IAEA Analytical Quality in Nuclear Applications Series No. 4

# ALMERA Proficiency Test on the Determination of Po-210 in Water

IAEA-CU-2007-09



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INTERNATIONAL ATOMIC ENERGY AGENCY VIENNA, 2009

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For further information on this publication, please contact:

Chemistry Unit, Agency's Laboratories, Seibersdorf International Atomic Energy Agency 2444 Seibersdorf Austria

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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 worldwide 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.

A primary requirement of the ALMERA members is participation in the IAEA interlaboratory comparisons which are specifically organized for ALMERA on a regular basis. These exercises are designed to monitor and demonstrate the performance and analytical capabilities of the network members, and to identify gaps and problem areas where further development is needed. Continued membership 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 interlaboratory comparisons performed in the frame of the ALMERA network are not anonymous for those laboratories nominated to participate as ALMERA members.

The Po-210 poisoning event which occurred in November 2006 brought into focus a number of issues, including the capacity of laboratories to rapidly and accurately determine this radionuclide in environmental samples. A number of requests were received from ALMERA members to address this issue. Responding to these requests, the Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the IAEA's Seibersdorf Laboratory in Austria, conducted a proficiency test in the frame of the ALMERA network on the determination of Po-210 in water. The aim was to gather information on the current state of practice for Po-210 measurements at various levels in aqueous samples. This report describes the methodology employed and the results obtained in this proficiency test.

The IAEA wishes to thank the participating laboratories to this intercomparison exercise and all the contributors to drafting and review of this report. The IAEA officer responsible for this publication was A. Shakhashiro of the Agency's Laboratories, Seibersdorf.

# EDITORIAL NOTE

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#### 1. INTRODUCTION

The Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the International Atomic Energy Agency's Seibersdorf Laboratory in Austria, has the programmatic responsibility to support global radionuclide measurement systems, in issues of international concern related to an accidental or intentional release of radioactivity in the environment. To fulfil this obligation and ensure a reliable worldwide, rapid and consistent response, the Chemistry Unit coordinates an international network of Analytical Laboratories for the Measurement of Environmental **RA**dioactivity (ALMERA).

The ALMERA network, established by the IAEA in 1995 [1, 2, 3] is a technical collaboration of existing institutions and makes available to Member States a worldwide 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. 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. ALMERA currently (June 2008) consists of 110 laboratories representing 68 countries. The Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the IAEA's Seibersdorf Laboratory in Austria is the central coordinator of the ALMERA network's activities and the IAEA Marine Environment Laboratory in Monaco is also a member of the network.

The IAEA helps the ALMERA network of laboratories to maintain their readiness by coordination activities, by development of standardized methods for sample collection and analysis, and by conducting interlaboratory comparison exercises and proficiency tests as a tool for external quality control. These exercises are designed to monitor and demonstrate the performance and analytical capabilities of the network members, and to identify gaps and problem areas where further development is needed. Continued membership 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 interlaboratory comparison exercises performed in the frame of the ALMERA network are not anonymous for those laboratories nominating to participate as ALMERA members.

The Po-210 poisoning event which occurred in November 2006 [4] brought into focus a number of issues, including the capacity of laboratories to rapidly and accurately determine this radionuclide in environmental samples. To this end, the Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the IAEA's Seibersdorf Laboratory in Austria, conducted an interlaboratory comparison exercise in the frame of the ALMERA network on the determination of Po-210 in water. The aim was to gather information on the current state of practice for Po-210 measurements at various levels in aqueous samples.

This report describes the methodology employed and the results obtained in this proficiency test. In all 180 test water samples were prepared and distributed to the participating laboratories during the last week of March 2007. Laboratories were sent five water samples containing known (to the organizer) activities of Po-210, and were requested to return the results within one week of receipt of the samples (short-term reporting).

The participating laboratories were requested to analyse the samples employing the methods used in their routine work, so that their performance on the test samples could be directly related to the real performance of the rapid reporting time.

Thirty three of the thirty six initially registered laboratories reported their results to the IAEA. Table 1 shows the assigned code to each participating laboratory. 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 list of participants is reported in Appendix IV.

Figure 1 and Table 2 shows a summary evaluation of the proficiency test results. The proficiency test results demonstrated that 24 of the 33 participants were able to report results which fit the purpose of rapid detection of Po-210 in water.



FIG. 1. The result of the overall performance evaluation of the participating ALMERA laboratories in Po-210 determination.

Lab. Code	Name	Country
4	CIEMAT	Spain
12	Risoe National Laboratory	Denmark
14	FTU/FZK Research Center Karlsruhe	Germany
16	Office of Atomic Energy for Peace	Thailand
21	Malaysian Nuclear Agency (Nuclear Malaysia)	Malaysia
22	Tarapur Atomic Power Station	India
23	Atomic Energy Commission of Syria (AECS)	Syrian Arab Republic
30	Central Laboratory For Radiological Protection	Poland
31	Korea Atomic Energy Research Institute	Korea, Republic of
35	Instituto Tecnologico e Nuclear	Portugal
38	Veterinary Laboratories Agency	United Kingdom of Great Britain and Northern Ireland
39	SIA "Radon"	Russian Federation
46	Institute of Atomic Energy Research	Saudi Arabia
52	Belarussian State Institute of Metrology	Belarus
54	Australian Radiation Protection & Nuclear Safety Agency (ARPANSA)	Australia
57	Bhabha Atomic Research Centre	India
58	National Physical Laboratory (NPL)	United Kingdom of Great Britain and Northern Ireland
63	China Institute of Atomic Energy	China
64	Australian Nuclear Science and Technology Organization ANSTO	Australia
65	Radiation & Nuclear Safety Authority (STUK)	Finland
67	National Radiation Laboratory	New Zealand
70	Jordan Atomic Energy Commission	Jordan
71	APAT - Italian Environmental Protection Agency	Italy
72	National Institute of Public Health & Environment	Netherlands
73	ENEA – Istituto di Radioprotezione – Laboratorio Casaccia	Italy
74	ENEA – Istituto di Radioprotezione – Laboratorio Saluggia	Italy
83	University of California, Lawrence Livermore National Laboratory	United States of America
92	Institute Jozef Stefan	Slovenia
101	National Nuclear Energy Agency	Indonesia
106	South African Nuclear Energy Corporation (NECSA)	South Africa
119	NRG	Netherlands
124	Executive Environment Agency	Bulgaria
125	Brazilian National Commission for Nuclear Energy	Brazil

Lab. Code	Sample 01 (52.8±1.4 Bq kg <sup>-1</sup> )	Sample 02 (101.6±2.8 Bq kg <sup>-1</sup> )	Sample 03 (52.8±1.4 Bq kg <sup>-1</sup> )	Sample 04 (101.6±2.8 Bq kg <sup>-1</sup> )	Sample 05 (<0.1 Bq kg <sup>-1</sup> )
4	Α	W	W	W	Α
12	Α	Α	Α	Α	Α
14	Α	W	W	W	Α
16	W	Α	Α	Α	Α
21	Α	Α	Α	Α	Α
22	Ν	Ν	W	Ν	Α
23	Ν	Ν	N	Ν	Ν
30	Α	W	W	W	Ν
31	W	W	W	W	Α
35	Α	Α	Α	W	Α
38	Α	Α	Α	Α	Α
39	Α	Α	Α	Α	Α
46	Α	Α	Α	Α	Α
52	Ν	W	Α	Ν	Α
54	Ν	Ν	Ν	Ν	Α
57	Ν	W	Α	Α	Α
58	Α	Α	Α	Α	Α
63	Ν	Α	Α	W	Α
64	Α	Α	W	Α	Α
65	Α	Α	Α	Α	Α
67	Α	Α	Α	Α	Α
70	Α	Ν	Ν	Ν	Α
71	Α	Α	Α	Α	Α
72	Α	W	Α	Α	Α
73	Α	Α	Α	Α	Ν
74	Α	Α	Α	Α	Α
83	Α	Α	Α	Α	Α
92	Α	Α	Α	Α	Α
101	Ν	Α	Ν	Α	Ν
106	Α	Α	Α	Α	Α
119	Α	Α	Α	Α	Α
124	Α	W	Α	W	Α
125	W	Α	Α	W	Α

# TABLE 2. SUMMARY EVALUATION OF THE IAEA-CU-2007-9 ALMERA PROFICIENCY TEST

A = Acceptable, W = Warning, N = Not acceptable

# 2. MATERIALS AND METHODS

# **2.1. Proficiency test objectives**

Rapid measurement of spiked water, with an unknown (to the participants) amount of Po-210 was aimed at:

- checking the preparedness of ALMERA network laboratories for rapid determination of Po-210 in aqueous samples,
- evaluating the probability of reporting false positive and/or false negative evaluating the repeatability of the reported results,
- and encouraging the participating laboratories to implement remedial actions where shortcomings in analytical performance are detected.

# 2.2. Participants

Thirty six participants from thirty countries were registered in this PT, thirty three participants from twenty seven countries reported their results back to the IAEA.

The participating laboratories consisted of seventeen and eleven laboratories from Europe and Asia respectively, two laboratories from Australia and three laboratories from North America, Latin America and Africa.

# 2.3. Composition of the proficiency test materials

The set of the proficiency test materials consisted of five samples each  $\sim$ 50 mL. The following proficiency test design was applied:

- two spiked dematerialized water samples (sample codes 01, 03) ~50 g each containing ~2.5 Bq Po-210;
- two spiked dematerialized water samples (sample codes 02, 04) ~50 g each containing ~5 Bq Po-210;
- one blank dematerialized water (sample code 05). This is the same water which was used as raw material to spike the test materials.

Table 3 lists the target values and the associated combined standard uncertainty of the proficiency test materials and the proficiency test performance criteria LAP (Limit of Acceptable Precision) and MAB (Maximum Acceptable Bias) (see para 3.2).

# 2.4. Preparation of the spiked samples

The spiked water samples were gravimetrically prepared in two batches: one batch for samples 01 and 03 and one batch for samples 02 and 04. To prepare each batch 20 kg of acidified demineralised water was spiked with a certified single Po-210 solution traceable to the international standard of radioactivity. Then a pump with multiple outlets was used to homogenise the bulk water sample in a 50 L tank. The first batch was divided in two samples: 01 and 03, the second batch in samples 02 and 04.

Sample 05 was prepared from the same bulk water used in preparation of the spiked samples 01 to 04. This sample (blank) was used to check for the false positive reporting. Figure 2 shows an example of the test materials set.

The final target activity concentration for Po-210 was calculated from the certified activity value assigned to the certified standard solution of Po-210<sup>,</sup> taking into account the successive

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.

The initial activity concentration of the standard solution was  $377\pm10$  Bq kg<sup>-1</sup>.

The reference date for results reporting was set to the 1<sup>st</sup> of April 2007.

Three bottles from each batch were measured using liquid scintillation in the Agency's Seibersdorf Laboratories to verify the homogeneity and stability of the PT materials. The three bottles were stored at ambient temperature and measured four times in the period from 19 March to 7 may 2007. Measurement results are presented in Figure 3.

The variations of the obtained measurement results are comparable to the method reproducibility and therefore it can be concluded that there was not any significant uncertainty arising from between bottles heterogeneity or material instability.



FIG. 2. The proficiency test materials set.

TABLE 3. TARGET VALUES AND THE ASSOCIATED COMBINED STANDARD UNCERTAINTIES OF THE PROFICIENCY TEST MATERIALS AND THE PROFICIENCY TEST PERFORMANCE CRITERIA (PARA 3.2). LAP = LIMIT OF ACCEPTABLE PRECISION. MAB = MAXIMUM ACCEPTABLE BIAS.

	Samples 01 and 03	Samples 02 and 04	Sample 05
Activity (Bq kg <sup>-1</sup> )	52.8±1.4	101.6±2.8	<0.1
LAP (%)	15	15	
MAB (%)	20	20	



FIG. 3. Homogeneity and stability test results, four sets of measurements, one set every two weeks. Reference date:  $1^{st}$  of April 2007.

#### 3. PERFORMANCE CRITERIA

Currently most laboratories produce test results accompanied, at best, with an indication of their repeatability only and provide no indication of their analytical uncertainty. However, testing laboratories intending to follow international best practice will need to quantify and report their measurement uncertainty. In particular, this is a requirement under international standard ISO/IEC 17025:2005 [5].

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 in 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 in the Agency's laboratories [6] takes into consideration the trueness and the precision of the reported data and it includes in the evaluation both the combined standard uncertainty associated with the target value of proficiency testing samples and the combined standard 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 the uncertainty statement given by the participating laboratories. 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: relative bias, z-score and IAEA/Laboratory result ratio are calculated as complementary information for the participating laboratories.

#### 3.1. Relative bias

The first stage in producing a score for a result  $Value_{Analyst}$  (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 Analyst's value and the IAEA value is calculated and expressed as a percentage:

Relative bias = 
$$\frac{Value_{Analyst} - Value_{IAEA}}{Value_{IAEA}} \times 100\%$$

#### 3.2. Proficiency test evaluation criteria

The proficiency test results were evaluated against the acceptance criteria for trueness and precision and assigned the status "Acceptable", "Warning" or "Not Acceptable" accordingly [6].

#### 3.2.1. Trueness

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

$$A1 \le A2$$

where:

$$A1 = |Value_{IAEA} - Value_{Analyst}|$$
$$A2 = 2.58 \times \sqrt{Unc_{IAEA}^{2} + Unc_{Analyst}^{2}}$$

#### 3.2.2. Blank evaluation

The results of the blank (sample 05) were evaluated to check if a false positive was reported using the following rule: if the reported result fulfils the following criteria it was considered acceptable:

$$|Value_{analyst} - Unc_{analyst}| < 0.1$$

Also if the laboratory reported the MDL (Method Detection Limit) as a result (a value with a sign <) it was considered acceptable. Otherwise, the reported value was not acceptable.

#### 3.3.The z-score value

The z-score is calculated from the laboratory results, the assigned value and a standard deviation in accordance with the following equation:

$$z_{Score} = \frac{Value_{Analyst} - Value_{IAEA}}{\sigma}$$

On the basis of the "fitness for purpose" principle, the target value for the standard deviation  $(\sigma)$  is:

The laboratory performance is evaluated as satisfactory if  $|z|_{Score} | \le 2$ ; questionable for  $2 < |z|_{Score} | <3$ , and unsatisfactory for  $|z|_{Score} | \ge 3$ .

#### 3.4. The u-score value

The value of the  $u_{\text{test}}$  was calculated according to the following equation [7]

$$u_{test} = \frac{\left| Value_{IAEA} - Value_{Analyst} \right|}{\sqrt{Unc_{IAEA}^{2} + Unc_{Analyst}^{2}}}$$

This value is compared with the critical value listed in the t-statistic tables to determine if the reported result differs significantly from the expected value at a given level of probability. The advantage of the  $u_{test}$  is that it takes into consideration the propagation of measurement uncertainties when defining the normalized error. This is especially useful when evaluating results, which uncertainty may overlap with the reference interval.

It should be noted that the choice of the significance level is subjective. For this proficiency test we have set the limiting value for the u-test parameter to 2.58 for a confidence level of 99% to determine if a result passes the test (u < 2.58).

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 fit specific requirements.

#### 4. **RESULTS AND DISCUSSION**

## 4.1. General

There were 155 measurement results reported to the IAEA in this proficiency test from 33 laboratories. The participants' data along with the statistical performance evaluation were compiled and presented in two tables which constitute an integral part of this report. Appendix I shows the data evaluation tables sorted by sample code. Performance evaluation tables sorted by laboratory code are reported in Appendix II.

The overall evaluation showed that 70% of all reported results fulfilled the PT criteria for both trueness and precision. Despite the fact that the matrix was easy and there was not any interference effect, 15% of all reported results were not acceptable against the PT criteria.

#### 4.2. Technical information provided by the participants

The technical information provided by the participants on the analytical procedures used in their own laboratories is compiled in Appendix III and coded with the same laboratory code used in data evaluation. The participants can benefit from the information exchange without revealing the laboratories' identity.

The provided technical information was compiled in the same format as it was received, without any modification or editing.

Most of the participants did not use any separation method due to the nature of the matrix. For source preparation all of the participants used auto deposition method on silver or stainless steel disk, only one participant (Lab 119) used electroplating.

Table 4 and Table 5 present a summary of the technical information applied by the participants related to the analytical procedure and estimation of the Minimum Detection Limit (MDL) respectively.

The reported technical parameters applied in the analysis of the samples are graphically presented in Figures 4 and 5. The counting time, sample mass, gross counts and reporting time is presented in Figure 4. The tracer recovery, counting efficiency and elapsed years of the Po-209 from the reference date are presented in Figure 5.

From the technical details of the analytical procedure provided by the participants who had low performance score, it was not possible to find any indication of a methodological error or problem. There was no substantial difference in the described procedures to which the root cause of discrepancy could be attributed. For instance, all of the participants indicated that they applied temperatures below 90° C while heating the solution during the plating.

#### 4.3. False positive reporting

In this proficiency test one of the analysed samples was the "blank material" which was used to prepare the spiked samples. In order to evaluate the results of the blank sample 05, the analysts were asked to report information on their Method Detection Limit (MDL).

From the provided information, it can be observed that there is no harmonised procedure for MDL estimation amongst the ALMERA laboratories which could lead to inappropriate

comparison of MDL estimated in different laboratories. The summary of the reported MDL and the used procedure to derive it are shown in Table 4

It was found that 4 laboratories (23, 30, 73 and 101) reported false positive for the blank sample 05, these 4 laboratories did not report any information on the method validation which could mean that MDL was not yet estimated, or the method validation for such a matrix was not yet performed.

# 4.4. Measurement repeatability

The PT samples contained duplicate samples 01, 03 and 02, 04. The variation between the results of the duplicate samples was checked. 17 laboratories of 31 had a difference between the duplicate samples more than 5%, which might indicate the need for improving the method stability. The method statistical control and repeatability should be controlled and monitored to ensure the method capability to detect low activity concentrations with high reliability.

Figures 6 and 7 show the graphical presentation of the variations between the duplicate samples. Laboratories 04, 12, 14, 16, 21, 23, 38, 46, 52, 57, 63, 64, 70, 71, 83, 92, 101, 119 and 125 reported the results of duplicate samples 01, 03 and 02, 04 with variations of more than 5% between duplicate samples.

## **4.5.** Evaluation of reporting time

The participants were asked to report the results within one week from the date of sample receipt. Nineteen laboratories reported within one week.

Figure 8 presents the laboratory average performance calculated based on the percentage of acceptable results against the number of working days between the receipt of the sample and submission of the results. It can be concluded from the graph that there is not any effect on the performance in rapid reporting. If the method is well established reliable results can be produced in short time. Some laboratories took more than 20 days but still reporting not acceptable results.

On Figure 8 the total number of elapsed days between the date of sample receipt and the date of results submission is also presented. This gives an idea about the time needed for the whole process from sample dispatch to results reporting. For most of the laboratories it took approximately 10 days, for some of participants it took up to 35 days depending on the destination.

#### 4.6. Recommendations to the laboratories

Based on the performance evaluation results the recommendations to the participants could be divided into four categories:

• Twenty three laboratories namely: 04, 12, 14, 16, 21, 35, 38, 39, 46, 58, 63, 64, 65, 67, 71, 72, 74, 83, 92, 106, 119, 124 and 125 were able to report results with a quality that fits for the purpose of rapid responding in emergency situation to trigger an alarm for remediation or any other decision for an action to be taken. However more efforts should be invested on method validation to determine the method performance characteristics in the laboratory's local conditions and to demonstrate that the targeted quality criteria of the analytical procedure are attained.

- Nineteen laboratories mentioned in 4.4 should improve the repeatability and the reproducibility of their determinations and to find out the source of variations, it could be attributed to the plating process, to inappropriate recovery correction or to other technical issues. Replicate analysis of spiked samples should be used to optimise the method and to reduce the source of variations. Target repeatability and reproducibility standard deviations should be set up by the analyst and to work on the method to attain these targets.
- Four laboratories mentioned in 4.3 reported false positive or a value for the blank sample higher than the target value. These laboratories should evaluate the analytical procedure blank and to subtract it from the sample value. Eurachem Guide on method validation suggests some guidelines on MDL determination. Many participants reported in Table 4 the procedure they applied in the estimation of MDL.
- Five laboratories (22, 23, 54, 70 and 101) could not report acceptable results and in few cases the method was not stable and could not differentiate between high and low activities or even report a large value for the blank sample. These laboratories should revise their method and look for the root cause of instability and perform method validation to check the reliability of the reported results.

# 5. CONCLUSIONS

The IAEA-CU-2007-09 ALMERA proficiency test determination of Po-210 in water was successfully conducted, 36 participants received the proficiency test samples, and 91% of the participants reported back their results to the IAEA which indicates a high rate of results reporting in this PT.

The PT results demonstrated that 22 of 33 participants were able to report results which fit the purpose of rapid detection of Po-210 in water.

However, although the matrix was a relatively straightforward one and the activity concentrations were relatively high, 15% of the reported results failed to pass the proficiency test criteria. In a few cases positive results were reported for the blank sample which suggests a possibility of false positive reporting.

The proficiency test organizer gave general recommendations to a group of laboratories to improve their analytical performance. However, if any participant needs any technical assistance to improve the analytical performance of Po-210 determination, the Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the International Atomic Energy Agency's Seibersdorf Laboratory in Austria, will be glad to respond to such requests.

The proficiency test results reveal the need for a harmonized analytical procedure for Po-210 rapid determination in case of emergency for high and low levels of activities since published analytical methods remain remarkably diverse [8]. The procedure should also contain a standardized quality control protocol to assist the analyst in the validation of the reported results.

This proficiency test provided the possibility to quantify the level of analytical performance of the ALMERA network members, and consequently should help the network members to improve their performance in the determination of Po-210 in water.

Lab. code	Sample preparation	Separation method	Source preparation	Measurement technique	Method validation
4	Evaporation	Auto deposition	Auto deposition	α-spec	yes
12	Evaporation	NR	Auto deposition	α-spec	yes
14	Evaporation	No separation	Auto deposition	α-spec	yes
16	Evaporation	No separation	Auto deposition	α-spec	no
21	Evaporation	No separation	Auto deposition	α-spec	no
22	Evaporation	NR	Auto deposition	α-spec	yes
23	NR	NR	NR	NR	NR
30	NR	Auto deposition	Auto deposition	α-spec	no
31	NR	No separation	Auto deposition	α-spec	yes
35	Evaporation	No separation	Auto deposition	α-spec	no
38	NR	Extraction chromatography	Auto deposition	α-spec	yes
39	NR	NR	Auto deposition	α-spec	yes
46	Deposition	Auto deposition	Auto deposition	α-spec	yes
52	Coprecepitation	NR	Auto deposition	α-spec	no
54	Evaporation	No separation	Auto deposition	α-spec	no
57	Evaporation	NR	Auto deposition	Gross $\alpha$ counter	yes
58	NR	NR	NR	NR	no
63	Evaporation	NR	Auto deposition	a-spec	yes
64	Evaporation	NR	Auto deposition	a-spec	no
65	NR	NR	Auto deposition	a-spec	yes
67	Evaporation	No separation	Auto deposition	α-spec	yes
70	Evaporation	Extraction chromatography	Auto deposition	α-spec	no
71	Coprecepitation	Auto deposition	Auto deposition	α-spec	yes
72	NR	No separation	Auto deposition	α-spec	yes
73	NR	No separation	Auto deposition	α-spec	yes
74	Dilution	No separation	Auto deposition	α-spec	no
83	Chelation	No separation	Auto deposition	α-spec	no
92	Auto deposition	No separation	Auto deposition	α-spec	no
101	NR	NR	Auto deposition	α-spec	no
106	Dilution	No separation	Auto deposition	a-spec	yes
119	Evaporation	No separation	Electro deposition	a-spec	no
124	Coprecepitation	No separation	NR	a-spec	no
125	Evaporation	NR	Auto deposition	α-spec	no
ND	4 1				

TABLE 4. SUMMARY INFORMATION ON THE ANALYTICAL PROCEDURE USED BY THE PARTICIPANTS

NR: not reported

Lab. code	Procedure for the estimation of MDL	Claimed MDL	Reported MDL	Sample 05 evaluation
4	Detection Limit calculated by ISO-11929 standard. LID= $0.013 \text{ Bq kg}^{-1}$ (99% Confidence level)	0.013	0.008	А
12	All 5 samples analysed first based on 20 mL aliquots with MDL's below 0.1 Bq kg <sup>-1</sup> . Analyses of samples 1-4 repeated based on 2-3 mL aliquots to match spike amounts.	0.1	0.03	А
14	LLD: 0.04 Bq kg <sup>-1</sup> ( <sup>210</sup> Po)	0.04	0.011	А
16	NR		0.0737	А
21	NR		0	А
22	Spectrometric system is checked by counting tracer( <sup>242</sup> Pu) for MDL level concentration. Background count rate for reproducibility		0.042	А
23	NR		27	Ν
30	NR		0.2	Ν
31	Minimum detection limit was calculated by L. A. Currie's equation. The MDL is 0.07 Bq kg <sup>-1</sup> . Repeatability was not tested. Each sample was counted once by alpha spectrometer. Reproducibility was tested. The identical sample was measured three times. The RSD of results is less than 1.5%.	0.07	<0.07	A
35	NR		0.0092	А
38	MDL is dependant on sample mass. For low k level work where 100g of sample is taken the MDL is 0.005 Bq kg <sup>-1</sup> For these IAEA samples where the activity was very high 4 g of sample was used to achieve an MDL around 0.1 Bq kg <sup>-1</sup> Repeatability at $k=2$ is 4.2%	0.1	<0.0453	A
39	For this PT MDL=0.15 Bq kg <sup>-1</sup> at Counting Time 25200s, sample Mass 10.4g, Counting Efficiency 37.2%, Recovery 96% (for low salinity waters)	0.15	0.041	А
46	LLD=0.00481 Bq L <sup>-1</sup> , Triplicate samples analysed. Above 95%.	0.005	0	А
52	NR		0.1	А
54	Method validation for polonium in solid, water and urine samples is in progress.		<0.2	А
57	Minimum Detection Limit for Sample Code 1 to 4 - 0.2 Bq kg <sup>-1</sup> (3sigma)Repeatability - +/- 3%Reproducibility - +/- 4%	0.2	<0.15	А
58	NR		0	A
65	MDA= $0.01$ Bq kg <sup>-1</sup> , repeatability = 6%	0.01	0.008	А
67	MDC 0.1 Bq kg <sup>-1</sup> for 5g sample and 23 hour counting time.	0.1	< 0.073	А

# TABLE 5. SUMMARY INFORMATION AS REPORTED BY THE PARTICIPANTS ON THE MINIMUM DETECTION LIMIT (MDL)

A = Acceptable, W = Warning, N = Not acceptable, NR = not reported

Lab code	Procedure for the estimation of MDL	Claimed MDL	Reported MDL	Sample 05 evaluation
70	NR		0.0026	А
71	Genie 2000 software's validated by CANBERRA. The method validation was performed by analysing IAEA- 326 Soil and IAEA-315 Sediment. The obtained data were all in good agreement with the recommended values. The obtained precision (relative standard deviations) is < 10% and the accuracy (relative bias) is < 2%. The minimum detection limit for 5 kg of water sample is 0.016 mBq kg <sup>-1</sup> and the corresponding value for 2.5 g of water 32 mBq kg <sup>-1</sup> .	0.02	0.07	A
72	In 2004 and 2005 the Dutch norm NEN 5694 (Methods for radiochemical determination of <sup>210</sup> Po and <sup>210</sup> Pb) was validated in various matrices; e.g. biological, silica- containing and non-Si-containing samples. I could give the validation parameters for these matrices. But as they have nothing to do with Seibersdorf Deminineralized water there is no sense in giving those data. Furthermore, the minimum detection limit highly depends on the processed sample volume. Again there is no sense in giving a detection limit as such. A water sample is much simpler than a silica -containing sample with a strongly oxidizing chemical treatment.		0.1	A
73	The method validation was performed on urine samples of 500 ml. Considering a counting time of 200000s the validation parameters were: Minimum detection limit: 5 mBq/l. Repeatability limit: 5%Reproducibility limit = 9%	0.005	0.19	N
74	NR		0.13	А
83	NR		0.0032	А
92	NR		0.019	А
101	NR		2.446	Ν
106	MDA=0.04 Bq kg <sup>-1</sup> where efficiency=0.2; yield=0.6; sample mass=25g, counting time=86400s.Accuracy: 15%Precision: 10% (12 samples on day of analyses)Reproducibility: 13% (more than 50 test samples analysed over a year as test control samples)	0.04	0.4	А
119	NR		< 0.0045	Α
124	NR		0.02	A
125	NR		0.018	A

A = Acceptable, W = Warning, N = Not acceptable, NR = not reported



FIG. 4. Technical information reported by the participants for samples 01. The net working days are the elapsed days from the date of sample reception to the date of results reporting. Counting time unit is second, sample mass unit is gram.



FIG. 5. Technical information reported by the participants for samples 03. The net working days are the elapsed days from the date of sample reception to the date of results reporting. Counting time unit is second, sample mass unit is gram.





FIGS. 6. and 7. Variations in % between the reported results for the duplicate samples.



FIG. 8. The laboratory average performance against the number of net working days needed to report the results and the total number of days elapsed between the sample dispatch and results submission.

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## APPENDIX I. DATA EVALUATION TABLES SORTED BY SAMPLE CODE

All participants reported values (Rep. Value) and uncertainties (Rep. Unc.) in this Appendix are expressed in Bq.kg<sup>-1</sup>. The abbreviations and calculation formulas used in the evaluation tables are explained in paragraph 3 of this report.

On the S-shape charts the IAEA target value is represented by a red line, and the respective combined standard uncertainty [u] is represented by two green lines. On the z-score charts warning limits are represented by blue lines, action limits by red lines.

The reference date for the IAEA target values and participants reported values has been set to 2007-April-01.

In this Appendix, laboratories data is presented in ascending order of the laboratory code.

# Data evaluation of sample 01







FIG. I-02: z-score chart of sample 01.

Lab code	Rep. Value	Reported Unc.	Unc. [%]	A1	A2	Trueness	Р	Precision	Final Score
4	48.80	2.00	4.10	4.00	6.30	А	4.88	А	А
12	50.50	5.60	11.09	2.30	14.89	А	11.40	А	А
14	51.00	1.80	3.53	1.80	5.88	А	4.41	А	А
16	58.92	1.42	2.41	6.12	5.14	Ν	3.58	А	W
21	49.41	3.01	6.09	3.39	8.56	А	6.64	А	А
22	40.98	2.98	7.27	11.82	8.49	Ν	7.74	А	Ν
23	102.00	10.00	9.80	49.20	26.05	Ν	10.16	А	Ν
30	58.15	1.65	2.84	5.35	5.58	А	3.88	А	А
31	48.12	0.88	1.83	4.68	4.27	N	3.22	А	W
35	47.90	1.70	3.55	4.90	5.68	А	4.43	А	А
38	45.90	3.20	6.97	6.90	9.01	А	7.46	А	А
39	55.00	3.00	5.45	2.20	8.54	А	6.06	А	А
46	62.35	3.57	5.73	9.55	9.90	А	6.31	А	А
52	34.60	1.42	4.10	18.20	5.14	N	4.89	А	Ν
54	71.20	4.90	6.88	18.40	13.15	N	7.38	А	Ν
57	65.60	3.00	4.57	12.80	8.54	N	5.29	А	Ν
58	51.70	0.40	0.77	1.10	3.76	А	2.76	А	А
63	39.50	4.42	11.19	13.3	11.96	Ν	11.50	А	Ν
64	49.97	2.12	4.24	2.83	6.55	А	5.00	А	А
65	46.20	3.00	6.49	6.60	8.54	А	7.01	А	А
67	50.00	1.70	3.40	2.80	5.68	А	4.31	А	А
70	59.00	6.60	11.19	6.20	17.41	А	11.50	А	А
71	47.37	3.32	7.01	5.43	9.30	А	7.49	А	А
72	47.50	1.90	4.00	5.30	6.09	А	4.80	А	А
73	50.60	2.70	5.34	2.20	7.85	А	5.96	А	А
74	48.00	3.00	6.25	4.80	8.54	А	6.79	А	А
83	50.80	2.90	5.71	2.00	8.31	А	6.29	А	А
92	49.30	3.80	7.71	3.50	10.45	А	8.15	А	А
101	115.42	9.54	8.26	62.62	24.87	N	8.68	А	N
106	57.70	6.40	11.09	4.90	16.90	А	11.40	А	А
119	49.00	2.00	4.08	3.80	6.30	А	4.87	А	А
124	49.36	1.05	2.13	3.44	4.52	А	3.40	А	А
125	46.70	0.20	0.43	6.10	3.65	N	2.69	А	W

# TABLE I.1. DATA EVALUATION OF SAMPLE 01

# Data evaluation of sample 02







FIG. I-04: z-score chart of sample 02.

# **Data evaluation of sample 02** Target Value: 101.6 ± 2.8 Bq/kg

Lab code	Rep. Value	Reported Unc.	Unc. [%]	A1	A2	Trueness	Р	Precision	Final Score
4	81.60	3.30	4.04	20.00	11.17	N	4.89	А	W
12	84.40	9.40	11.14	17.20	25.31	А	11.47	А	А
14	83.90	2.80	3.34	17.70	10.22	Ν	4.33	А	W
16	102.01	2.56	2.51	0.41	9.79	А	3.73	А	А
21	101.15	6.16	6.09	0.45	17.46	А	6.68	А	А
22	80.55	5.78	7.18	21.05	16.57	Ν	7.69	А	Ν
23	148.00	12.00	8.11	46.40	31.79	Ν	8.56	А	Ν
30	116.50	2.66	2.28	14.90	9.96	Ν	3.58	А	W
31	89.92	1.32	1.47	11.68	7.99	Ν	3.12	А	W
35	93.60	3.20	3.42	8.00	10.97	А	4.39	А	А
38	90.80	6.20	6.83	10.80	17.55	А	7.36	А	А
39	101.20	5.20	5.14	0.40	15.24	А	5.83	А	А
46	113.80	6.49	5.70	12.20	18.23	А	6.33	А	А
52	86.50	3.50	4.05	15.10	11.56	Ν	4.90	А	W
54	132.00	8.30	6.29	30.40	22.60	N	6.87	А	Ν
57	116.10	2.50	2.15	14.50	9.68	Ν	3.50	А	W
58	99.60	0.50	0.50	2.00	7.34	А	2.80	А	А
63	82.90	7.53	9.08	18.7	20.73	А	9.49	А	А
64	94.59	3.69	3.90	7.01	11.95	А	4.78	А	А
65	95.20	6.20	6.51	6.40	17.55	А	7.07	А	А
67	95.80	3.10	3.24	5.80	10.78	А	4.25	А	А
70	53.90	5.82	10.80	47.70	16.66	Ν	11.14	А	Ν
71	90.93	6.37	7.01	10.67	17.95	А	7.53	А	А
72	88.00	3.00	3.41	13.60	10.59	Ν	4.38	А	W
73	90.60	4.70	5.19	11.00	14.11	А	5.87	А	А
74	95.00	5.00	5.26	6.60	14.78	А	5.94	А	А
83	101.70	5.70	5.60	0.10	16.38	А	6.25	А	А
92	99.90	4.70	4.70	1.70	14.11	А	5.45	А	А
101	89.70	6.38	7.11	11.89	17.96	А	7.62	А	А
106	103.30	11.60	11.23	1.70	30.79	А	11.56	А	А
119	93.00	4.00	4.30	8.60	12.60	А	5.11	А	А
124	89.62	2.82	3.15	11.98	10.25	N	4.18	А	W
125	102.60	0.60	0.58	1.00	7.39	А	2.82	А	А

# TABLE I.2. DATA EVALUATION OF SAMPLE 02






FIG. I-06: z-score chart of sample 03.

Target Value:  $52.8 \pm 1.4$  Bq/kg

TABLE I.3. DATA EVALUATION OF SAMPLE 03

Lab code	Rep. Value	Reported Unc.	Unc. [%]	A1	A2	Trueness	Р	Precision	Final Score
4	44.70	1.90	4.25	8.10	6.09	Ν	5.01	А	W
12	41.90	4.70	11.22	10.90	12.65	А	11.53	А	А
14	45.50	1.80	3.96	7.30	5.88	Ν	4.76	А	W
16	50.66	1.49	2.94	2.14	5.27	А	3.96	А	А
21	51.75	3.15	6.09	1.05	8.89	А	6.64	А	А
22	42.64	3.11	7.29	10.16	8.80	Ν	7.76	А	W
23	85.00	8.00	9.41	32.20	20.95	Ν	9.78	А	Ν
30	58.70	1.73	2.95	5.90	5.74	Ν	3.96	А	W
31	48.27	0.88	1.82	4.53	4.27	Ν	3.22	А	W
35	48.40	1.60	3.31	4.40	5.49	А	4.24	А	А
38	48.30	3.30	6.83	4.50	9.25	А	7.33	А	А
39	54.50	3.00	5.50	1.70	8.54	А	6.11	А	А
46	56.21	3.14	5.59	3.41	8.87	А	6.19	А	А
52	46.96	1.93	4.11	5.84	6.15	А	4.89	А	А
54	70.90	4.70	6.63	18.10	12.65	Ν	7.14	А	Ν
57	54.10	2.50	4.62	1.30	7.39	А	5.33	А	А
58	51.70	0.40	0.77	1.10	3.76	А	2.76	А	А
63	45.55	4.97	10.91	7.25	13.32	А	11.23	А	А
64	46.97	1.69	3.60	5.83	5.66	Ν	4.47	А	W
65	48.20	3.10	6.43	4.60	8.78	А	6.96	А	А
67	52.60	1.80	3.42	0.20	5.88	А	4.33	А	А
70	15.11	1.56	10.34	37.69	5.41	Ν	10.68	А	N
71	44.45	3.11	7.00	8.35	8.80	А	7.48	А	А
72	47.00	2.00	4.26	5.80	6.30	А	5.01	А	А
73	50.50	2.70	5.35	2.30	7.85	А	5.97	А	А
74	49.00	3.00	6.12	3.80	8.54	А	6.67	А	А
83	54.30	3.00	5.52	1.50	8.54	А	6.13	А	А
92	51.70	2.70	5.22	1.10	7.85	А	5.86	А	А
101	72.09	5.67	7.87	19.29	15.08	Ν	8.30	А	Ν
106	58.20	6.50	11.17	5.40	17.15	А	11.48	А	A
119	58.00	2.00	3.45	5.20	6.30	А	4.35	А	А
124	49.16	0.92	1.87	3.64	4.32	A	3.25	А	А
125	52.80	0.20	0.38	0.00	3.65	A	2.68	A	А



FIG. I-07: S-shape chart of sample 04.



FIG. I-08: z-score chart of sample 04.

# **Data evaluation of sample 04** Target Value: 101.6 ± 2.8 Bq/kg

Lab code	Rep. Value	Reported Unc.	Unc. [%]	A1	A2	Trueness	Р	Precision	Final Score
4	83.30	3.40	4.08	18.30	11.36	Ν	4.92	А	W
12	83.00	9.30	11.20	18.60	25.06	А	11.54	А	А
14	85.90	2.80	3.26	15.70	10.22	Ν	4.27	А	W
16	97.57	2.53	2.59	4.03	9.74	А	3.79	А	А
21	100.97	6.15	6.09	0.63	17.43	А	6.69	А	А
22	78.28	5.61	7.17	23.32	16.18	Ν	7.68	А	Ν
23	140.00	24.00	17.14	38.40	62.34	А	17.36	Ν	Ν
30	119.40	3.09	2.59	17.80	10.76	Ν	3.78	А	W
31	91.30	1.40	1.53	10.30	8.08	N	3.15	А	W
35	90.60	3.00	3.31	11.00	10.59	N	4.31	А	W
38	88.20	6.00	6.80	13.40	17.08	А	7.34	А	А
39	100.20	5.10	5.09	1.40	15.01	А	5.79	А	А
46	116.70	6.49	5.56	15.10	18.24	А	6.21	А	А
52	70.70	2.88	4.07	30.90	10.36	Ν	4.92	А	Ν
54	129.00	8.50	6.59	27.40	23.09	N	7.14	А	N
57	102.80	3.40	3.31	1.20	11.36	А	4.31	А	А
58	99.60	0.50	0.50	2.00	7.34	А	2.80	А	А
63	85.22	4.71	5.53	16.38	14.14	Ν	6.18	А	W
64	95.81	3.53	3.68	5.79	11.62	А	4.60	А	А
65	94.40	6.10	6.46	7.20	17.32	А	7.03	А	А
67	97.20	3.10	3.19	4.40	10.78	А	4.22	А	А
70	6.69	0.70	10.46	94.91	7.45	Ν	10.82	А	Ν
71	85.94	6.02	7.00	15.66	17.13	А	7.53	А	А
72	93.00	4.00	4.30	8.60	12.60	А	5.11	А	А
73	90.60	4.50	4.97	11.00	13.67	А	5.68	А	А
74	98.00	5.00	5.10	3.60	14.78	А	5.80	А	А
83	96.10	5.30	5.52	5.50	15.46	А	6.17	А	А
92	102.90	4.60	4.47	1.30	13.89	А	5.25	А	А
101	118.78	8.68	7.31	17.18	23.53	А	7.81	А	А
106	101.30	11.30	11.15	0.30	30.04	А	11.49	А	А
119	97.00	5.00	5.15	4.60	14.78	А	5.85	А	А
124	91.90	1.92	2.09	9.70	8.76	Ν	3.46	А	W
125	91.60	0.50	0.55	10.00	7.34	N	2.81	А	W

# TABLE I.4. DATA EVALUATION OF SAMPLE 04



FIG. I -09: Results of the "Blank" sample.

Lab code	Rep. Value	Reported Unc.	Final Score	Lab code	Rep. Value	Reported Unc.	Final Score
4	0.01	0.00	А	63	0.03	0.03	А
12	0.03	0.03	А	64	< 0.01		А
14	0.01	0.01	А	65	0.01	0.01	А
16	0.07	0.04	А	67	< 0.073		А
21	0.00	0.00	А	70	0.00	0.00	А
22	0.04	0.02	А	71	0.07	0.01	А
23	27.00	5.00	Ν	72	0.10		А
30	0.20	0.02	Ν	73	0.19	0.07	Ν
31	< 0.07		А	74	0.13	0.04	А
35	0.01	0.00	А	83	0.00	0.00	А
38	< 0.05		А	92	0.02	0.00	А
39	0.04	0.03	А	101	2.45	0.38	Ν
46	0.00	0.00	А	106	0.40	0.31	А
52	0.10	0.03	А	119	< 0.0045		А
54	<0.2		А	124	0.02	0.00	А
57	< 0.15		А	125	0.02	0.00	А
58	0.00	1.00	А				

Target value: <0.1 Bq/kg

Table I.5. Data evaluation of sample 05

#### APPENDIX II. DATA EVALUATION TABLES SORTED BY LABORATORY CODE

All participants reported values and uncertainties in this Appendix are expressed in Bq.kg<sup>-1</sup>. The abbreviations and calculation formulas used in the evaluation tables are explained in paragraph 3 of this report.

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

The reference date for the IAEA target values and participants reported values has been set to 2007-April-01.

÷	0.04												
Lab. L Value U	n N	ab. nc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	ď	Prec.	Final Score
48.80 2.0	2.0	0	4.10	-7.58	-0.76	-1.64	0.92	4.00	6.30	Α	4.88	Α	V
81.60 3.30	3.3(	C	4.04	-19.69	-1.97	-4.62	0.8	20.00	11.17	Z	4.89	Υ	8
44.70 1.9	1.9	0	4.25	-15.34	-1.53	-3.43	0.85	8.10	6.09	Z	5.01	Α	M
83.30 3.4	3.4	0	4.08	-18.01	-1.8	-4.15	0.82	18.30	11.36	Z	4.92	A	M
0.01 0.00	0.0(												A
0.12 Lab. I	Π	ab.	Unc.	Rel.	Z-	11 T.004	Datio	1	Ş	Ĕ	2	Duco	Final
Value U	Û	nc.	[%]	Bias	Score	159 T-N	Kauo	I	A2	1 rue	4	rrec.	Score
50.50 5.6	5.6	0	11.09	-4.36	-0.44	-0.4	0.96	2.30	14.89	Α	11.40	Α	V
84.40 9.4	9.4	0	11.14	-16.93	-1.69	-1.75	0.83	17.20	25.31	А	11.47	Α	A
41.90 4.7	4.7	0	11.22	-20.64	-2.06	-2.22	0.79	10.90	12.65	A	11.53	A	V
83.00 9.3(	9.3(		11.20	-18.31	-1.83	-1.92	0.82	18.60	25.06	A	11.54	A	A
0.03 0.03	0.03												A

DRAJ	FORY NC	). 14												
	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
	1.40	51.00	1.80	3.53	-3.41	-0.34	-0.79	0.97	1.80	5.88	Α	4.41	Α	A
	2.80	83.90	2.80	3.34	-17.42	-1.74	-4.47	0.83	17.70	10.22	Z	4.33	A	M
	1.40	45.50	1.80	3.96	-13.83	-1.38	-3.2	0.86	7.30	5.88	Z	4.76	Α	M
	2.80	85.90	2.80	3.26	-15.45	-1.55	-3.96	0.85	15.70	10.22	Z	4.27	V	M
	0.10	0.01	0.01											A
<b>4 t</b>	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	P	Prec.	Final Score
	1.40	58.92	1.42	2.41	11.59	1.16	3.07	1.12	6.12	5.14	Z	3.58	Α	W
_	2.80	102.0	2.56	2.51	0.41	0.04	0.11	1.0	0.41	9.79	Α	3.73	A	A
	1.40	50.66	1.49	2.94	-4.06	-0.41	-1.05	0.96	2.14	5.27	A	3.96	Α	A
	2.80	97.57	2.53	2.59	-3.97	-0.4	-1.07	0.96	4.03	9.74	Α	3.79	A	A
	0.10	0.07	0.04											A

35

<b>FABLE II.5</b> .	LABORA	TORY NC	<b>D</b> . 21												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	49.41	3.01	6.09	-6.42	-0.64	-1.02	0.94	3.39	8.56	Α	6.64	A	A
02	101.60	2.80	101.1 5	6.16	6.09	-0.44	-0.04	-0.07	1.0	0.45	17.46	A	6.68	A	A
03	52.80	1.40	51.75	3.15	6.09	-1.99	-0.2	-0.3	0.98	1.05	8.89	Α	6.64	A	V
04	101.60	2.80	100.9 7	6.15	6.09	-0.62	-0.06	-0.09	66.0	0.63	17.43	A	69.9	A	V
05	<0.10	0.10	0.00	0.00										Α	V
	E		,	,	;	(	t								, į
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	40.98	2.98	7.27	-22.39	-2.24	-3.59	0.78	11.82	8.49	Z	7.74	Υ	Z
02	101.60	2.80	80.55	5.78	7.18	-20.72	-2.07	-3.28	0.79	21.05	16.57	Z	7.69	A	Z
03	52.80	1.40	42.64	3.11	7.29	-19.24	-1.92	-2.98	0.81	10.16	8.80	Z	7.76	A	M
04	101.60	2.80	78.28	5.61	7.17	-22.95	-2.3	-3.72	0.77	23.32	16.18	Z	7.68	A	Z
05	<0.10	0.10	0.04	0.02											V

Value Unc. [%]	e Lab. Unc. E Unc. 1%1	Unc. [%]	1	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
102	0	10.00	9.80	93.18	9.32	4.87	1.93	49.20	26.05	Z	10.16	Α	N
148	0.	12.00	8.11	45.67	4.57	3.77	1.46	46.40	31.79	Z	8.56	А	Z
85.(	00	8.00	9.41	60.98	6.1	3.96	1.61	32.20	20.95	Z	9.78	Α	Z
140	0.0	24.00	17.14	37.80	3.78	1.59	1.38	38.40	62.34	Α	17.36	Z	Z
27.0	00	5.00	18.52										Z
	Lab. Value	Lab.	Unc.	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Ч	Prec.	Final Score
58	.15	1.65	2.84	10.13	1.01	2.47	1.1	5.35	5.58	A	3.88	A	V
11	6.5	2.66	2.28	14.67	1.47	3.86	1.15	14.90	9.96	Z	3.58	А	M
58	.70	1.73	2.95	11.17	1.12	2.65	1.11	5.90	5.74	Z	3.96	Α	M
11	9.4	3.09	2.59	17.52	1.75	4.27	1.18	17.80	10.76	Z	3.78	А	M
0	20	0.02	12.00										Ζ

	Final Score	M	M	M	M	A	Final	A	A	A	M	
	Prec.	Α	Α	A	Α		Prec.	Α	A	A	A	
	Ч	3.22	3.12	3.22	3.15		Р	4.43	4.39	4.24	4.31	
	True	z	z	Z	Z		True	A	A	Α	Z	
	A2	4.27	7.99	4.27	8.08		A2	5.68	10.97	5.49	10.59	
	<b>A1</b>	4.68	11.68	4.53	10.30		A1	4.90	8.00	4.40	11.00	
	Ratio	0.91	0.89	0.91	0.9		Ratio	0.91	0.92	0.92	0.89	
	U-Test	-2.83	-3.77	-2.74	-3.29		U-Test	-2.22	-1.88	-2.07	-2.68	
	Z- Score	-0.89	-1.15	-0.86	-1.01		Z- Sooro	-0.93	-0.79	-0.83	-1.08	
	Rel. Bias	-8.86	-11.50	-8.58	-10.14		Rel. Bias	-9.28	-7.87	-8.33	-10.83	
	Unc. [%]	1.83	1.47	1.82	1.53		Unc.	3.55	3.42	3.31	3.31	
	Lab. Unc.	0.88	1.32	0.88	1.40		Lab.	1.70	3.20	1.60	3.00	
. 31	Lab. Value	48.12	89.92	48.27	91.30	<0.07	Lab. Value	47.90	93.60	48.40	90.60	
ORY NO.	Unc	1.40	2.80	1.40	2.80	0.10	Unc	1.40	2.80	1.40	2.80	
LABORAT	Target Activity	52.80	101.60	52.80	101.60	<0.10	Target	52.80	101.60	52.80	101.60	
ABLE II.9. 1	Sample code	01	02	03	04	05	Sample	01	02	03	04	

TABLE II.1	1. LABOR∕	ATORY N	4O. 38												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	P	Prec.	Final Score
01	52.80	1.40	45.90	3.20	6.97	-13.07	-1.31	-1.98	0.87	6.90	9.01	Α	7.46	Α	A
02	101.60	2.80	90.80	6.20	6.83	-10.63	-1.06	-1.59	0.89	10.80	17.55	A	7.36	A	A
03	52.80	1.40	48.30	3.30	6.83	-8.52	-0.85	-1.26	0.91	4.50	9.25	A	7.33	Α	A
04	101.60	2.80	88.20	6.00	6.80	-13.19	-1.32	-2.02	0.87	13.40	17.08	Α	7.34	Α	A
05	<0.10	0.10	<0.05												A
Sample	Target	llnc	Lab.	Lab.	Unc.	Rel.	-Z-	11_Test	Ratio	41	۸٦	True	٩	Drec	Final
code	Activity		Value	Unc.	[%]	Bias	Score	1631-0	INAULO	IV	74	2011	-	1100	Score
01	52.80	1.40	55.00	3.00	5.45	4.17	0.42	0.66	1.04	2.20	8.54	A	6.06	Α	A
02	101.60	2.80	101.2	5.20	5.14	-0.39	-0.04	-0.07	1.0	0.40	15.24	V	5.83	A	A
03	52.80	1.40	54.50	3.00	5.50	3.22	0.32	0.51	1.03	1.70	8.54	A	6.11	Α	A
04	101.60	2.80	100.2 0	5.10	5.09	-1.38	-0.14	-0.24	0.99	1.40	15.01	A	5.79	A	A
05	<0.10	0.10	0.04	0.03											A

Final Score	Z	Z	Z	Z	A		Final Score	Z	M	A	A	V
Prec.	Υ	Α	Α	Α			Prec.	A	Α	Α	A	
Ρ	7.38	6.87	7.14	7.14			P	5.29	3.50	5.33	4.31	
True	Z	Z	Z	Z			True	z	Z	A	Α	
A2	13.15	22.60	12.65	23.09			A2	8.54	9.68	7.39	11.36	
A1	18.40	30.40	18.10	27.40			A1	12.80	14.50	1.30	1.20	
Ratio	1.35	1.3	1.34	1.27			Ratio	1.24	1.14	1.02	1.01	
U-Test	3.61	3.47	3.69	3.06			U-Test	3.87	3.86	0.45	0.27	
Z- Score	3.48	2.99	3.43	2.7			Z- Score	2.42	1.43	0.25	0.12	
Rel. Bias	34.85	29.92	34.28	26.97			Rel. Bias	24.24	14.27	2.46	1.18	
Unc. [%]	6.88	6.29	6.63	6.59			Unc. [%]	4.57	2.15	4.62	3.31	
Lab. Unc.	4.90	8.30	4.70	8.50			Lab. Unc.	3.00	2.50	2.50	3.40	
Lab. Value	71.20	132.0	70.90	129.0	<0.2	0.57	Lab. Value	65.60	116.1 0	54.10	102.8	<0.15
Unc	1.40	2.80	1.40	2.80	0.10	TORY N	Unc	1.40	2.80	1.40	2.80	0.10
Target Activity	52.80	101.60	52.80	101.60	<0.10	LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10
Sample code	01	02	03	04	05	LABLE II.16	Sample code	01	02	03	04	05

TABLE II.1	7. LABOR/	ATORY N	4O. 58												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	ď	Prec.	Final Score
01	52.80	1.40	51.70	0.40	0.77	-2.08	-0.21	-0.76	0.98	1.10	3.76	A	2.76	A	A
02	101.60	2.80	<u>99.60</u>	0.50	0.50	-1.97	-0.2	-0.7	0.98	2.00	7.34	A	2.80	A	A
03	52.80	1.40	51.70	0.40	0.77	-2.08	-0.21	-0.76	0.98	1.10	3.76	Α	2.76	A	V
04	101.60	2.80	<u>99.60</u>	0.50	0.50	-1.97	-0.2	-0.7	0.98	2.00	7.34	Α	2.80	A	A
05	<0.10	0.10	0.00												A
TABLE II.18	8. LABOR∕	VTORY N	10. 63												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Ρ	Prec.	Final Score
01	52.80	1.40	39.50	4.42	11.19	-25.19	-2.52	-2.87	0.75	13.30	11.96	Z	11.50	Α	Z
02	101.60	2.80	82.90	7.53	9.08	-18.41	-1.84	-2.33	0.82	18.70	20.73	A	9.49	A	A
03	52.80	1.40	45.55	4.97	10.91	-13.73	-1.37	-1.4	0.86	7.25	13.32	A	11.23	A	A
04	101.60	2.80	85.22	4.71	5.53	-16.12	-1.61	-2.99	0.84	16.38	14.14	Z	6.18	Υ	M
05	0.10	0.01	0.03	0.03											A

ATORY NO. 64 Lab. Lab.	40.64 Lab. Lab.	Lab.	1	Unc.	Rel.	Ζ-	Ē	: 4			E	4	4	Final
Unc Lat	Valı	e e	Unc.	UIIC. [%]	Bias	z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Score
1.40 49.97	49.97		2.12	4.24	-5.36	-0.54	-1.11	0.95	2.83	6.55	A	5.00	Α	A
2.80 94.59	94.59		3.69	3.90	-6.90	-0.69	-1.51	0.93	7.01	11.95	Α	4.78	Α	V
1.40 46.97	46.97		1.69	3.60	-11.04	-1.1	-2.66	0.89	5.83	5.66	Z	4.47	Α	M
2.80 95.81	95.81		3.53	3.68	-5.70	-0.57	-1.29	0.94	5.79	11.62	Α	4.60	Α	V
0.01 <0.01	<0.01													V
Unc Lal	Lal Vali	.с	Lab. Unc	Unc.	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	4	Prec.	Final Score
1.40 46.20	46.20		3.00	6.49	-12.50	-1.25	-1.99	0.88	6.60	8.54	A	7.01	V	V
2.80 95.20	95.20	_	6.20	6.51	-6.30	-0.63	-0.94	0.94	6.40	17.55	A	7.07	V	V
1.40 48.20	48.20	0	3.10	6.43	-8.71	-0.87	-1.35	0.91	4.60	8.78	A	6.96	Υ	A
2.80 94.40	94.4(		6.10	6.46	-7.09	-0.71	-1.07	0.93	7.20	17.32	А	7.03	Υ	A
0.10 0.01	0.01		0.01											A

TABLE II.2	1. LABOR∕	ATORY N	4O. 67												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	50.00	1.70	3.40	-5.30	-0.53	-1.27	0.95	2.80	5.68	Α	4.31	Α	A
02	101.60	2.80	95.80	3.10	3.24	-5.71	-0.57	-1.39	0.94	5.80	10.78	A	4.25	A	A
03	52.80	1.40	52.60	1.80	3.42	-0.38	-0.04	-0.09	1.0	0.20	5.88	Α	4.33	A	A
04	101.60	2.80	97.20	3.10	3.19	-4.33	-0.43	-1.05	0.96	4.40	10.78	Α	4.22	A	A
05	<0.10	0.10	<0.07												A
	E		1 - 1	1.	11		Ľ								
Sample code	1 arget Activity	Unc	Lab. Value	Lab. Unc.	Unc.	keı. Bias	z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	59.00	6.60	11.19	11.74	1.17	0.92	1.12	6.20	17.41	Α	11.50	A	A
02	101.60	2.80	53.90	5.82	10.80	-46.95	-4.69	-7.39	0.53	47.70	16.66	Z	11.14	Α	Z
03	52.80	1.40	15.11	1.56	10.34	-71.38	-7.14	-17.96	0.29	37.69	5.41	Z	10.68	A	Z
04	101.60	2.80	69.9	0.70	10.46	-93.42	-9.34	-32.88	0.07	94.91	7.45	Z	10.82	Α	Z
05	<0.10	0.10	0.00												V

	ec. Final Score	A	A	A	A	A		ec. Score	A	M	A	A	A
	Pr	A	Α	Α	Υ			Pr	A	Υ	Α	Α	
	P	7.49	7.53	7.48	7.53			Ч	4.80	4.38	5.01	5.11	
	True	Α	V	Α	A			True	V	Z	Α	Α	
	A2	9.30	17.95	8.80	17.13			A2	60.9	10.59	6.30	12.60	
	A1	5.43	10.67	8.35	15.66			A1	5.30	13.60	5.80	8.60	
	Ratio	0.9	0.89	0.84	0.85			Ratio	0.9	0.87	0.89	0.92	
	U-Test	-1.51	-1.53	-2.45	-2.36			U-Test	-2.25	-3.31	-2.38	-1.76	
	Z- Score	-1.03	-1.05	-1.58	-1.54			Z- Score	-1.0	-1.34	-1.1	-0.85	
	Rel. Bias	-10.28	-10.50	-15.81	-15.41			Rel. Bias	-10.04	-13.39	-10.98	-8.46	
	Unc. [%]	7.01	7.01	7.00	7.00			Unc. [%]	4.00	3.41	4.26	4.30	
	Lab. Unc.	3.32	6.37	3.11	6.02	0.01		Lab. Unc.	1.90	3.00	2.00	4.00	
J. 71	Lab. Value	47.37	90.93	44.45	85.94	0.07	). 72	Lab. Value	47.50	88.00	47.00	93.00	0.10
TORY NC	Unc	1.40	2.80	1.40	2.80	0.10	TORY NC	Unc	1.40	2.80	1.40	2.80	0.10
LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10	LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10
TABLE II.23.	Sample code	01	02	03	04	05	TABLE II.24.	Sample code	01	02	03	04	L C

FABLE II.2:	5. LABORA	ATORY N	4O. 73												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	50.60	2.70	5.34	-4.17	-0.42	-0.72	0.96	2.20	7.85	Α	5.96	Α	A
02	101.60	2.80	90.60	4.70	5.19	-10.83	-1.08	-2.01	0.89	11.00	14.11	A	5.87	А	V
03	52.80	1.40	50.50	2.70	5.35	-4.36	-0.44	-0.76	96.0	2.30	7.85	A	5.97	Α	A
04	101.60	2.80	90.60	4.50	4.97	-10.83	-1.08	-2.08	0.89	11.00	13.67	Α	5.68	А	V
05	<0.10	0.10	0.19	0.07											Z
TABLE II.20	5. LABORA	ATORY N	VO. 74												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	<b>A1</b>	A2	True	Р	Prec.	Final Score
01	52.80	1.40	48.00	3.00	6.25	-9.09	-0.91	-1.45	0.91	4.80	8.54	Α	6.79	A	A
02	101.60	2.80	95.00	5.00	5.26	-6.50	-0.65	-1.15	0.94	6.60	14.78	Α	5.94	Α	A
03	52.80	1.40	49.00	3.00	6.12	-7.20	-0.72	-1.15	0.93	3.80	8.54	A	6.67	Α	A
04	101.60	2.80	98.00	5.00	5.10	-3.54	-0.35	-0.63	0.96	3.60	14.78	A	5.80	Α	A
05	<0.10	0.10	0.13	0.04											V

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TABLE II.2	7. LABOR/	ATORY N	4O. 83												
Sample code	Target Activity	Unc	Lab. Value	Lab. Unc.	Unc. [%]	Rel. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Ч	Prec.	Final Score
01	52.80	1.40	50.80	2.90	5.71	-3.79	-0.38	-0.62	0.96	2.00	8.31	Α	6.29	Α	A
02	101.60	2.80	101.7	5.70	5.60	0.10	0.01	0.02	1.0	0.10	16.38	А	6.25	Α	A
03	52.80	1.40	54.30	3.00	5.52	2.84	0.28	0.45	1.03	1.50	8.54	Α	6.13	Α	A
04	101.60	2.80	96.10	5.30	5.52	-5.41	-0.54	-0.92	0.95	5.50	15.46	A	6.17	Υ	V
05	<0.10	0.10	0.00	0.00										Υ	V
Sample	Target Activity	Unc	Lab. Value	Lab. Unc	Unc.	Rel. Rias	Z- Score	U-Test	Ratio	A1	A2	True	P	Prec.	Final Score
01	52.80	1.40	49.30	3.80	7.71	-6.63	-0.66	-0.86	0.93	3.50	10.45	A	8.15	Α	A
02	101.60	2.80	<u>99.90</u>	4.70	4.70	-1.67	-0.17	-0.31	0.98	1.70	14.11	V	5.45	A	A
03	52.80	1.40	51.70	2.70	5.22	-2.08	-0.21	-0.36	0.98	1.10	7.85	Υ	5.86	Α	A
04	101.60	2.80	102.9	4.60	4.47	1.28	0.13	0.24	1.01	1.30	13.89	V	5.25	Α	V
05	<0.10	0.10	0.02	0.00											A

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	Final Score	Z	A	Z	V	Z		Final Score	V	A	A	A	A
	Prec.	A	Α	A	A			Prec.	A	Α	A	A	
	P	8.68	7.62	8.30	7.81			P	11.40	11.56	11.48	11.49	
	True	z	A	Z	Α			True	A	A	A	Α	
	A2	24.87	17.96	15.08	23.53			A2	16.90	30.79	17.15	30.04	
	A1	62.62	11.89	19.29	17.18			A1	4.90	1.70	5.40	0.30	
	Ratio	2.19	0.88	1.37	1.17			Ratio	1.09	1.02	1.1	1.0	
	U-Test	6.5	-1.71	3.3	1.88			U-Test	0.75	0.14	0.81	-0.03	
	Z- Score	11.86	-1.17	3.65	1.69			Z- Score	0.93	0.17	1.02	-0.03	
	Rel. Bias	118.6 0	-11.71	36.53	16.91			Rel. Bias	9.28	1.67	10.23	-0.30	
	Unc. [%]	8.26	7.11	7.87	7.31			Unc. [%]	11.09	11.23	11.17	11.15	
	Lab. Unc.	9.54	6.38	5.67	8.68	0.38		Lab. Unc.	6.40	11.60	6.50	11.30	0.31
D. 101	Lab. Value	115.4 2	89.70	72.09	118.7	2.45	). 106	Lab. Value	57.70	103.3	58.20	101.3	0.40
TORY NO	Unc	1.40	2.80	1.40	2.80	0.10	TORY NO	Unc	1.40	2.80	1.40	2.80	0.10
. LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10	LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10
TABLE II.29.	Sample code	01	02	03	04	05	TABLE II.30.	Sample code	01	02	03	04	05

Final Score	A	A	A	A	A		Final Score	A	M	A	M	A
Prec.	Α	Α	Α	А			Prec.	Α	Α	Α	А	Α
Ρ	4.87	5.11	4.35	5.85			P	3.40	4.18	3.25	3.46	
True	A	Α	A	A			True	A	Z	A	Z	
A2	6.30	12.60	6.30	14.78			A2	4.52	10.25	4.32	8.76	
A1	3.80	8.60	5.20	4.60			A1	3.44	11.98	3.64	9.70	
Ratio	0.93	0.92	1.1	0.95			Ratio	0.93	0.88	0.93	0.9	
U-Test	-1.56	-1.76	2.13	-0.8			U-Test	-1.97	-3.01	-2.17	-2.86	
L- Score	-0.72	-0.85	0.98	-0.45			Z- Score	-0.65	-1.18	-0.69	-0.95	
Rei. Bias	-7.20	-8.46	9.85	-4.53			Rel. Bias	-6.52	-11.79	-6.89	-9.55	
[%]	4.08	4.30	3.45	5.15			Unc. [%]	2.13	3.15	1.87	2.09	
Lab. Unc.	2.00	4.00	2.00	5.00			Lab. Unc.	1.05	2.82	0.92	1.92	
Lab. Value	49.00	93.00	58.00	97.00	<0.005	O. 124	Lab. Value	49.36	89.62	49.16	91.90	0.00
Unc	1.40	2.80	1.40	2.80	0.10	TORY N	Unc	1.40	2.80	1.40	2.80	0.10
Target Activity	52.80	101.60	52.80	101.60	<0.10	. LABORA	Target Activity	52.80	101.60	52.80	101.60	<0.10
Sample code	01	02	03	04	05	<b>FABLE II.32</b>	Sample code	01	02	03	04	05

	E			1.1	11		Ľ								i
ample code	1 arget Activity	Unc	Lab. Value	Lab. Unc.	Unc.	kei. Bias	Z- Score	U-Test	Ratio	A1	A2	True	Р	Prec.	Final Score
01	52.80	1.40	46.70	0.20	0.43	-11.55	-1.16	-4.31	0.88	6.10	3.65	z	2.69	А	M
02	101.60	2.80	102.6	0.60	0.58	0.98	0.1	0.35	1.01	1.00	7.39	V	2.82	A	V
03	52.80	1.40	52.80	0.20	0.38	0.00	0.0	0.0	1.0	0.00	3.65	Α	2.68	Α	V
04	101.60	2.80	91.60	0.50	0.55	-9.84	-0.98	-3.52	0.9	10.00	7.34	Z	2.81	Α	M
05	<0.10	0.10	0.02	0.00											V

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#### APPENDIX III. TECHNICAL INFORMATION PROVIDED BY THE LABORATORIES

The technical information provided by the participants on the analytical procedures used in their own laboratories is compiled in this Appendix and coded with the same laboratory code used in the data evaluation. The participants can benefit from the information exchange.

#### Short description of sample preparation:

The whole sample was taken from the IAEA bottles and added to a 100 ml beaker. The bottle was washed four times with 3M HCl. The internal tracer 209Po was added to the solution so obtained and stirred for equilibrating tracer and sample. The dissolution was evaporated to near dryness into a hot plate (temperature 90°C). The residue was dissolved with concentrated HCl and evaporated again (this step was repeated three times). The residue was treated with concentrated HCl. hydroxylamine hydrochloride. Bismuth and sodium citrate were also added. The residue was rinsed with distilled water until reaching the appropriate volume for the auto-deposition of polonium following Flynn's method.

#### Short description of the separation method:

Auto-deposition n Ag disc, Flynn.W.W. (1968) .The determination of low level of polonium-210 in environmental materials. Analytical Chimica Acta. 43. pp 221-227.

#### Short description of source preparation:

t is described by Flynn. The Ag disks (2.5 cm of diameter) are placed in a Teflon-cell that contents the sample in citrate medium. The sample is stirred and heated (90° C) during three hours to optimize the auto-deposition recovery.

#### Short description of the detection technique

The counting equipment employed to quantify alpha-emitters is Canberra 7404. The detectors are PIPS (Passivated Implanted Planar Silicon) working with a resolution of 18 keV and very low background counts (0.05 cts/cm2/h). These detectors are placed in chambers connected to vacuum pumps. In a routine basis the counting equipment is calibrated in energy and efficiency with a standard source containing 233U. 239+240Pu and 241Am electroplated onto a stainless steel disc of 2.5 cm of diameter, just like the ones used in the routine analyses. Calibrations are performed every three months and whenever any instrumental changes are performed.

The spectrum analysis is done manually. using both the calibration spectrum to highlight the peaks to be measured in the sample disc and the background spectrum. Due to the high purity of the tracers employed and the good resolution obtained by electroplating no correction of tails is needed.

#### Short description of sample preparation:

Add po-208 tracer, add concentrated nitric acid, heat and evaporate to dryness.

#### Short description of the separation method:

None.

#### Short description of source preparation:

Dissolve residue with concentrated HCl and dry, dissolve with 6M HCl and transfer to beaker, add hydroxyl ammonium chloride, adjust pH to 2 with ammonia or HCl, heat to 85° C, plate on Ag disk. Auto deposition for 3 h.

#### Short description of the detection technique

Not reported.

# Information provided by laboratory No. 14

#### Short description of sample preparation:

Evaporation just to dryness (avoid any heating of the dry residue).

#### Short description of the separation method:

No separation was performed.

#### Short description of source preparation:

Dissolve residue in 120 ml 0.5 M HCl add 10 mg Bi-carrier add 1 g hydroxyl ammonium chloride heat to 95° C. Deposit for 4 hours on cleaned silver disc.

#### Short description of the detection technique

Alpha-spectrometry using surface barrier detectors (PIPs, 300 cm<sup>2</sup>).

#### Short description of sample preparation:

About 5 g of sample was added with about 0.5 g\*23.5396 dpm/g  $^{209}$ Po and evaporated on hot plate until dry.

#### Short description of the separation method:

None.

#### Short description of source preparation:

Dissolved residue from above with small amount of 6N HCl and then dissolved and made up volume to 100ml with 0.3N HCl. Auto deposition on 99.99%purity Ag disc with addition of small amount of solid ascorbic acid to prevent deposition of Fe+3 on disc. The deposition took overnight at room temperature with continuous stirring.

At the end of plating, the sample was taken out and washed with distilled water, let dry and counted under ORTEC ion-implanted, silicon, partially depleted, charged-particle detector coupled to Tennelec multi-channel analyzers. The sample was counted until at least 1,000 counts obtained to minimize the counting error.

#### Short description of the detection technique

Tennelec Nucleus Model TC256

# Information provided by laboratory No. 21

#### Short description of sample preparation:

About 5 ml of sample was taken and weighted. To the sample, 0.1-0.5 Bq of Po-209 was added and weighted. Sample was evaporated on a hot plate at temperature less than 70 °C till dryness. Then, 5 ml of concentrated HCl (37%) was added and again been evaporate to dryness. Then the sample was dissolved in 120 ml 0.5 Molar HCl solution.

#### Short description of the separation method:

No separation.

#### Short description of source preparation:

The solution was heated while stirring to temperature around 60 °C. Then ~0.5 gram ascorbic acid was added, followed by ~ 0.5 gram boric acid. After the salt was dissolved, the heater was turn off. A 2cm x 2cm polished silver foil was dip and hook in the solution (one side of the foil was coated with varnish). With continuous stirring, the foil was left in the solution for 24 hours. Then, the silver foil was rinsed with distilled water and air dried before counting.

#### Short description of the detection technique

OCTETE PLUS Alpha Spectrometer. Samples were counted using alpha spectrometry at shelf 3 for few hours (depend on the counts of sample peak area). The peak area was marked and calculated, then corrected to the reference date.

# Information provided by laboratory No. 22

#### Short description of sample preparation:

Weighed aliquot Sample was taken in a glass beaker, acidified with 1 ml Conc. HCl and evaporated to near dryness under IR lamp (80 °C).

#### Short description of the separation method:

None.

#### Short description of source preparation:

Nearly dried sample was taken in a 100ml of 1N HCL and added 100mg of ascorbic acid. Heat the solution for 60-80 °C on a burner. Silver disc was dipped in the warm solution for 2 hour under continuous stirring.

#### Short description of the detection technique

PIPS detector, disc was counted in alpha spectrometric system on both sides for 10000sec each. Canberra model 7401, 1K MCA, 30-40  $\mu$ mHg vacuum.

# Information provided by laboratory No. 23

#### Short description of sample preparation:

No information reported.

#### Short description of the separation method:

No information reported.

#### Short description of source preparation:

No information reported.

#### Short description of the detection technique

No information reported.

#### Short description of sample preparation:

Po-209, hydrochloric acid and water were added to samples to obtain 200 mL of 0.5 M HCl solution. Spontaneous deposition of polonium took place on one side of silver disk (18 mm in diameter) in the stirred solution at 85  $^{\circ}$ C for 4 hrs.

#### Short description of the separation method:

Deposition.

#### Short description of source preparation:

Deposition on one side of a silver disk from the solution as given above.

#### Short description of the detection technique

Alpha spectrometry, The alpha spectrometer consisted of PIPS detector (A-300-17) with efficiency of 34% placed in vacuum chamber, connected to a multi-channel analyzer MULTIPORT II MCA and GENIE-2000, Canberra for spectra analyses.

# Information provided by laboratory No. 31

#### Short description of sample preparation:

The sample was prepared by EML Procedure Manual (HASL-300, Po-01-RC).

#### Short description of the separation method:

No separation method was used.

#### Short description of source preparation:

Not reported.

#### Short description of the detection technique

Alpha Analyst (CANBERRA, 8 Chamber) was used.

#### Short description of sample preparation:

Addition of tracer to the sample, addition of acid, evaporation to residue. Dissolution of residue with 0.5M HCl, addition of ascorbic acid.

#### Short description of the separation method:

Not applied in this case.

#### Short description of source preparation:

Deposition onto silver disc overnight.

#### Short description of the detection technique

Low background 400  $\,\mathrm{mm}^2$  ion implanted detectors. Use of Octecte Plus and Maestro software.

# Information provided by laboratory No. 38

#### Short description of sample preparation:

Addition of Po-209 tracer and Evaporation.

#### Short description of the separation method:

Eichrom Sr Spec 2g column.

#### Short description of source preparation:

Plated at 85 °C for 4 hours at pH 2.0 onto 27mm 92 % silver discs.

#### Short description of the detection technique

PIPS

#### Short description of sample preparation:

Tenfold dilution, addition of lemon and ascorbic acid. Auto-deposition (2 hours) on stainless steel discs at 80 °C.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

Alpha-spectrometer with PIPS detector of 3000mm<sup>2</sup> of active area was used.

# Information provided by laboratory No. 45

#### Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

No information was provided.

#### Short description of sample preparation:

The sample is evaporated to dryness by heating at 85 °C and residue is taken with 100 mL of 0.5M HCl in a baby nursing bottle with bottom removed. A silver disk (2.5cm diameter) rested on Teflon base disc was held in the screw top of the bottle by a neoprene gasket. The bottle was placed in a water beaker that was heated at about 100?C over heater for about 5 hours continuously. A plastic cover with a centre hole for the stirrer was used to reduce evaporation.

#### Short description of the separation method:

Deposition.

#### Short description of source preparation:

After completion of 5 hours heating and stirring of the solution, polonium will be deposited on the silver disk. The disk is then removed and washed with distilled water and dried under normal temperature.

#### Short description of the detection technique

The silver disk is used for measurement by alpha spectrometer. Silver disk source is placed inside detector chamber and the source is counted for 18 hours following the recommended procedure. 5.30 MeV energy of Po-210 is used for peak area counts.

# Information provided by laboratory No. 52

#### Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

CANBERRA 7401/VR, detector PIPS d=450 mm<sup>2</sup> resolution 18keV.

#### Short description of sample preparation:

Evaporation was carried on the sample as supplied, no carrier was added. Sample was taken to near dryness (temperature not exceeding 130 °C) and 5mL of concentrated hydrochloric acid was added. Sample was taken to near dryness before adding 20 mL 0.5 M hydrochloric acid for the deposition.

#### Short description of the separation method:

Not applied.

#### Short description of source preparation:

With the sample from evaporation step re-dissolved in 20 mL 0.5 M hydrochloric acid, 0.2 g of ascorbic acid and 2 mL of 20% hydroxlamine hydrochloride were added. pH adjusted to 1.5-2.0 using 25 % ammonia solution. The sample was auto-deposited for 1.5 hours at temperature of 85-90 degrees C and stirring at 350 rpm. The disk is rinsed with de-ionised water and acetone and counted after 24 hours.

#### Short description of the detection technique

1.5 torr chamber pressure, source-detector separation 0.5mm, ion re-coil protection 5 V, PIPS  $450 \text{mm}^2$ .Canberra genie 2000 software with alpha analysis option. ROI -100 to +50 keV around nominal alpha energy efficiency correction using Polonium-208 tracer.

# Information provided by laboratory No. 57

#### Short description of sample preparation:

Known weight of sample aliquot was taken in a glass beaker and evaporated to dryness on low heat to prevent volatilization of Po-210.The residue was dissolved in 100 ml of 0.5M HCl.

#### Short description of the separation method:

No separation method was used.

#### Short description of source preparation:

Auto-deposition on Silver disc from a 0.5M HCl solution at 70-80 °C for 4 hours was applied.

#### Short description of the detection technique

Gross Alpha counting done using ZnS(Ag) Alpha counting system.

# Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

No information was provided.

# Information provided by laboratory No. 61

#### Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

No information was provided.

#### Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

Spontaneous deposition onto silver disk, silver content 99.9%, diameter of the silver disk is 23 mm. Deposition time 4 hours, temperature 70 - 80 °C.

#### Short description of the detection technique

Alpha Analyst, genie2000, Canberra Alpha Analyst alpha spectrometer was used.

# Information provided by laboratory No. 67

#### Short description of sample preparation:

sample is evaporated to dryness with addition of 2M HCL between evaporations. Sample is finally dissolved in 30mL of 2M HCL.

#### Short description of the separation method:

Not applied.

#### Short description of source preparation:

Ascorbic acid is added to reduce Fe(III). Silver disks are mounted on stirrer and plated for 1 hour at 60 °C.

#### Short description of the detection technique

Canberra alpha Analyst system. Area determination by region of interest. Calibration with tracer (Po-208 or Po-209).

#### Short description of sample preparation:

No information was provided.

#### Short description of the separation method:

No information was provided.

#### Short description of source preparation:

No information was provided.

#### Short description of the detection technique

No information was provided.

# Information provided by laboratory No. 71

#### Short description of sample preparation:

The sample preparation is based on a co-precipitation method for the determination of Po-210 in the biological and environmental samples. In this work, a simplified procedure is used as described below:

Some of concentrated HCL, Fe(III), and Po-209 as a yield tracer are added to 2.5 g of water sample, which is then heated on a hot-plate until boiling for some minutes. 5 ml of 20% hydroxylamine hydrochloride and 5 ml of 25% sodium citrate solution are added. The solution is adjusted to pH 1-1.5 with ammonia, diluted to 50 ml, and heated and stirred on a magnetic hot-plate.

#### Short description of the separation method:

Auto deposition.

#### Short description of source preparation:

A Perspex holder with a silver disk is placed on the beaker and the silver disk is immersed into the solution. Po deposition is continued for 4h at 85-90 ?C, and then the disk is removed, washed with distilled water and acetone, dried and assayed by alpha spectrometry.

#### Short description of the detection technique

CANBERRA SYSTEM, Alpha spectrometer(Model 7401 VR, Canberra) is equipped with a passivated implanted planar silicon detector (450mmq), PIPS A450 18AM. Acquisition Interface Model 556 A. Genie 2000 ver 3.1 ALPHA acquisition and spectrometry analysis software. Vacuum Pump was used.
# Short description of sample preparation:

A small part of the sample was brought to pH about 1.5; hydroxyl ammonium chloride was added.

# Short description of the separation method:

None.

# Short description of source preparation:

The silver plating is so selective that a pre-separation is not necessary, on Ag-disk.

# Short description of the detection technique

Ortec Octete detector.

# Information provided by laboratory No. 73

# Short description of sample preparation:

2 ml sample diluted in 250 ml HCl 0,5 M. addition of 20 mBq of Po-209 as a tracer.

# Short description of the separation method:

None.

# Short description of source preparation:

Auto-deposition on silver disc by agitation at 95°C temperature for 4 hours.

# Short description of the detection technique

Alpha spectrometry, measurement of the deposited sample using 450 mm<sup>2</sup> PIPS detectors in vacuum chamber.

# Short description of sample preparation:

Dilution with water to 0.5 M HCl, spiked with Po-209.

# Short description of the separation method:

None.

# Short description of source preparation:

Auto deposition on silver disc.

# Short description of the detection technique

Alpha spectrometry with ion implanted Si detector and measurement with cells under vacuum.

# Information provided by laboratory No. 83

# Short description of sample preparation:

A Po-209 tracer was added to the solution, in addition to citric acid and hydroxylamine hydrochloride. Short description of the separation method:

# Short description of the separation method:

No separation methods were used.

### Short description of source preparation:

Auto deposition onto silver in dilute hydrochloric acid (2 hours at 85 °C)

### Short description of the detection technique

Alphas spectrometry with surface barrier detectors and manually set regions of interest was used. No tailing corrections were made.

### Short description of sample preparation:

Sample and tracer Po-209 were weighed on analytical balance and 10 mL of 2 M HCl was added to the sample. Sample was diluted to the 100 mL with distilled water, stirred with magnetic stirrer and heated to 60 °C.

# Short description of the separation method:

No separation methods were used.

### Short description of source preparation:

Po-210 and tracer Po-209 were auto deposited on copper disc for 4 hours.

# Short description of the detection technique

Canberra Alpha Analyst with PIPS detectors and Genie-2000 software; some older IPS detectors with Maestro software.

Source was measured in alpha spectrometer and activity was calculated with the help of tracer Po-209.

# Information provided by laboratory No. 101

# Short description of sample preparation:

Direct method by silver plate was used.

# Short description of the separation method:

No information was reported.

### Short description of source preparation:

No information was reported.

# Short description of the detection technique

No information was reported.

# Short description of sample preparation:

The sample aliquot was diluted to 200 ml with distilled water and the pH adjusted to between 1.5 and 2 with HCl.

# Short description of the separation method:

Spontaneous deposition.

# Short description of source preparation:

The spontaneous deposition procedure is very specific for Polonium; no other separation procedure is required.

Hydroxyl ammonium chloride is added to sample to prevent interference from Fe.

Polonium-209 was added as yield tracer. Sample with silver disc (covered on one side) is warmed to a temperature of 90 °C and stirred for 4 hours to induce spontaneous deposition.

The insides of the beaker are washed down and the volume adjusted to 200 ml frequently during the plating period. Disc removed from solution, rinsed and air-dried.

# Short description of the detection technique

Alpha Analyst Model S470, PIPS detectors. Samples counted for 24 hours. The number of pulses in the specified ROI in the pulse height spectra are used for data reduction (Po-209:4.51-4.91 MeV and Po-210:4.92-5.39 MeV).

# Information provided by laboratory No. 119

# Short description of sample preparation:

Only evaporation with nitric acid was applied.

### Short description of the separation method:

No separation.

# Short description of source preparation:

No information was reported.

### Short description of the detection technique

Si detector in vacuum was used.

# Short description of sample preparation:

Precipitation with FeCl3.

# Short description of the separation method:

Auto deposition.

Short description of source preparation:

Po-210 is plated out of a weak acid solution onto copper foil.

Short description of the detection technique

**PIPS - DETECTOR** 

# Information provided by laboratory No. 125

### Short description of sample preparation:

An aliquot of the sample was taken for preparation. To it a known quantity of tracer was added (approximately 0.09 Bq). The sample was heated under controlled temperature (< 80 °C) until almost the dryness. The volume was adjusted with HCL 0.5 M until 75 ml. The deposition was undertaken with controlled temperature (< 80 °C) during 4 hours.

### Short description of the separation method:

None.

### Short description of source preparation:

Spontaneous deposition onto a silver disk was used during 4 hours in HCl 0.5 M.

### Short description of the detection technique

Time counting: 60000 seconds.

# LIST OF PARTICIPANTS

AUSTRALIA	Australian Radiation Protection & Nuclear Safety Agency (ARPANSA) Lower Plenty Road 619 Yallambie, Victoria 3085
	Australian Nuclear Science and Technology Organization ANSTO – Environment New Illawarra Rd 2234, Lucas Heights Menai N.S.W.
BELARUS	Belarussian State Institute of Metrology Research Department of Radioactive Metrology (RDRM) Starovilenski Trakt 93 220053, Minsk
BRAZIL	Brazilian National Commission for Nuclear Energy (CNEN) Instituto de Radioprotecao e Dosimetria (IRD) Avda Salvador Allende S/N - Jacarepagua Cep - 22780-160 Rio de Janeiro, RJ
BULGARIA	Executive Environment Agency Ministry Of Environment & Water 136 Tzar Boris Iii, Blvd. P.O. Box 251 Bg-1618, Sofia
CHINA	China Institute of Atomic Energy P.O. Box 275-24 Beijing, 102413
DENMARK	Risoe National Laboratory P.O. Box 49 Dk-4000, Roskilde
FINLAND	Radiation & Nuclear Safety Authority (STUK) P.O. Box 14 Fin-00881, Helsinki
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UNITED STATES OF AMERICA	University of California Lawrence Livermore National Laboratory 7000 East Avenue P.O. Box 5001 Livermore, CA 94551

# CONTRIBUTORS TO DRAFTING AND REVIEW

C.K. Kim

G. Kis-Benedek P. Martin

- U. Sansone
- A. Shakhashiro
- A. Trinkl

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