Worldwide Open Proficiency Test
IAEA-CU-2007-09/A
Determination of Po-210 in Water
The following States are Members of the International Atomic Energy Agency:

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<th>Nigeria</th>
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<td>Denmark</td>
<td>Mexico</td>
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<tr>
<td>Dominican Republic</td>
<td>Monaco</td>
<td>United Kingdom of Great Britain and Northern Ireland</td>
</tr>
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<td>Ecuador</td>
<td>Mongolia</td>
<td>United Republic</td>
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<td>Zimbabwe</td>
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<tr>
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The Agency’s Statute was approved on 23 October 1956 by the Conference on the Statute of the IAEA held at United Nations Headquarters, New York; it entered into force on 29 July 1957. The Headquarters of the Agency are situated in Vienna. Its principal objective is “to accelerate and enlarge the contribution of atomic energy to peace, health and prosperity throughout the world”.

Worldwide Open Proficiency Test
IAEA-CU-2007-09/A: Determination of Po-210 in Water

IAEA-CU-2007-09/A
FOREWORD

The IAEA helps the Member States’ laboratories to maintain their readiness by coordination activities, by development of standardized methods for sample collection and analysis, and by conducting interlaboratory comparisons and proficiency tests as a tool for external quality control.

The Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the International Atomic Energy Agency’s Seibersdorf Laboratories in Austria, has the programmatic responsibility to support global radionuclide measurement systems. To fulfil this obligation and ensure a reliable worldwide, rapid and consistent response, the Chemistry Unit organises interlaboratory studies and proficiency tests.

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 Member States to address this issue. Responding to these requests, the Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the Agency’s Laboratories, conducted a worldwide proficiency test 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 officer responsible for this publication is A. Shakhashiro of the Agency’s Laboratories, Seibersdorf.
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1. INTRODUCTION

The Po-210 poisoning event which occurred in November 2006 [1] demonstrated the need for rapid and accurate determination of this radionuclide in environmental samples. Responding to many requests from Member States, it was decided to conduct a world wide proficiency test on the determination of Po-210 in water. The aim of this proficiency test was to gather information on the current state of practice for Po-210 measurements at various levels in aqueous samples and to assist the participating laboratories in improving the quality of the analytical results.

In the proficiency test described in this report, 635 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 activities of Po-210 and were requested to return the results within one week of receipt of the samples.

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.

114 laboratories from 127 initially registered 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 list of participants is reported in Appendix III.

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

![Fig.1. Summary evaluation of 570 measurement results of Po-210.

Although the matrix was pure acidified water and the activity concentrations were relatively high, 19% of the reported results failed to pass the proficiency test criteria. In few cases positive results were reported for the blank sample which suggests a possibility of false positive reporting.

The result of the overall summary evaluation of this proficiency test showed that 86 laboratories reported results which fit the purpose of rapid detection of Po-210 in water.

Figure 1 reports the summary of the analytical data evaluation of this proficiency test. 68% of all reported results were ‘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 Member States laboratories for rapid determination of Po-210 in liquid matrix,
- evaluating the probability of reporting false positive and 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

In this proficiency test 127 laboratories from 56 countries all over the world were registered on-line. 114 participants from 46 countries reported their results back to the IAEA via the designated on-line application. The list of participants can be found in Appendix III. The participating laboratories geographical distribution is shown in Figures 2 and 3.

2.3. Composition of the proficiency test materials

The set of the proficiency test materials consisted of 5 samples each 50 mL. The following proficiency test design was applied:

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

Figure 4 shows a set of the packed PT samples.

Table 1 lists the target values and the associated combined standard uncertainty of the PT materials and the PT performance criteria LAP and MAB (see Section 3).

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.
Fig. 2. Participants’ distribution by country.

Fig. 3. Geographical distribution of the participants.
The final target activity concentration for Po-210 was calculated from the certified activity value assigned to the certified standard solution of Po-210, 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±10 Bq.g⁻¹.
The reference date for results reporting was set to the 1st of April 2007.

2.5. Homogeneity testing

Three bottles from each batch were measured using liquid scintillation counter 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 5.

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.

![Homogeneity and Stability test](image)

*Fig. 5. Homogeneity and stability test results, four sets of measurements, and one set every two weeks. Reference date: 1st 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.

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 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 $\text{Value}_{\text{Analyst}}$ (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:

$$\text{Relative bias} = \frac{\text{Value}_{\text{Analyst}} - \text{Value}_{\text{IAEA}}}{\text{Value}_{\text{IAEA}}} \times 100\%$$

3.2. PT 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 [3].
3.2.1. Trueness

The participant result is assigned ‘acceptable’ status for trueness if:

\[ A1 \leq A2 \]

where:

\[ A1 = \left| \text{Value}_{\text{IAEA}} - \text{Value}_{\text{Analyst}} \right| \]

\[ A2 = 2.58 \times \sqrt{\text{Unc}_{\text{IAEA}}^2 + \text{Unc}_{\text{Analyst}}^2} \]

3.2.2. Precision

For evaluation of precision an estimator P is calculated for each participant, according to the following formula:

\[ P = \frac{\text{Unc}_{\text{IAEA}}^2}{\text{Value}_{\text{IAEA}}} + \frac{\text{Unc}_{\text{Analyst}}^2}{\text{Value}_{\text{Analyst}}} \times 100\% \]

P directly depends on the measurement 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 precision when \( P \leq \text{LAP} \). The LAP value used in the evaluation of all radionuclides is listed in Table 1.

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 R. Bias > MAB, the result will be ‘not acceptable’. However, if R. Bias \( \leq \text{MAB} \), the final score will be ‘warning’. A ‘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 results close to the assigned property value are reported, but the associated uncertainty is large. The MAB value used in the evaluation of all radionuclides is listed in Table 1.

3.2.3. 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:

\[ |\text{Value}_{\text{Analyst}} - \text{Unc}_{\text{Analyst}}| < 0.1 \]
Also if the laboratory reported the MDL 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:

\[
0.10 \times Value_{IAEA}
\]

The laboratory performance is evaluated as satisfactory if \(| Z_{score} | \leq 2\); questionable for \(2 < | Z_{score} | < 3\), and unsatisfactory for \(| Z_{score} | \geq 3\).

### 3.4. The u-score value

The value of the \(u\)-test was calculated according to the following equation [4]

\[
u_{test} = \frac{| Value_{IAEA} - Value_{analyst} |}{\sqrt{Unc_{IAEA}^2 + Unc_{analyst}^2}}
\]

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 normalised 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 level of probability at 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

570 measurement results were reported to the IAEA in this PT from 114 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 68% 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, 19% of all reported results were not acceptable against the PT criteria.

4.2. Technical information provided by the participants

The summary of the technical information provided by the participants on the analytical procedures used in their own laboratories is compiled in Tables 4 and 5. The information is 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.

From the reported information on the applied analytical procedure, most of the participants did not use any separation method due to the nature of the matrix. For source preparation, 88 laboratories used auto deposition method on silver or stainless steel disk, only 8 participants used electro-deposition, one laboratory (42) used co-precipitation.

Different measurement techniques were used by the participating laboratories as illustrated in Table 2.

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.

4.3. False positive reporting

In this PT the method detection limit (MDL) was an important performance indicator. The participants were asked to report the MDL estimated through method validation.

From the technical information provided by the laboratories, it can be observed that there is no harmonised method for MDL estimation amongst Member States 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 the MDL is shown in Table 6.

It can be noticed that 21 laboratories (group C, Table 3) reported false positive for the blank sample 05, this might indicate that the MDL was not appropriately estimated, or the method validation for such a matrix was not yet performed.
### TABLE 2. AVERAGE PERFORMANCE SCORE AGAINST THE USED MEASUREMENT TECHNIQUE

<table>
<thead>
<tr>
<th>Measurement technique</th>
<th>Number of laboratories</th>
<th>Average score [%]</th>
</tr>
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<tbody>
<tr>
<td>Alpha spectrometry</td>
<td>88</td>
<td>76</td>
</tr>
<tr>
<td>Liquid scintillation</td>
<td>11</td>
<td>69</td>
</tr>
<tr>
<td>Proportional counter</td>
<td>5</td>
<td>56</td>
</tr>
<tr>
<td>Gross alpha counter</td>
<td>2</td>
<td>75</td>
</tr>
<tr>
<td>ZnS counter</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>Not reported</td>
<td>6</td>
<td>-</td>
</tr>
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</table>

### TABLE 3. LABORATORIES GROUPING RELATED TO THE CATEGORY OF RECOMMENDATIONS

<table>
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<th>Group</th>
<th>Laboratory code</th>
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<tr>
<td>Group A</td>
<td>1, 2, 3, 4, 5, 7, 8, 9, 12, 14, 15, 16, 18, 19, 21, 24, 25, 26, 27, 28, 30, 31, 33, 34, 35, 38, 39, 40, 42, 43, 46, 47, 50, 51, 55, 56, 57, 58, 59, 60, 63, 64, 65, 67, 69, 71, 72, 73, 74, 76, 78, 79, 81, 83, 84, 85, 87, 88, 89, 91, 92, 93, 94, 96, 97, 100, 102, 103, 104, 105, 106, 107, 108, 109, 110, 112, 114, 115, 117, 119, 121, 123, 124, 125, 126, 127.</td>
</tr>
<tr>
<td>Group B</td>
<td>2, 6, 17, 18, 19, 20, 23, 24, 26, 29, 37, 41, 44, 49, 51, 52, 56, 57, 62, 69, 70, 77, 82, 83, 86, 89, 90, 91, 93, 95, 98, 99, 101, 110, 125</td>
</tr>
<tr>
<td>Group C</td>
<td>3, 9, 13, 17, 20, 23, 30, 59, 62, 73, 80, 84, 85, 93, 95, 98, 99, 101, 110, 114, 118.</td>
</tr>
</tbody>
</table>
### TABLE 4. SUMMARY INFORMATION ON THE ANALYTICAL PROCEDURE AS REPORTED BY THE PARTICIPANTS IS PRESENTED AGAINST THE AVERAGE SCORE, WHERE NUMBER OF ‘A’ WAS MULTIPLIED BY 20, ‘W’ BY 10. NR: NOT REPORTED

<table>
<thead>
<tr>
<th>Lab.</th>
<th>Sample</th>
<th>Source preparation</th>
<th>Measurement technique</th>
<th>Average score</th>
</tr>
</thead>
<tbody>
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<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>90</td>
</tr>
<tr>
<td>3</td>
<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>80</td>
</tr>
<tr>
<td>4</td>
<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>70</td>
</tr>
<tr>
<td>5</td>
<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>100</td>
</tr>
<tr>
<td>6</td>
<td>Chelation</td>
<td>Electro deposition</td>
<td>ZnS counter</td>
<td>20</td>
</tr>
<tr>
<td>7</td>
<td>Evaporation</td>
<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>100</td>
</tr>
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<td>8</td>
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<td>Deposition</td>
<td>Liquid scintillation counter</td>
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<td>Auto deposition</td>
<td>Alpha spec.</td>
<td>80</td>
</tr>
<tr>
<td>19</td>
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<td>Auto deposition</td>
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<tr>
<td>21</td>
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<td>NR</td>
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<tr>
<td>2</td>
<td>yes</td>
<td>Minimum detection limit (MDL) = 0.06Bq/kg. Repeatability = 0.64 at 10.3Bq/kg</td>
<td>A</td>
<td></td>
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<tr>
<td>3</td>
<td>yes</td>
<td>Reproducibility: analysis on SRM (f.e. IAEA 134 + 368), one intercomparison test, Repeatability on spiked water samples (variation coefficient of 5.3% with uncertainty of 15%)</td>
<td>N</td>
<td></td>
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<tr>
<td>4</td>
<td>yes</td>
<td>Detection Limit (MDL) calculated according to ISO-11929 standard, MDL= 0.013 Bq/Kg (99%) (Confidence level)</td>
<td>A</td>
<td></td>
<td></td>
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<tr>
<td>5</td>
<td>no</td>
<td>For a 1000 minute count the MDL= 0.01Bq/kg</td>
<td>A</td>
<td></td>
<td></td>
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<tr>
<td>6</td>
<td>yes</td>
<td>Documents and records were first set to meet trackability and traceability requirements, where internal quality control mechanisms have been adopted. Methods stability was checked by means of Z-score control charts. Internal method validation parameters including method detection limits, repeatability limits, reproducibility limits, recovery coefficient and relative error were estimated. External method validation has been achieved by participating in national inter-laboratory exercises and international intercomparison exercises and proficiency tests.</td>
<td>A</td>
<td></td>
<td></td>
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<tr>
<td>7</td>
<td>no</td>
<td>detection limit: 0.01 - 0.06 Bq/kg</td>
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<td></td>
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<tr>
<td>8</td>
<td>no</td>
<td>NR</td>
<td>A</td>
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<tr>
<td>9</td>
<td>yes</td>
<td>Minimum detection limit: ranges between 0.2-0.4 mBq depending on the camera used.</td>
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<td></td>
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<td>12</td>
<td>yes</td>
<td>All 5 samples analysed first based on 20 mL aliquots with DL's below 0.1 Bq/kg. Analyses of samples 1-4 repeated based on 2-3 mL aliquots to match spike amounts.</td>
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<td>13</td>
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<td>14</td>
<td>yes</td>
<td>MDL: 0.04 Bq/kg (Po-210)</td>
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<tr>
<td>15</td>
<td>no</td>
<td>We do not routinely analyse for Po-210. We have performed limited cross-checking with other laboratories.</td>
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<tr>
<td>18</td>
<td>yes</td>
<td>MDL is 0.001 Bq/sample</td>
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<td>19</td>
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<td>Minimum Detection Limit : 0.04 Bq/kg, Repeatability: 5%, Reproducibility: 8%</td>
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<tr>
<td>22</td>
<td>yes</td>
<td>a) Spectrometric system is checked by counting tracer (Pu-242) for MDL level concentration. b) Background count rate fro reproducibility</td>
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<td>24</td>
<td>yes</td>
<td>MDL is about 1 mBq for about 24 h counting time</td>
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<td>25</td>
<td>yes</td>
<td>Method was validated against SRM4337 (Pb-210 standard that is equilibrium with Po-210 standard)</td>
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<tr>
<td>28</td>
<td>yes</td>
<td>ROI 40-400 keV, statistics of single measurement ±5%, statistics of 3 repetitive measurements for result</td>
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<td>29</td>
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<tr>
<td>31</td>
<td>yes</td>
<td>Minimum detection limit was calculated by L. A. Currie's equation. The MDL is 0.07 Bq/kg. 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%.</td>
<td>A</td>
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<tr>
<td>Lab code</td>
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<tr>
<td>33</td>
<td>yes</td>
<td>MDL = &lt; 0.05 Bq/kg</td>
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<td>34</td>
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<td>MDL = &lt; 0.05 Bq/kg</td>
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<td>35</td>
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<tr>
<td>36</td>
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<td>MDL = 0.65 +/- 0.16 Bq/kg, Repeatability limit = 0.88 Bq/kg. No reproducibility limits are available at this time. Minimum detectable activity is determined by applying the average of eight replicate analysis of a blank sample to the Currie formula. Repeatability is determined using eight replicate analysis of a NIST traceable reference material. Similar reference materials are used to determine four point linearity.</td>
<td>A</td>
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<tr>
<td>37</td>
<td>yes</td>
<td>Minimum Detection Limit = 0.002 Bq/L, Standard deviation of the mean = 13%, Bias = 1.4 %</td>
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<td>38</td>
<td>yes</td>
<td>MDL is dependant on sample mass. For low level work where 100g of sample is taken the MDL is 0.005Bq/kg. For these IAEA samples where the activity was very high 4 g of sample was used to achieve an MDL around 0.1Bq/kg. Repeatability at k=2 is 4.2%</td>
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<td>39</td>
<td>yes</td>
<td>For this PT MDL=0.15Bq/kg, at Counting Time 25200s, sample Mass 10.4g, Counting Efficiency 37.2%, Recovery 96% (for low salinity waters)</td>
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<td>40</td>
<td>no</td>
<td>Although we did not participate yet in a Po-210 intercomparison exercise, we have calculated our Minimum detection limit: for 20 grams of sample, 3 hours of measuring time, efficiency 0.4 and chemical yield 0.95= 0.08 Bq/Kg</td>
<td>-</td>
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<tr>
<td>41</td>
<td>yes</td>
<td>Minimum detection activity MDL: 0.0123 Bq on TENNELEC S5HP / Gas flow type proportional counter , 36000 sec. counting time ; Minimum detection activity: 0.0062 Bq on TENNELEC TC 257/IPA Silicon detector, SCA counting mode, 60000 sec Counting time .</td>
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<td>42</td>
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<tr>
<td>46</td>
<td>yes</td>
<td>1-MDL=0.00481Bq/L, 2-Triplicate samples analysed, 3-Above 95%.</td>
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<tr>
<td>49</td>
<td>yes</td>
<td>Minimum Detection Limit: 0.31 Bq/kg, Repeatability limit: 0.15%, Reproducibility limit: 0.25%</td>
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<td>50</td>
<td>yes</td>
<td>Minimum detection limit: 0.1 Bq/kg</td>
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<td>51</td>
<td>yes</td>
<td>Not especially for Po-210, Validation parameters.</td>
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<td>52</td>
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<tr>
<td>54</td>
<td>no</td>
<td>Method validation for polonium in solid, water and urine samples are in progress.</td>
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<td>55</td>
<td>yes</td>
<td>Testing of reproducibility with certified standard</td>
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<tr>
<td>56</td>
<td>yes</td>
<td>The validation method was carried out for urine sample and is believed to be valid for water sample as well. Minimum detection limit: 1 mBq/L when 500 ml of urine is introduced in the analysis, repeatability: 10% at the 20 mBq/L level of activity.</td>
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<tr>
<td>57</td>
<td>yes</td>
<td>Minimum Detection Limit for Sample Code 1 to 4 - 0.2 Bq/Kg (3 sigma). Repeatability ±3%, Reproducibility ±4%</td>
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<tr>
<td>58</td>
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<td>59</td>
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<td>NR</td>
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</table>

The detection limit for Po-210 detection in water samples was calculated by the following formula:

\[ DL = \frac{3 \times \sqrt{C_b}}{(T \times E \times M)} \]

where DL is the detection limit, \( C_b \) is the background counts, \( T \) is the background counting time, \( E \) is the efficiency of detecting alpha particles and \( M \) is the sample mass. For \( C_b = 3000 \) counts, \( T = 300000 \) sec, \( E = 0.867 \) and \( M = 4 \) g the detection limit is \( DL = 0.16 \) Bq/Kg. If a water sample of 7 g is used the detection limit is reduced to 0.08 Bq/Kg.

The repeatability limit was calculated by using the formula \( R = 1.96 \times \sqrt{2} \times S \) where \( S \) is the standard deviation of activity A calculated by repeated measurements of each sample (4 measurements for each sample). Repeatability Limits in Bq/Kg were calculated for each sample and were found: sample#1: 3.5, sample#2: 5.4, sample#3: 8.0, sample#4: 8.8, sample#5: 1.2.
<table>
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<tr>
<th>Lab code</th>
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<th>Blank sample score</th>
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<tr>
<td>62</td>
<td>yes</td>
<td>In order to determine any bias, the method validation procedure was carried out via the analysis of a standard Po-210 solution supplied in National Metrological Institute, with identical sample counting geometry and detector configuration. For the purposes of this exercise, the minimum detection limit achieved for this methodology was 0.34 mBq/kg (95% confidence level). The repeatability limit was 3%.</td>
<td>N</td>
</tr>
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<td>63</td>
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</tr>
<tr>
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<tr>
<td>65</td>
<td>yes</td>
<td>MDL = 0.01 Bq/kg, repeatability = 6%</td>
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<tr>
<td>67</td>
<td>yes</td>
<td>MDC 0.1 Bq/kg for 5 g sample and 23 hour counting time.</td>
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<tr>
<td>69</td>
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<tr>
<td>70</td>
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<tr>
<td>Genie 2000 software validated by CANBERRA</td>
<td></td>
<td></td>
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<tr>
<td>71</td>
<td>yes</td>
<td>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 &lt; 10% and the accuracy (relative bias) is &lt; 2%. The minimum detection limit for 5 kg of water sample is 0.016 mBq/kg and the corresponding value for 2.5 g of water 32 mBq/kg.</td>
<td>A</td>
</tr>
<tr>
<td>72</td>
<td>yes</td>
<td>I could give the validation parameters for these matrices. 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 silicon-containing sample with a strongly oxidizing chemical treatment.</td>
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</tr>
<tr>
<td>73</td>
<td>yes</td>
<td>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%.</td>
<td>N</td>
</tr>
<tr>
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<td>Blank sample score</td>
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<td>76</td>
<td>yes</td>
<td>Detection limit (MDL): 0.005 bq/kg, Repeatability limit: 1.4 bq/kg for sample of 50 bq/kg radioactivity, at 95% confidence level. Reproducibility limit: No data</td>
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<td>77</td>
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<td>MDL 0.1 Bq/kg, reproducibility limit 10%</td>
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<td>Validation was performed as emergency method; MDL: 0.16 Bq/kg.</td>
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<td>1 pCi/l MDL, 20% precision limit, 25% bias limit</td>
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<td>Limit of detection (MDL) 0.05 Bq/kg, Repeatability 0.6%</td>
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<td>MDL = 6.225E-04 Bq/sample, LCS = Found-to-Added 98%</td>
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<td>MDL = 0.0089 Bq</td>
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<td>Water samples were spiked with a NIST traceable standard at 4.92, 11.5, 26.3, 42.6, 82.2, 392, and 755 mBq/L. The %recoveries ranged from 91% to 112%. The relative percent difference ranged from 3.5% to 12%. The typical MDL values for samples analyzed in this study ranged from 0.00477 to 0.09648 Bq/kg.</td>
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<td>Reported procedure for Method Detection Limit estimation in the participants laboratories</td>
<td>Blank sample score</td>
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<td>MDL 0.3 Bq/l</td>
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<tr>
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<td>The Currie detection limits ranged over 0.09 to 0.17 Bq/kg for these particular samples</td>
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<td>yes</td>
<td>The MDL for our procedure is less than 0.05 Bq/kg. The reproducibility parameter is 0.95.</td>
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<td>Minimum detection limit: 0.08 Bq/kg, Repeatability limit: smaller than 8%, Reproducibility limit: smaller than 10%</td>
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<td>MDL: 0.02. Use two different method to check reproductively.</td>
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<td>MDL is 0.05 Bq/kg</td>
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<td>106</td>
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<td>MDL=0.04 Bq/kg 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)</td>
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<td>Based on peer reviewed publication</td>
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<td>Minimum detection limit (MDL):12 counts/measuring sample</td>
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<td>Minimum detection limit: 0.5Bq/sample in about 24hours (95% confidence level)</td>
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<td>Repeatability: 5%. Reproducibility: 7%</td>
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<td>Minimum detection limit, Repeatability limit, Reproducibility limit.</td>
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<td>Reagent blank (triplicates) was spiked with the same amount of tracer (Po-209). MDA was determined to be 0.084 Bq/Kg.</td>
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<td>121</td>
<td>yes</td>
<td>The detection limit (MDL) for a 1 gram solid sample is 0.005Bq/g. Note, the detection limit will vary depending on the sample weight. Historically, the method has yielded a precision (relative standard deviation) of +/- 10%. The method has yielded an accuracy [Average Recovery (%) ± standard deviation (%)] of 99.0 ± 9.1</td>
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<td>MDL=0.007Bq/kg. Repeatability limit: $r=2.8*S$, #1: 2.7; #2: 7.6; #3: 3.9; #4: 8.0; #5: 0.093 Bq/kg, S was used as Standard deviation of 4 analyses.</td>
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<td>Blank sample score</td>
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<td>Repeated analysis on all samples. Note! reported values denote arithmetic means</td>
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<tr>
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<td>MDL: 3.00E-02 Bq/g. Accuracy : u=2.41. Precision: 6.41%</td>
<td>-</td>
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</table>
4.4. Measurement repeatability

The PT samples contained duplicate samples 01, 03 and 02, 04. The difference between the results of the duplicate samples was checked. 45 laboratories of 114 had a variation 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 insure 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.

4.5. Recommendations to the laboratories

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

a) Eighty six laboratories (Table 3, group A) reported results with a quality which 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.

b) Thirty five laboratories (Table 3, group B) should improve the repeatability and the reproducibility of their determinations and to find out the source of variations, is it from the plating or from inappropriate recovery correction or from other source. 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 the analytical procedure should be optimised to attain these targets.

c) Twenty one laboratory (Table 3, group C) 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 6 the procedure they applied in the estimation of MDL.

d) Seventeen laboratories (Table 3, group D) could not report acceptable results due to either significant bias or in few cases due to unstable method. These laboratories should revise their method and look for the root cause of bias or instability and perform method validation to check the reliability of the reported results.

5. CONCLUSIONS

The IAEA-CU-2007-09 world wide proficiency test for the determination of Po-210 in water was successfully conducted, 127 participants received the PT samples, and 88% 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 around 70% of the participants were able to report results which fit the purpose of rapid detection of Po-210 in water.
Fig. 6 and 7. Variations in % between the reported results for the duplicate samples 01, 03 and 02, 04.

However, although the matrix was a straightforward easy one and the activity concentrations were relatively high 19% of the reported results failed to pass the PT criteria. In few cases positive results were reported for the blank sample which suggests a possibility of false positive reporting.

The PT organiser proposed four categories of general recommendations to the participating laboratories to improve their analytical performance. However, if any participant needs any technical assistance to improve the analytical performance of Po-210 determination, Chemistry Unit at the Agency’s Seibersdorf laboratories will be glad to respond to such requests.

The PT results revealed the need for a harmonised analytical procedure for Po-210 rapid determination in case of emergency for high and low levels of activities. The procedure should also contain a standardized quality control protocol to assist the analyst in the validation of the reported results.
APPENDIX I. DATA EVALUATION TABLES SORTED BY SAMPLE CODE

All results listed in this Appendix are expressed in Bq/kg units at a reference date set to 2007-April-01. The abbreviations and calculation formulas used in the individual evaluation tables are explained in Section 3 of this report.

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

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.
Data evaluation of sample 01

Fig. I-01: S-shape chart of sample 01

Fig. I-02: z-score chart of sample 01
## Data evaluation of sample 01

**Target Value:** 52.8 ± 1.4 Bq/kg

### TABLE I-01: DATA EVALUATION OF SAMPLE 01

<table>
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<th>Lab code</th>
<th>Rep. Value</th>
<th>Rep Unc.</th>
<th>Unc. [%]</th>
<th>A1</th>
<th>A2</th>
<th>Trueness</th>
<th>P</th>
<th>Precision</th>
<th>Final Score</th>
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Data evaluation of sample 02

Fig. I-03: S-shape chart 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

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Data evaluation of sample 03

Fig. I-05: S-shape chart of sample 03.

Fig. I-06: z-score chart of sample 03.
### Data evaluation of sample 03

**Target Value:** 52.8 ± 1.4 Bq/kg

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Data evaluation of sample 04

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

Fig. I-08: z-score chart of sample 04.
**Data evaluation of sample 04**

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Data evaluation of sample 05, Blank

Fig. I-09: Graphical presentation of the ‘Blank’ sample results.
**Data evaluation of sample 05, Blank**

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APPENDIX II. DATA EVALUATION TABLES SORTED BY LABORATORY CODE

All results listed in this Appendix are expressed in Bq/kg units at a reference date set to 2007-April-01. The abbreviations and calculation formulas used in the individual evaluation tables are explained in Section 3 of this report.

The individual laboratory evaluation reports are presented in ascending order of the laboratory code.
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Laboratory No. 7

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</table>
## APPENDIX III. LIST OF PARTICIPATING LABORATORIES

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[3] SHAKHASHIRO, A; FAJGELJ, A; SANSONE, U; Comparison of Different Approaches to Evaluate Proficiency Test Data. Presented and accepted in the publications of the International Workshop on Combining and Reporting Analytical Results. The Role of (metrological) Traceability and (measurement) Uncertainty for Comparing Analytical Results, Rome 6-8 March, (2006).

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