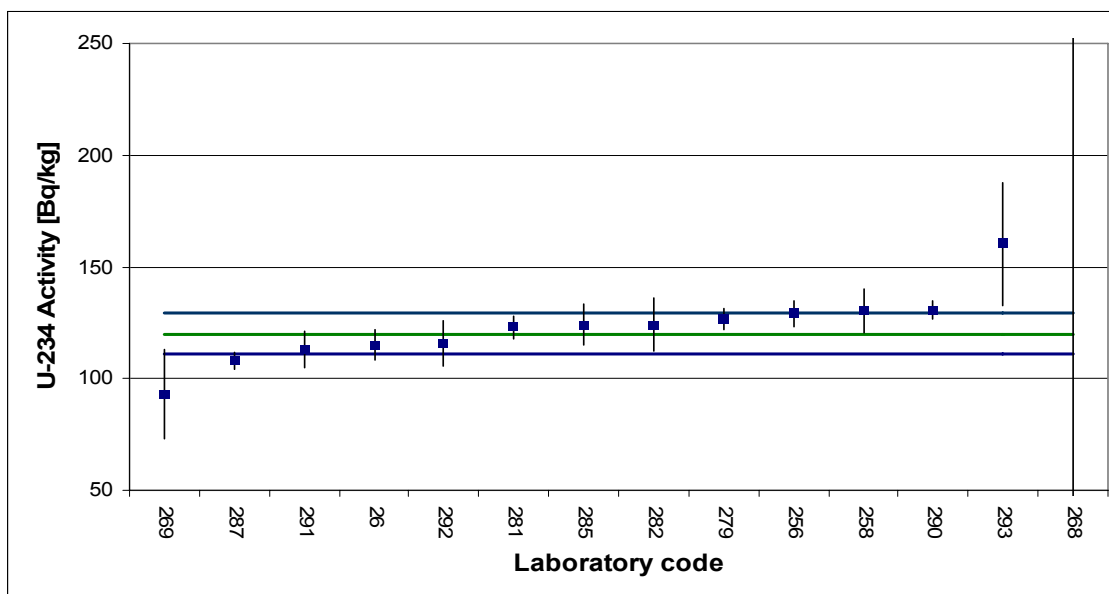
Performance evaluation of U-234 measurement results
Spiked water sample 02
Target Value: 1.2 ± 0.04 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	1.11	0.10	9.01	-7.5	0.09	0.28	A	9.6	A	A
255	1.11	0.11	9.93	-7.7	0.09	0.30	A	10.5	A	A
256	1.14	0.05	4.39	-5.0	0.06	0.17	A	5.5	A	A
257	1.19	0.07	5.48	-1.1	0.01	0.20	A	6.4	A	A
258	1.15	0.06	5.13	-4.2	0.05	0.18	A	6.1	A	A
259	1.27	0.10	7.87	5.8	0.07	0.28	A	8.6	A	A
260	0.95	0.04	4.21	-20.8	0.25	0.15	N	5.4	A	N
263	1.06	0.16	15.09	-11.7	0.14	0.43	A	15.5	A	A
264	95.02	61.7	65	7818	93.82	159.34	A	65.1	N	N
266	0.72	0.04	5.11	-39.7	0.48	0.14	N	6.1	A	N
267	1.19	0.08	6.88	-0.7	0.01	0.24	A	7.6	A	A
271	1.46	0.03	2.05	21.7	0.26	0.13	N	3.9	A	N
273	1.10	0.17	15.45	-8.3	0.10	0.45	A	15.8	A	A
274	1.14	0.11	9.65	-5.0	0.06	0.30	A	10.2	A	A
275	1.00	0.05	5.00	-16.7	0.20	0.17	N	6.0	A	W
276	1.16	0.04	3.40	-3.0	0.04	0.15	A	4.8	A	A
277	1.19	0.04	2.94	-0.8	0.01	0.14	A	4.4	A	A
278	1.16	0.10	8.62	-3.3	0.04	0.28	A	9.2	A	A
279	1.11	0.04	3.60	-7.5	0.09	0.15	A	4.9	A	A
281	1.17	0.02	1.71	-2.5	0.03	0.12	A	3.7	A	A
282	1.38	0.24	17.39	15.0	0.18	0.63	A	17.7	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
283	0.25	0.03	12.00	-79.2	0.95	0.13	N	12.5	A	N
285	1.17	0.11	9.41	-2.6	0.03	0.30	A	10.0	A	A
286	1.13	0.07	5.84	-5.8	0.07	0.20	A	6.7	A	A
287	0.85	0.04	4.71	-29.2	0.35	0.15	N	5.8	A	N
290	1.10	0.02	1.64	-8.6	0.10	0.11	A	3.7	A	A
291	1.03	0.05	4.85	-14.2	0.17	0.17	N	5.9	A	W
292	1.07	0.10	9.35	-10.8	0.13	0.28	A	9.9	A	A
294	1.13	0.08	7.24	-5.6	0.07	0.24	A	8.0	A	A
296	1.17	0.03	2.56	-2.5	0.03	0.13	A	4.2	A	A
297	1.32	0.07	5.30	10.0	0.12	0.21	A	6.3	A	A
298	1.20	0.07	5.83	0.0	0.00	0.21	A	6.7	A	A

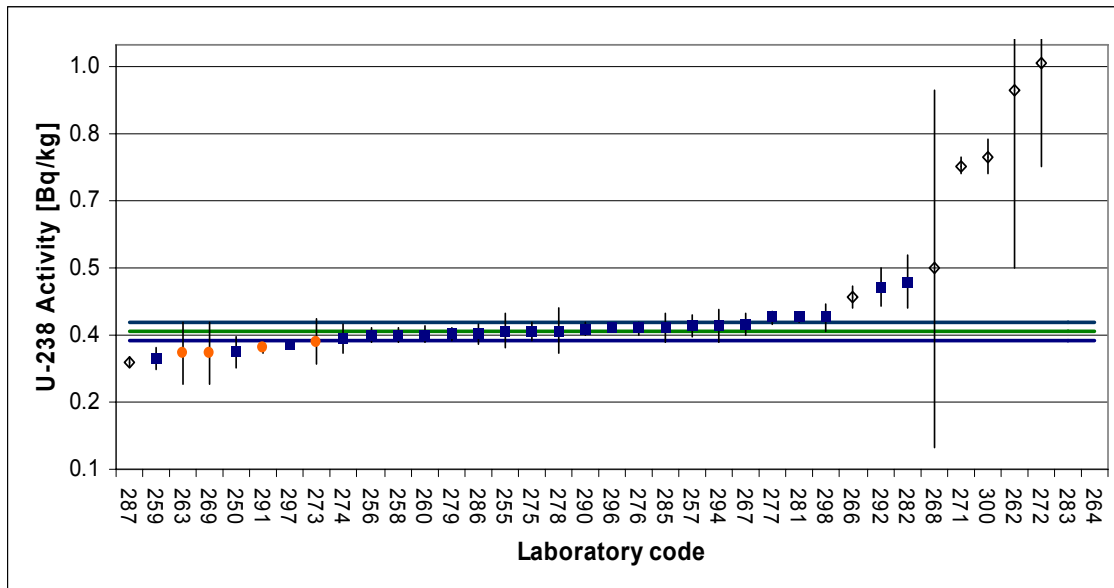
Performance evaluation of U-234 measurement results
Phosphogypsum IAEA-434 sample 6
Target Value: 120.0 ± 4.5 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
26	115	6.8	5.9	-4.1	5	21	A	7.0	A	A
250	156	15.0	9.6	30.0	36	40	A	10.3	A	A
256	129	6.0	4.7	7.5	9	19	A	6.0	A	A
258	130	10.1	7.7	8.7	10	29	A	8.6	A	A
260	101	6.0	5.9	-15.8	19	19	A	7.0	A	A
261	120	17.0	14.2	0.0	0	45	A	14.7	A	A
263	130	15.0	11.5	8.3	10	40	A	12.1	A	A
264	5094	3131.0	61.5	4144.8	4974	8078	A	61.6	N	N
266	91	4.7	5.2	-24.6	30	17	N	6.4	A	N
267	140	5.0	3.6	16.7	20	17	N	5.2	A	W
268	2808	3422	121.9	2240.0	2688	8829	A	121.9	N	N
269	93	20.0	21.5	-22.5	27	53	A	21.8	N	N
271	132	4.9	3.7	10.1	12	17	A	5.3	A	A
273	118	20.7	17.5	-1.6	2	55	A	17.9	A	A
274	124	12.0	9.7	3.3	4	33	A	10.4	A	A
275	116	7.0	6.0	-3.3	4	21	A	7.1	A	A
276	120	4.1	3.4	0.2	0	16	A	5.1	A	A
277	131	3.3	2.5	9.2	11	14	A	4.5	A	A
278	121	8.0	6.6	0.8	1	24	A	7.6	A	A
279	127	4.7	3.7	5.5	7	17	A	5.3	A	A
281	123	5.0	4.1	2.5	3	17	A	5.5	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
282	124	12.1	9.7	3.5	4	33	A	10.4	A	A
283	92	10.0	10.9	-23.3	28	28	A	11.5	A	A
285	124	9.2	7.4	3.5	4	26	A	8.3	A	A
286	117	7.8	6.7	-2.8	3	23	A	7.7	A	A
287	108	4.0	3.7	-10.0	12	16	A	5.3	A	A
290	131	4.0	3.1	8.8	11	16	A	4.8	A	A
291	113	8.0	7.1	-5.8	7	24	A	8.0	A	A
292	116	10.2	8.8	-3.3	4	29	A	9.5	A	A
293	160	27.3	17.0	33.5	40	71	A	17.4	A	A
294	57	3.5	6.1	-52.5	63	15	N	7.2	A	N
296	122	3.0	2.5	1.7	2	14	A	4.5	A	A
297	150	15.0	10.0	25.0	30	40	A	10.7	A	A
299	114	23.5	20.6	-5.3	6	62	A	21.0	N	W

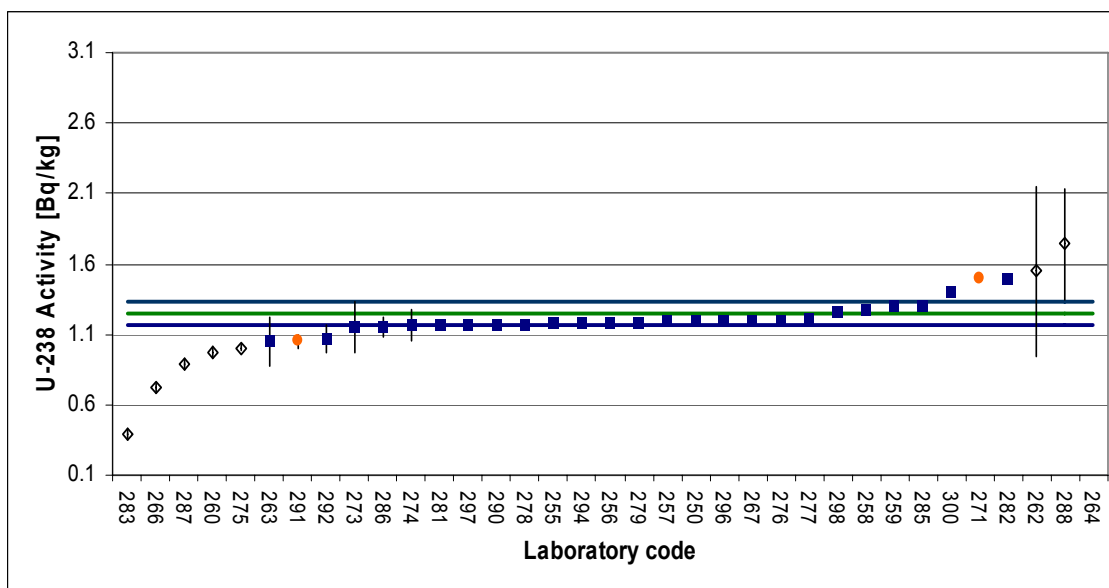
Performance evaluation of U-238 measurement results
Spiked water sample 01
Target Value: 0.36 ± 0.01 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	0.31	0.03	10.90	-13.3	0.05	0.09	A	11.2	A	A
255	0.36	0.04	10.56	0.0	0.00	0.10	A	10.9	A	A
256	0.35	0.02	4.86	-2.8	0.01	0.05	A	5.6	A	A
257	0.37	0.02	6.47	3.1	0.01	0.07	A	7.0	A	A
258	0.35	0.02	4.29	-2.8	0.01	0.05	A	5.1	A	A
259	0.30	0.02	8.11	-17.8	0.06	0.07	A	8.6	A	A
260	0.35	0.02	5.13	-2.5	0.01	0.05	A	5.8	A	A
262	0.90	0.40	44.44	150.0	0.54	1.03	A	44.5	N	N
263	0.31	0.07	22.58	-13.9	0.05	0.18	A	22.8	N	W
264	3.76	2.44	64.95	943.9	3.40	6.30	A	65.0	N	N
266	0.44	0.02	5.52	20.8	0.08	0.07	N	6.2	A	N
267	0.38	0.03	6.67	4.2	0.02	0.07	A	7.2	A	A
268	0.50	0.40	80.00	38.9	0.14	1.03	A	80.0	N	N
269	0.31	0.07	22.58	-13.9	0.05	0.18	A	22.8	N	W
271	0.73	0.02	2.33	102.8	0.37	0.05	N	3.6	A	N
272	0.96	0.23	23.96	166.7	0.60	0.59	N	24.1	N	N
273	0.34	0.05	14.88	-6.7	0.02	0.13	A	15.1	N	W
274	0.34	0.03	9.06	-5.0	0.02	0.08	A	9.5	A	A
275	0.36	0.02	5.56	0.0	0.00	0.06	A	6.2	A	A
276	0.37	0.01	3.80	1.6	0.01	0.04	A	4.7	A	A
277	0.39	0.02	3.85	8.3	0.03	0.05	A	4.7	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
278	0.36	0.05	13.89	0.0	0.00	0.13	A	14.2	A	A
279	0.35	0.02	4.25	-1.9	0.01	0.05	A	5.1	A	A
281	0.39	0.01	2.56	8.3	0.03	0.04	A	3.8	A	A
282	0.47	0.06	12.77	30.6	0.11	0.16	A	13.1	A	A
283	2.74	0.25	9.12	661.1	2.38	0.65	N	9.5	A	N
285	0.37	0.03	8.74	1.7	0.01	0.09	A	9.2	A	A
286	0.35	0.02	6.23	-1.9	0.01	0.06	A	6.8	A	A
287	0.29	0.01	3.45	-19.4	0.07	0.04	N	4.4	A	N
290	0.36	0.01	3.85	1.1	0.00	0.04	A	4.7	A	A
291	0.32	0.01	3.13	-11.1	0.04	0.04	N	4.2	A	W
292	0.46	0.04	8.95	27.2	0.10	0.11	A	9.4	A	A
294	0.37	0.04	9.43	3.1	0.01	0.09	A	9.8	A	A
296	0.37	0.01	2.19	1.4	0.01	0.03	A	3.5	A	A
297	0.33	0.01	3.03	-8.3	0.03	0.04	A	4.1	A	A
298	0.39	0.03	7.69	8.3	0.03	0.08	A	8.2	A	A
300	0.75	0.04	5.33	108.3	0.39	0.11	N	6.0	A	N

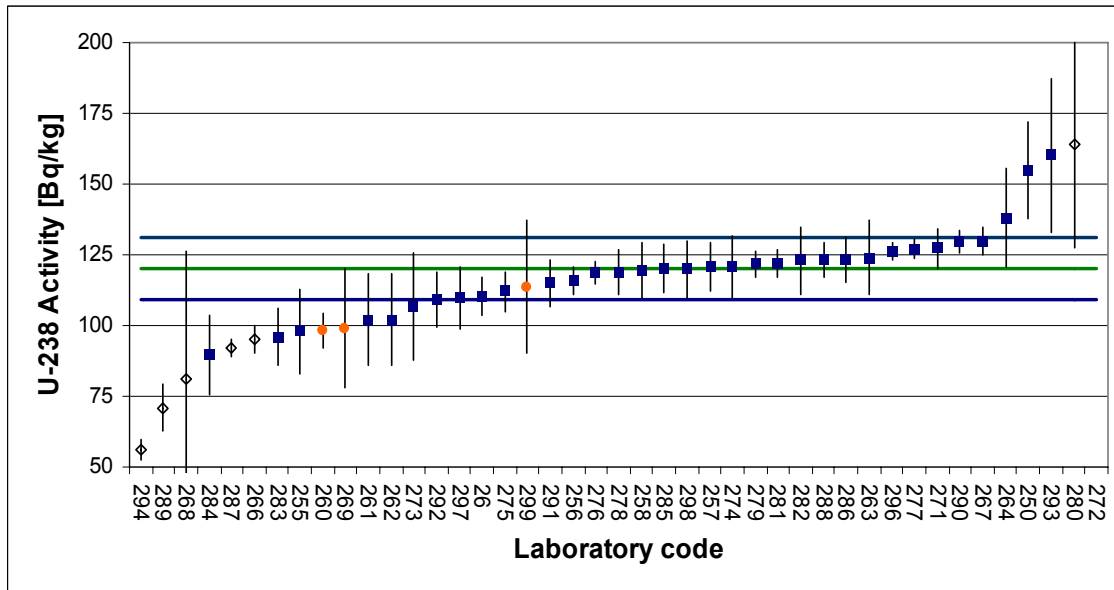
**Performance evaluation of U-238 measurement results
Spiked water sample 2
Target Value: 1.25 +/- 0.04 [Bq/kg]**



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	1.19	0.11	9.24	-4.8	0.06	0.30	A	9.8	A	A
255	1.18	0.12	10.20	-5.9	0.07	0.33	A	10.7	A	A
256	1.18	0.05	4.24	-5.6	0.07	0.17	A	5.3	A	A
257	1.19	0.07	5.47	-5.0	0.06	0.20	A	6.3	A	A
258	1.28	0.06	4.92	2.4	0.03	0.19	A	5.9	A	A
259	1.30	0.11	8.46	4.0	0.05	0.30	A	9.0	A	A
260	0.97	0.04	4.12	-22.4	0.28	0.15	N	5.2	A	N
262	1.55	0.60	38.71	24.0	0.30	1.55	A	38.8	N	N
263	1.05	0.17	16.19	-16.0	0.20	0.45	A	16.5	A	A
264	6.04	2.30	38.08	383.4	4.79	5.94	A	38.2	N	N
266	0.72	0.04	5.15	-42.5	0.53	0.14	N	6.1	A	N
267	1.19	0.08	6.88	-4.6	0.06	0.24	A	7.6	A	A
271	1.49	0.02	1.34	19.2	0.24	0.12	N	3.5	A	W
273	1.15	0.18	15.65	-8.0	0.10	0.48	A	16.0	A	A
274	1.16	0.11	9.48	-7.2	0.09	0.30	A	10.0	A	A
275	1.00	0.02	2.00	-20.0	0.25	0.12	N	3.8	A	N
276	1.20	0.04	3.39	-4.4	0.05	0.15	A	4.7	A	A
277	1.21	0.04	2.89	-3.2	0.04	0.14	A	4.3	A	A
278	1.17	0.10	8.55	-6.4	0.08	0.28	A	9.1	A	A
279	1.18	0.04	3.39	-5.6	0.07	0.15	A	4.7	A	A
281	1.16	0.02	1.72	-7.2	0.09	0.12	A	3.6	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
282	1.50	0.26	17.33	20.0	0.25	0.68	A	17.6	A	A
283	0.39	0.04	10.26	-68.8	0.86	0.15	N	10.7	A	N
285	1.30	0.12	9.31	4.0	0.05	0.33	A	9.8	A	A
286	1.15	0.07	5.81	-7.8	0.10	0.20	A	6.6	A	A
287	0.89	0.04	4.49	-28.8	0.36	0.15	N	5.5	A	N
288	1.74	0.39	22.41	39.2	0.49	1.01	A	22.6	N	N
290	1.16	0.02	1.55	-7.0	0.09	0.11	A	3.6	A	A
291	1.05	0.05	4.76	-16.0	0.20	0.17	N	5.7	A	W
292	1.07	0.10	9.35	-14.4	0.18	0.28	A	9.9	A	A
294	1.18	0.09	7.22	-5.8	0.07	0.24	A	7.9	A	A
296	1.19	0.03	2.52	-4.8	0.06	0.13	A	4.1	A	A
297	1.16	0.03	2.59	-7.2	0.09	0.13	A	4.1	A	A
298	1.26	0.07	5.56	0.8	0.01	0.21	A	6.4	A	A
300	1.4	0.07	5.00	12.0	0.15	0.21	A	5.9	A	A

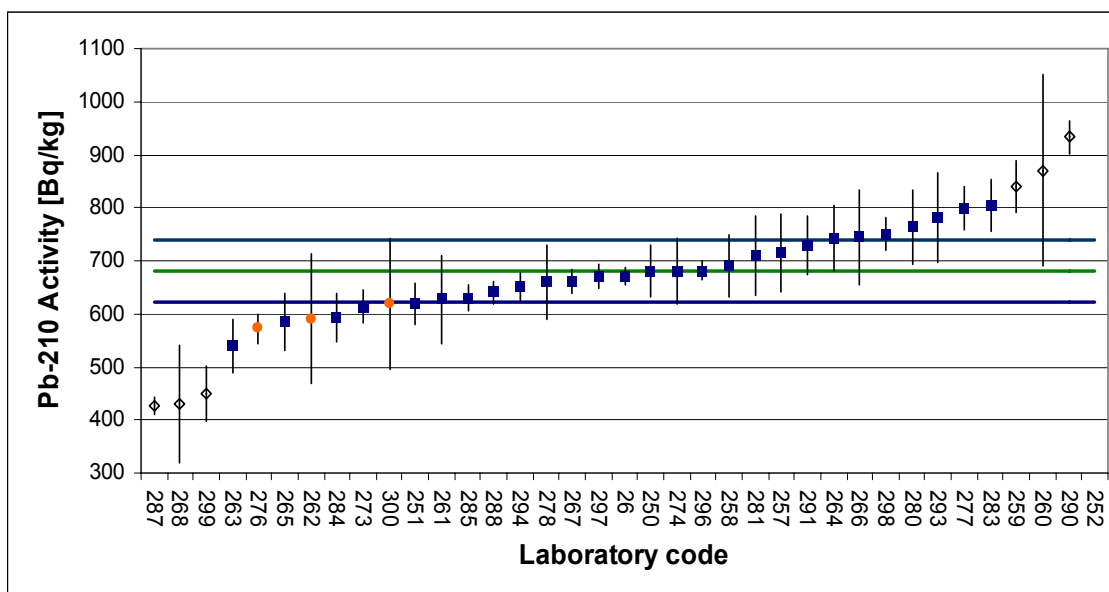
Performance evaluation of U-238 measurement results
Phosphogypsum IAEA-434 sample 6
Target Value: 120.0 ± 5.5 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
26	110	6.5	26	110	9.70	21.97	A	7.5	A	A
250	155	17	250	155	35.00	46.10	A	11.9	A	A
255	98	15	255	98	22.10	41.22	A	16.0	A	A
256	116	5.0	256	116	4.00	19.18	A	6.3	A	A
257	121	8.5	257	121	0.60	26.12	A	8.4	A	A
258	120	9.7	258	120	0.40	28.77	A	9.3	A	A
260	98	6	260	98	22.00	21.00	N	7.6	A	W
261	102	16	261	102	18.00	43.65	A	16.3	A	A
262	102	16	262	102	18.00	43.65	A	16.3	A	A
263	124	13	263	124	4.00	36.42	A	11.4	A	A
264	138	17.2	264	138	18.00	46.59	A	13.3	A	A
266	95	4.9	266	95	25.00	19.00	N	6.9	A	N
267	130	5	267	130	10.00	19.18	A	6.0	A	A
268	81	45	268	81	39.00	116.96	A	55.7	N	N
269	99	21	269	99	21.00	56.01	A	21.7	N	W
271	127	7.03	271	127	7.40	23.03	A	7.2	A	A
272	722	34.6	272	722	602.15	90.44	N	6.6	A	N
273	107	18.8	273	107	13.10	50.54	A	18.2	A	A
274	121	11	274	121	1.00	31.73	A	10.2	A	A
275	112	7	275	112	8.00	22.97	A	7.8	A	A
276	119	4.0	276	119	1.40	17.61	A	5.7	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
277	127	3.2	277	127	7.00	16.42	A	5.2	A	A
278	119	8	278	119	1.00	25.05	A	8.1	A	A
279	122	4.51	279	122	1.84	18.35	A	5.9	A	A
280	164	36	280	164	43.90	94.47	A	22.6	N	N
281	122	5	281	122	2.00	19.18	A	6.1	A	A
282	123	12	282	123	3.00	34.06	A	10.8	A	A
283	96	10	283	96	24.00	29.44	A	11.4	A	A
284	90	14.0	284	90	30.20	38.81	A	16.2	A	A
285	120	8.5	285	120	0.02	26.12	A	8.4	A	A
286	123	8.2	286	123	3.20	25.47	A	8.1	A	A
287	92	3.00	287	92	28.00	16.16	N	5.6	A	N
288	123	6	288	123	3.00	21.00	A	6.7	A	A
289	71	8.22	289	71	49.19	25.52	N	12.5	A	N
290	130	4	290	130	9.80	17.55	A	5.5	A	A
291	115	8	291	115	5.00	25.05	A	8.3	A	A
292	109	9.7	292	109	10.92	28.75	A	10.0	A	A
293	160	27.3	293	160	40.20	71.85	A	17.6	A	A
294	56	3.5	294	56	64.00	16.82	N	7.8	A	N
296	126	3	296	126	6.00	16.16	A	5.2	A	A
297	110	11	297	110	10.00	31.73	A	11.0	A	A
298	120	10	298	120	0.00	29.44	A	9.5	A	A
299	114	23.46	299	114	6.31	62.17	A	21.1	N	W

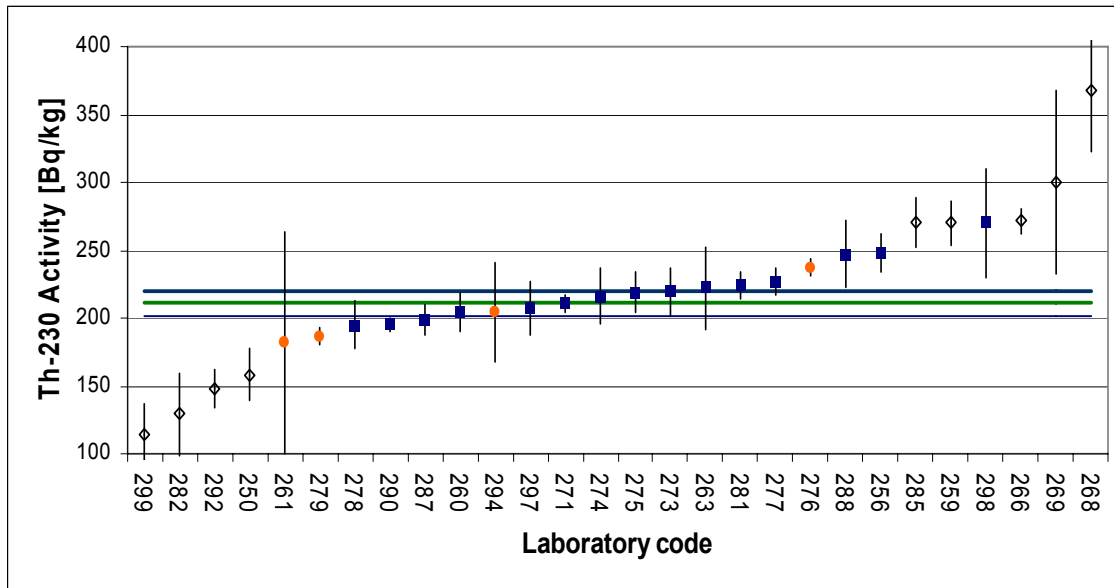
Performance evaluation of Pb-210 measurement results
Phosphogypsum IAEA-434 sample 6
Target Value: 680.0 ± 29.0 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
26	671	15	2.3	-1.3	9	85	A	4.8	A	A
250	681	49	7.2	0.1	1	147	A	8.4	A	A
251	620	39	6.3	-8.8	60	125	A	7.6	A	A
252	1276	105	8.2	87.6	596	281	N	9.3	A	N
257	716	73	10.2	5.3	36	203	A	11.1	A	A
258	691	58	8.4	1.6	11	167	A	9.4	A	A
259	840	50	6.0	23.5	160	149	N	7.3	A	N
260	870	180	20.7	27.9	190	470	A	21.1	N	N
261	627	82	13.1	-7.8	53	224	A	13.8	A	A
262	590	122	20.7	-13.2	90	324	A	21.1	N	W
263	540	51	9.4	-20.6	140	151	A	10.4	A	A
264	742	62	8.4	9.1	62	177	A	9.4	A	A
265	585	54	9.2	-14.0	95	158	A	10.2	A	A
266	744	91	12.2	9.4	64	245	A	12.9	A	A
267	662	23	3.5	-2.6	18	95	A	5.5	A	A
268	430	112	26.0	-36.8	250	298	A	26.4	N	N
273	614	31	5.0	-9.8	67	109	A	6.6	A	A
274	681	62	9.1	0.1	1	177	A	10.1	A	A
276	572	28	4.9	-15.8	108	104	N	6.5	A	W
277	799	40	5.0	17.5	119	127	A	6.6	A	A
278	660	70	10.6	-2.9	20	195	A	11.4	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
280	765	70	9.1	12.5	85	195	A	10.1	A	A
281	711	75	10.5	4.6	31	207	A	11.4	A	A
283	804	50	6.2	18.2	124	149	A	7.5	A	A
284	592	45	7.6	-12.9	88	138	A	8.7	A	A
285	629	24	3.9	-7.5	51	98	A	5.8	A	A
287	428	16	3.7	-37.1	252	85	N	5.7	A	N
288	640	21	3.3	-5.9	40	92	A	5.4	A	A
290	933	31	3.3	37.2	253	110	N	5.4	A	N
291	728	55	7.6	7.1	48	160	A	8.7	A	A
293	782	86	11.0	14.9	102	234	A	11.8	A	A
294	651	26	4.0	-4.3	29	100	A	5.8	A	A
296	682	17	2.5	0.3	2	87	A	4.9	A	A
297	670	23	3.4	-1.5	10	95	A	5.5	A	A
298	750	30	4.0	10.3	70	108	A	5.8	A	A
299	449	53	11.7	-34.0	231	155	N	12.4	A	N
300	619	124	20.0	-9.0	61	329	A	20.5	N	W

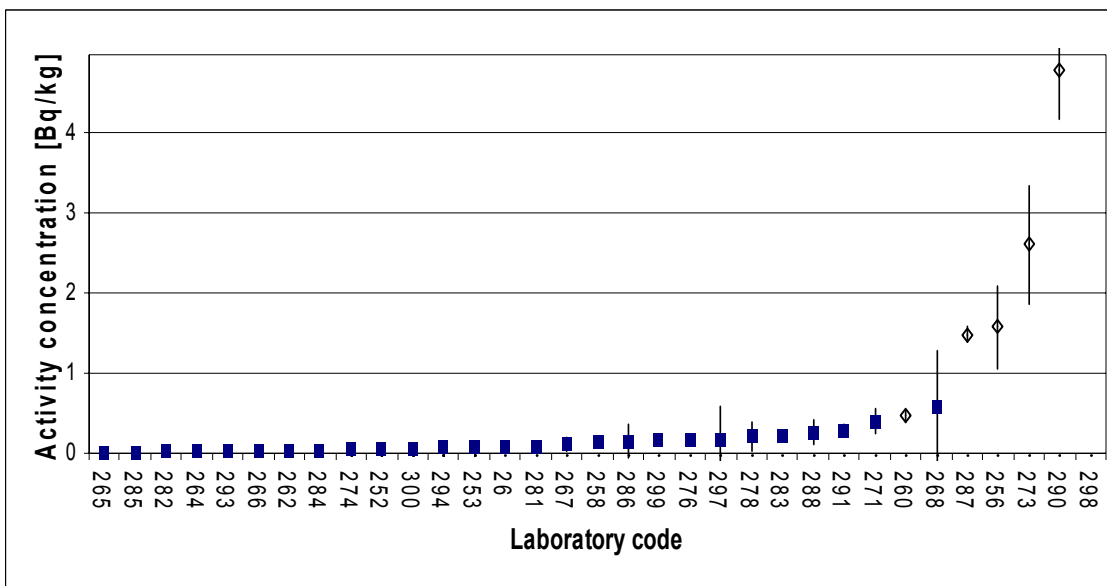
Performance evaluation of Th-230 measurement results
Phosphogypsum IAEA-434 sample 6
Target Value: 211.0 ± 4.5 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
250	158	19	12.0	-25.1	53	50	N	12.2	A	N
256	248	14	5.6	17.5	37	38	A	6.0	A	A
259	270	16	5.9	28.0	59	43	N	6.3	A	N
260	204	14	6.9	-3.3	7	38	A	7.2	A	A
261	182	82	45.1	-13.7	29	212	A	45.1	N	W
263	222	30	13.5	5.2	11	78	A	13.7	A	A
266	271	9	3.4	28.5	60	26	N	4.0	A	N
268	367	45	12.3	73.9	156	117	N	12.4	A	N
269	300	67	22.3	42.2	89	173	A	22.4	N	N
271	211	6	3.0	0.0	0	20	A	3.7	A	A
273	219	17	7.8	3.8	8	45	A	8.0	A	A
274	216	20	9.3	2.4	5	53	A	9.5	A	A
275	219	15	6.8	3.8	8	40	A	7.2	A	A
276	237	6	2.7	12.3	26	20	N	3.5	A	W
277	227	10	4.2	7.6	16	27	A	4.7	A	A
278	195	18	9.2	-7.6	16	48	A	9.5	A	A
279	187	6	3.1	-11.6	24	19	N	3.7	A	W
281	224	10	4.5	6.2	13	28	A	4.9	A	A
282	129	30	23.3	-38.9	82	78	N	23.4	N	N
285	270	18	6.8	27.9	59	48	N	7.1	A	N
287	199	11	5.5	-5.7	12	31	A	5.9	A	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	A1	A2	True	P	Prec	Final Score
288	247	25	10.1	17.1	36	66	A	10.3	A	A
290	195	5	2.5	-7.4	16	17	A	3.3	A	A
292	148	14	9.3	-30.0	63	37	N	9.5	A	N
294	204	37	18.1	-3.3	7	96	A	18.3	N	W
297	207	20	9.7	-1.9	4	53	A	9.9	A	A
298	270	40	14.8	28.0	59	104	A	15.0	A	A
299	114	23	20.6	-46.1	97	62	N	20.7	N	N

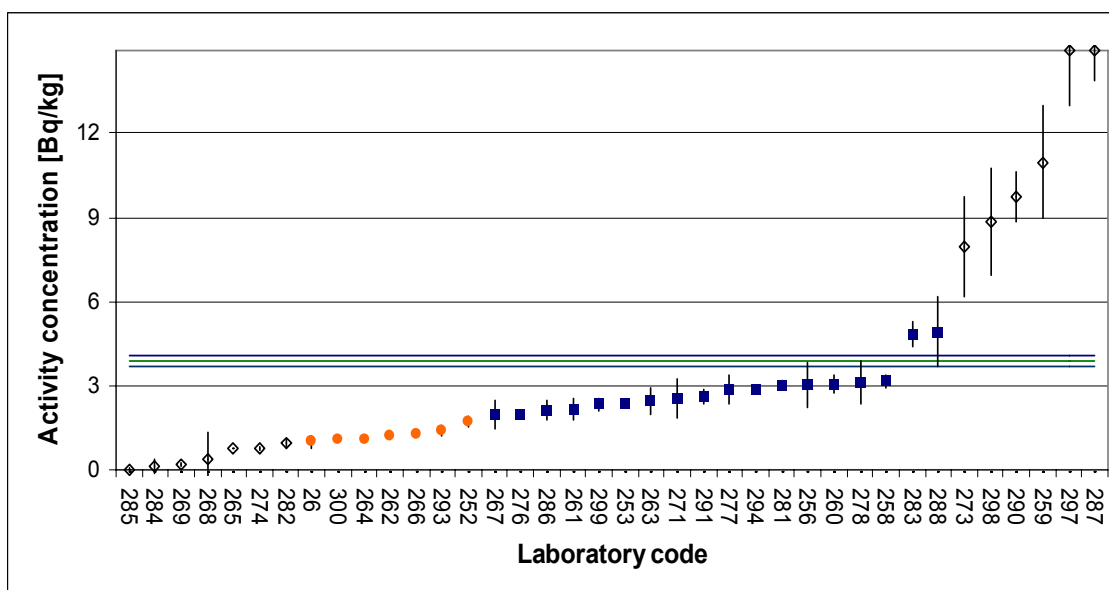
**Performance evaluation gross alpha measurement results
Water Sample 3 (Blank water)**



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	F	Final Score
26	0.11	0.06	55	-0.01	A
252	0.07	0.007	9	0.06	A
253	0.10	0.015	16	0.07	A
256	1.60	0.52	33	0.56	N
258	0.15	0.02	15	0.10	A
260	0.48	0.08	17	0.32	N
262	0.06	0.04	63	-0.02	A
264	0.04	0.005	11	0.03	A
265	0.03	0.004	16	0.02	A
266	0.06	0.001	2	0.06	A
267	0.13	0.09	69	-0.05	A
268	0.60	0.70	117	-0.80	A
271	0.42	0.15	37	0.11	A
273	2.63	0.74	28	1.15	N
274	0.06	0.01	21	0.04	A
276	0.19	0.033	17	0.13	A
278	0.23	0.19	83	-0.15	A
281	0.11	0.04	36	0.03	A
282	0.04	0.007	18	0.03	A
283	0.25	0.04	16	0.17	A
284	<0.777	0.00	-	-	A
285	0.03	0.00	9	0.03	A
286	0.17	0.20	118	-0.23	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	F	Final Score
287	1.50	0.10	7	1.30	N
288	0.28	0.15	54	-0.02	A
290	4.80	0.60	13	3.60	N
291	0.31	0.06	19	0.19	A
293	0.05	0.05	100	-0.05	A
294	0.09	0.02	24	0.05	A
297	0.20	0.40	200	-0.60	A
298	26.50	3.5	13	19.50	N
299	0.19	0.032	17	0.12	A
300	0.08	0.004	5	0.07	A

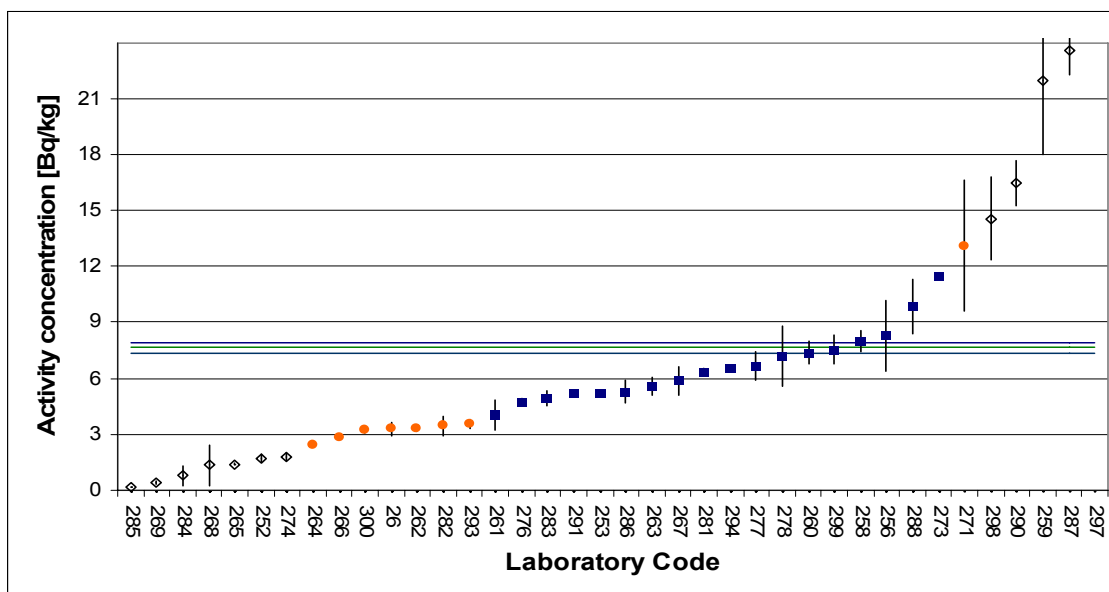
Performance evaluation of gross alpha measurement results
Spiked water sample 4
Target Value: 3.93 ± 0.08 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
26	1.04	0.2	19.23	-73.5	W
252	1.78	0.18	10.11	-54.7	W
253	2.39	0.09	3.77	-39.2	A
256	3.08	0.81	26.30	-21.6	A
258	3.20	0.22	6.88	-18.6	A
259	11.00	2.00	18.18	179.9	N
260	3.10	0.30	9.68	-21.1	A
261	2.20	0.4	18.18	-44.0	A
262	1.24	0.11	8.62	-68.4	W
263	2.50	0.50	20.00	-36.4	A
264	1.15	0.07	5.81	-70.7	W
265	0.80	0.015	1.88	-79.7	N
266	1.31	0.02	1.45	-66.7	W
267	2.00	0.50	25.00	-49.1	A
268	0.40	1.00	250.00	-89.8	N
269	0.26	0.05	19.22	-93.5	N
271	2.61	0.716	27.44	-33.6	A
273	8.00	1.80	22.50	103.6	N
274	0.82	0.08	10.02	-79.2	N
276	2.03	0.11	5.17	-48.4	A
277	2.90	0.50	17.24	-26.2	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
278	3.15	0.77	24.44	-19.8	A
281	3.05	0.15	4.92	-22.4	A
282	0.97	0.15	14.99	-75.2	N
283	4.91	0.44	8.96	24.9	A
284	0.18	0.25	138.67	-95.4	N
285	0.08	0.01	7.41	-97.9	N
286	2.18	0.36	16.51	-44.5	A
287	20.10	1.10	5.47	411.5	N
288	4.95	1.24	25.05	26.0	A
290	9.80	0.90	9.18	149.4	N
291	2.65	0.26	9.81	-32.6	A
293	1.46	0.18	12.33	-62.8	W
294	2.91	0.08	2.68	-26.0	A
297	15.90	1.99	12.52	304.6	N
298	8.90	1.9	21.35	126.5	N
299	2.38	0.24	10.00	-39.4	A
300	1.1	0.06	5.45	-72.0	W

Performance evaluation of gross alpha measurement results
Spiked water sample 5
Target Value: 7.68 ± 0.15 [Bq/kg]

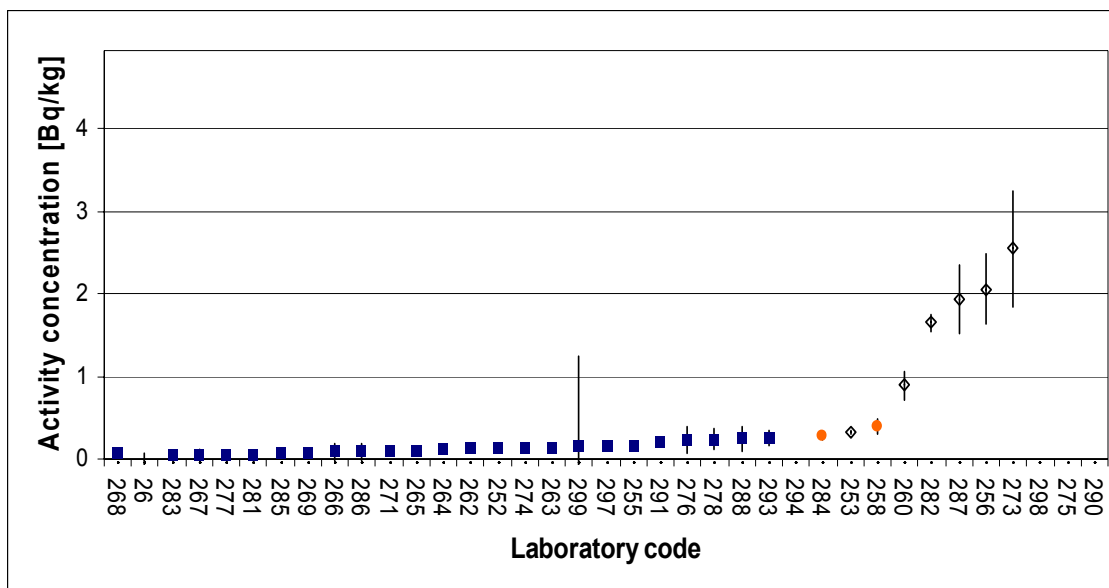


Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
26	3.32	0.38	11.45	-56.8	W
252	1.77	0.18	10.17	-77.0	N
253	5.21	0.2	3.84	-32.2	A
256	8.33	1.92	23.05	8.5	A
258	8.00	0.56	7.00	4.2	A
259	22.00	4.00	18.18	186.5	N
260	7.40	0.60	8.11	-3.6	A
261	4.10	0.80	19.51	-46.6	A
262	3.36	0.18	5.45	-56.3	W
263	5.60	0.50	8.93	-27.1	A
264	2.50	0.11	4.58	-67.4	W
265	1.46	0.02	1.37	-81.0	N
266	2.90	0.04	1.52	-62.2	W
267	5.90	0.80	13.56	-23.2	A
268	1.40	1.10	78.57	-81.8	N
269	0.46	0.09	19.57	-94.0	N
271	13.12	3.504	26.71	70.8	W
273	11.50	1.90	16.52	49.7	A
274	1.85	0.19	10.27	-75.9	N
276	4.76	0.20	4.26	-38.0	A
277	6.70	0.73	10.90	-12.8	

A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
278	7.20	1.6	22.22	-6.2	A
281	6.36	0.21	3.30	-17.2	A
282	3.48	0.51	14.66	-54.7	W
283	4.99	0.41	8.22	-35.0	A
284	0.82	0.52	63.81	-89.3	N
285	0.19	0.01	4.32	-97.6	N
286	5.30	0.61	11.51	-31.0	A
287	23.60	1.30	5.51	207.3	N
288	9.89	1.48	14.96	28.8	A
290	16.50	1.20	7.27	114.8	N
291	5.18	0.07	1.35	-32.6	A
293	3.59	0.26	7.24	-53.3	W
294	6.57	0.14	2.13	-14.5	A
297	29.10	2.91	10.00	278.9	N
298	14.60	2.2	15.07	90.1	N
299	7.57	0.76	10.04	-1.4	A
300	3.3	0.16	4.85	-57.0	W

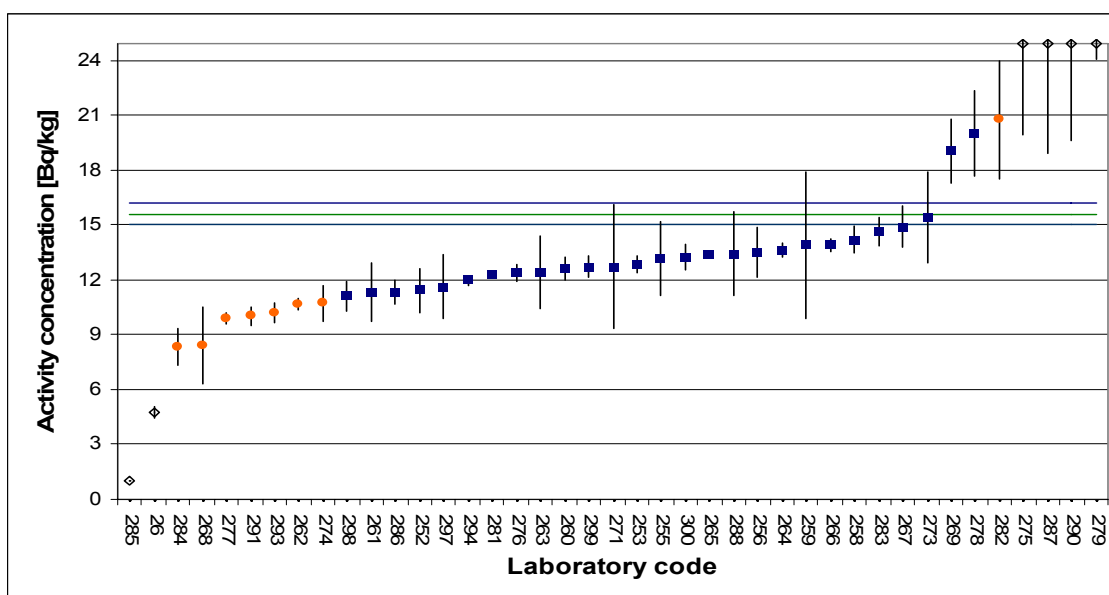
**Performance evaluation of gross beta measurement results
Water sample 3 (Blank water)**



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	F	Final Score
26	0.11	0.03	27.27	0.05	A
252	0.18	0.018	9.94	0.15	A
253	0.33	0.046	13.86	0.24	W
255	0.21	0.05	24.76	0.10	A
256	1.98	0.41	20.71	1.16	N
258	0.38	0.03	6.84	0.33	N
260	0.44	0.09	20.45	0.26	W
262	0.16	0.05	32.72	0.06	A
263	0.19	0.02	7.89	0.16	A
264	0.15	0.01	-	0.13	A
265	0.14	0.013	9.35	0.11	A
266	0.12	0.00	1.67	0.12	A
267	0.10	0.03	30.00	0.04	A
268	0.00	2.00	-	-4.00	A
269	0.12	0.01	10.92	0.09	A
271	0.14	0.109	80.15	-0.08	A
273	2.11	0.42	19.91	1.27	N
274	0.19	0.03	14.97	0.13	A
275	14.00	5	35.71	4.00	N
276	0.25	0.02	-	0.21	A
277	0.10	0.06	58.00	-0.02	A
278	0.28	0.15	53.57	-0.02	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	F	Final Score
279	4251.74	0.82	0.02	4250.10	N
281	0.10	0.03	30.00	0.04	A
282	0.94	0.18	19.15	0.58	N
283	0.03	0.09	300.00	-0.15	A
284	<2.820	0.00	-	-	A
285	0.10	0.01	7.84	0.09	A
286	0.13	0.10	71.97	-0.06	A
287	1.70	0.10	5.88	1.50	N
288	0.29	0.12	41.38	0.05	A
290	334.40	5.30	1.58	323.80	N
291	0.21	0.02	9.52	0.17	A
293	0.30	0.15	50.00	0.00	A
294	0.31	0.09	29.97	0.12	A
297	0.20	1.10	550.00	-2.00	A
298	2.60	0.7	26.92	1.20	N
299	0.19	0.03	15.79	0.13	A

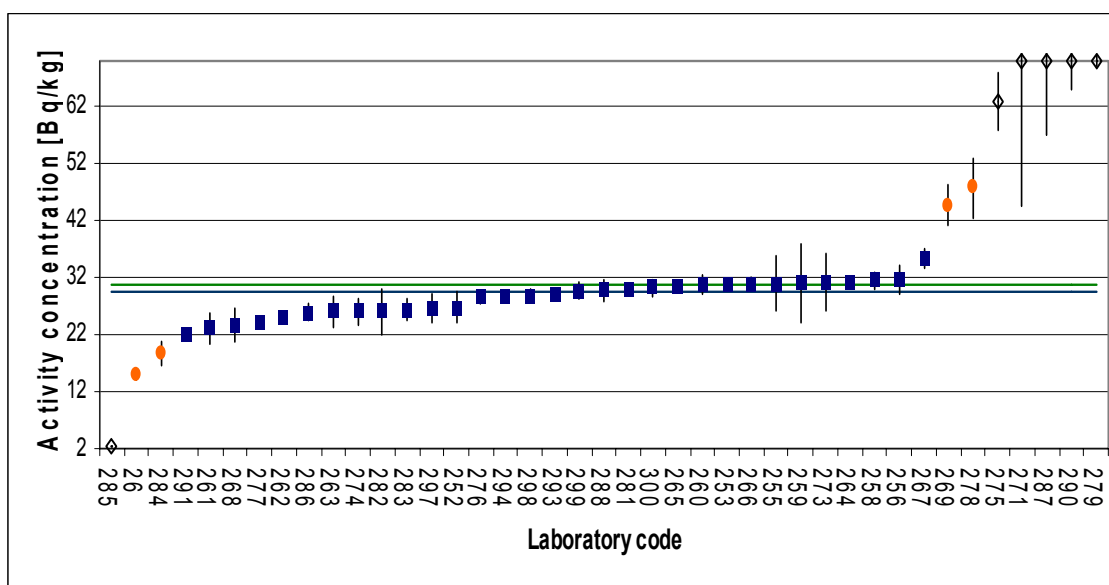
Performance evaluation of gross beta measurement results
Spiked water sample 4
Target Value: 15.7 ± 0.3 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
26	4.87	0.3	6.16	-69.0	N
252	11.50	1.2	10.43	-26.8	A
253	12.95	0.45	3.47	-17.5	A
255	13.26	2.02	15.20	-15.5	A
256	13.60	1.40	10.29	-13.4	A
258	14.30	0.74	5.17	-8.9	A
259	14.00	4.00	28.57	-10.8	A
260	12.70	0.60	4.72	-19.1	A
261	11.40	1.60	14.04	-27.4	A
262	10.75	0.28	2.62	-31.5	W
263	12.50	2.00	16.00	-20.4	A
264	13.72	0.39	2.87	-12.6	A
265	13.50	0.06	0.44	-14.0	A
266	14.00	0.33	2.32	-10.8	A
267	15.00	1.10	7.33	-4.5	A
268	8.50	2.10	24.71	-45.9	W
269	19.13	1.72	8.99	21.8	A
271	12.82	3.413	26.62	-18.3	A
273	15.50	2.50	16.13	-1.3	A
274	10.80	1.00	9.26	-31.2	W
275	36.00	5	13.89	129.3	N
276	12.45	0.46	3.72	-20.7	A

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
277	10.00	0.30	3.00	-36.3	W
278	20.10	2.3	11.44	28.0	A
279	5664.41	0.82	0.01	35979.0	N
281	12.38	0.16	1.29	-21.1	A
282	20.84	3.21	15.40	32.7	W
283	14.72	0.79	5.37	-6.2	A
284	8.41	1.01	11.95	-46.4	W
285	1.11	0.01	1.26	-92.9	N
286	11.41	0.65	5.70	-27.3	A
287	113.00	6.00	5.31	619.7	N
288	13.50	2.3	17.04	-14.0	A
290	331.90	5.30	1.60	2014.0	N
291	10.10	0.50	4.95	-35.7	W
293	10.30	0.55	5.34	-34.4	W
294	12.06	0.26	2.16	-23.2	A
297	11.70	1.75	14.96	-25.5	A
298	11.20	0.8	7.14	-28.7	A
299	12.80	0.57	4.48	-18.5	A
300	13.3	0.7	5.26	-15.3	A

Performance evaluation of gross beta measurement results
Spiked water sample 5
Target Value: 30.7 ± 0.6 [Bq/kg]



Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
26	14.89	0.7	4.70	-51.5	W
252	26.80	2.7	10.07	-12.7	A
253	30.82	0.9	2.92	0.4	A
255	30.94	4.69	15.14	0.8	A
256	31.60	2.40	7.59	2.9	A
258	31.42	1.60	5.09	2.3	A
259	31.00	7.00	22.58	1.0	A
260	30.80	1.50	4.87	0.3	A
261	23.10	2.70	11.69	-24.8	A
262	25.10	0.49	1.94	-18.2	A
263	26.00	2.80	10.77	-15.3	A
264	31.13	0.84	2.70	1.4	A
265	30.50	0.09	0.30	-0.7	A
266	30.90	0.95	3.08	0.7	A
267	35.30	1.60	4.53	15.0	A
268	23.80	3.00	12.61	-22.5	A
269	44.70	3.40	7.61	45.6	W
271	97.53	25.345	25.99	217.7	N
273	31.10	5.10	16.40	1.3	A
274	26.00	2.30	8.85	-15.3	A
275	63.00	5	7.94	105.2	N

Lab code	Rep. Value	Rep. Unc.	Unc. [%]	Rel. Bias	Final Score
276	28.61	1.03	3.60	-6.8	A
277	24.00	0.50	2.08	-21.8	A
278	47.70	5.3	11.11	55.4	W
279	139352	0.82	0.00	453816.1	N
281	30.06	0.25	0.83	-2.1	A
282	26.04	3.98	15.28	-15.2	A
283	26.31	1.99	7.56	-14.3	A
284	18.72	2.02	10.81	-39.0	W
285	2.38	0.09	3.90	-92.2	N
286	25.90	1.40	5.41	-15.6	A
287	250.00	13.00	5.20	714.3	N
288	29.80	1.79	6.01	-2.9	A
290	328.50	5.20	1.58	970.0	N
291	22.10	0.70	3.17	-28.0	A
293	29.07	0.84	2.89	-5.3	A
294	28.70	0.56	1.95	-6.5	A
297	26.50	2.50	9.43	-13.7	A
298	28.80	1.1	3.82	-6.2	A
299	29.70	1.32	4.44	-3.3	A
300	30.3	1.5	4.95	-1.3	A

APPENDIX III
LIST OF PARTICIPATING LABORATORIES¹

CODE	LABORATORY
26	CENTRAL RADIOLOGICAL LABORATORY, HUNGARIAN AGRICULTURAL AUTHORITY, FOGOLY U. 13 - 15 P.O. BOX 1740 H-1182, BUDAPEST, 94 HUNGARY
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