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**Report on the IAEA-CU-2006-01 proficiency test on the determination of  
radionuclides and trace elements in soil and compost**

TC project: IAEA/RAS/2/011

“Quality Assurance and Quality Control of Nuclear Analytical Techniques”

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## Executive summary

This report summarises the results of a proficiency test conducted under the IAEA Technical Cooperation project RAS/2/011 “Quality Assurance and Control of Nuclear Analytical Techniques” (formerly RAW/2/005). The aim of project RAS/2/011 is to introduce and implement quality management systems for nuclear analytical techniques in Member State laboratories from West Asia. The proficiency test was addressed to assess the analytical performance of 19 laboratories for the determination of certain radionuclides and trace elements in soil and compost materials.

The test was organized and conducted by the Chemistry Unit of the IAEA's Laboratories located in Seibersdorf (Austria). The soil and compost materials were provided by the Italian Environmental Protection Agency (APAT). Compost is a common name for humus, which is the result of the decomposition of organic matter. Generally, compost is the raw material obtained by the aerobic decomposition of the organic residues of municipal waste or of vegetable market waste. Full technical details of both materials are reported in Appendix C.

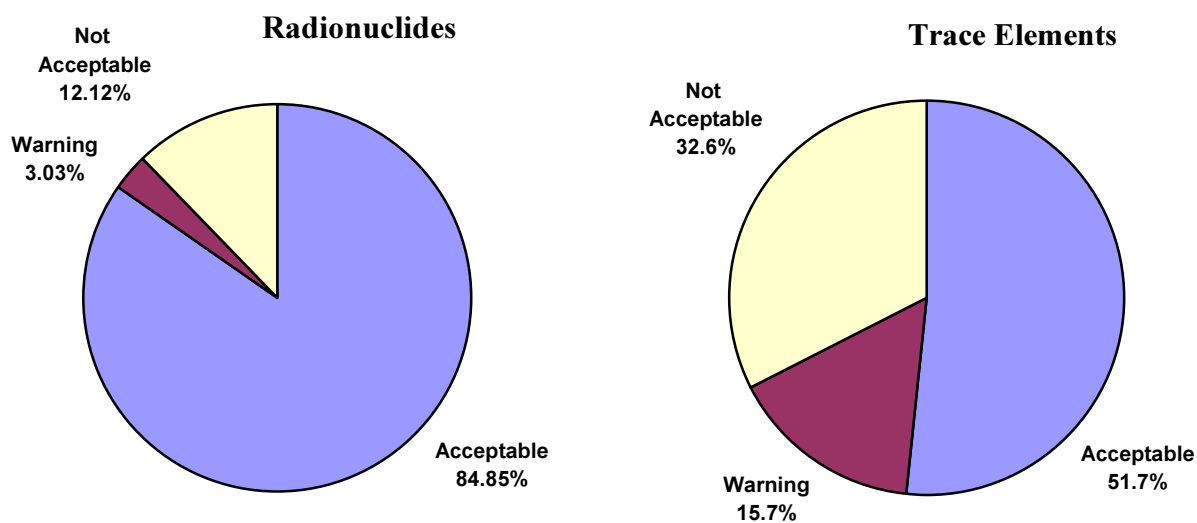
102 test samples (reference materials) were distributed to the participating laboratories in July 2005. The deadline for receiving the results from the participants was set to 15 December 2005. The participating laboratories were requested to analyse the samples employing the methods used in their routine work, so that their performance on the test samples could be directly related to the real performance of the laboratory. Each laboratory was given a confidential code to assure the anonymity of the evaluation results. 13 laboratories from the 19 initially registered reported to the IAEA their results. The analytical results of the participating laboratories were compared with the reference values assigned to the reference materials, and a rating system was applied.

In the case of the radionuclides, the analytical results were satisfactory for  $^{137}\text{Cs}$  and  $^{40}\text{K}$ , while the  $^{238}\text{U}$  analysis indicated the need for corrective actions in the analysis process. The analytical uncertainties associated with the results were, in general, appropriate for the analytes and matrices considered in the current proficiency test.

In the case of trace elements, the laboratory performances are satisfactory when the uncertainty of the participant measurement results is not taken into account for the evaluation of performance.

With the advent of “mutual recognition” on a world wide basis, it is now essential that laboratories participate in proficiency testing schemes that will provide an interpretation and assessment of results which is transparent to the participating laboratory and its “customer”. New requirements coming into force (ISO/IEC 17025:2005) require that laboratories have to express their measurement uncertainty. The subject of the evaluation of measurement uncertainty in analytical laboratories is of relevant interest, and although several guides have been published analytical scientists frequently regard the process as too theoretical and not suitable for the estimation of uncertainties of complex techniques. This is because identifying and quantifying all sources of uncertainty is difficult and laborious for procedures consisting of numerous steps, many of which are not clearly distinguishable. In addition, to find objective techniques for deciding how much uncertainty is acceptable in measurements intended for particular purposes is a difficult task. To this end it is recommended that a training effort be organised to help the laboratory staff to estimate measurement uncertainty on the analytical techniques used.

The following pictures report the analytical data evaluation of this proficiency test. 85% of the laboratories reported “acceptable” results for the radionuclides and 52% for the trace elements.



## Acknowledgement

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

We would like also to thank the following institutions, which contributed materials, time, know how and facilities for the preparation and characterization of the reference materials used in this proficiency test. Their contributions were at no cost to the IAEA.

- the Italian Environmental Protection Agency (APAT), Servizio Laboratori Misure ed Analisi di Campo, Roma, Italy;
- the Hungarian National Food Investigation Centre (NFII), Budapest, Hungary (IAEA collaborating centre);
- the Korea Institute of Nuclear Safety (KINS), Korea;
- the University of Roma Tre, Department of Physics, Roma, Italy.

## 1. Introduction

The results of analytical measurements play a vital role in our daily lives. Analytical data may be the basis upon which economic, legal or environmental management decisions are made, and they are essential in international trade, environmental protection, safe transportation, law enforcement, consumer safety and the preservation of human health. As an incorrect decision can be extremely costly and detrimental, it is essential that such measurements are accurate, reliable, cost effective and defensible. In addition, measurements performed by laboratories located worldwide should yield traceable and comparable results.

It is now widely recognised that for a laboratory to produce consistently reliable data it must implement an appropriate programme of quality assurance measures. Amongst such measures is the need for the laboratory to demonstrate that its analytical systems are under statistical control, that it uses methods of analysis that are validated, that its results are “fit-for-purpose”, and that it participates in proficiency testing exercises [1]. The competence of laboratories is demonstrated in accreditation processes following the ISO/IEC 17025:2005 [2] and in the frame of accreditation systems, the use of reference materials, both for quality control and proficiency testing, has therefore increased in recent years.

Proficiency testing is a method for regularly assessing the accuracy of the analytical data produced by the laboratories of particular measurements. In analytical chemistry, proficiency testing usually comprises the distribution of effectively homogenous portions of the test material to each participant for analysis as an unknown. The laboratories conduct the test under routine conditions, and report the result to the organiser by a deadline. The results generated in proficiency testing should be used for the purpose of a continuing assessment of the technical competence of the participating laboratories [1].

Since the 1960s the International Atomic Energy Agency (IAEA) has played an important role assisting laboratories in Member States to improve the quality of their analytical results and their traceability to basic standards. This is accomplished through the provision of matrix reference materials and validated procedures, training in the implementation of quality control, and the evaluation of measurement performance by the organization of proficiency tests and intercomparison exercises. The Chemistry Unit of the IAEA's Laboratories, located in the vicinity of the village of Seibersdorf (Lower Austria), about 35 km southeast of Vienna, at the premises of the Austrian Research Centre, is actively involved in the production and characterization of matrix reference materials of terrestrial origin, widely used for method and measurement validation and organization of proficiency tests and intercomparison exercises. The Chemistry Unit is a part of the Physics, Chemistry and Instrumentation Laboratory.

In the frame of the IAEA Technical Cooperation project RAS/2/011 “Quality Assurance and Control of Nuclear Analytical Techniques (formerly RAW/2/005)”, aimed to introduce and implement quality management systems for nuclear analytical techniques in Member State laboratories, in accordance with internationally accepted standards, a proficiency test was organized and conducted by the Chemistry Unit. The proficiency test was addressed to assess the analytical performance of 20 laboratories from West Asia, on the determination of certain radionuclides and trace elements in soil and compost materials.

## 2. Proficiency test objectives

Four distinct aims of proficiency test can be formulated:

- To check the trueness and precision of the analytical results produced by the participating laboratories for the determination of radionuclides and trace elements in soil and compost;
- to assist and encourage the participating laboratories in finding remedial actions where shortcoming in analytical performance are detected;
- to encourage the use of proper routine quality control measures within individual laboratories;
- to provide general evaluation and comment on the overall performance of participating laboratories; in order to enable the laboratories to compare their performances with those of other laboratories.

## 3. Proficiency test materials

In the planning-preparation phase of the proficiency test, the technical requirements regarding the type of matrices, the array and the concentration levels of analytes were proposed by the Technical Officer of the IAEA TC Project /RAS/2/011. The prime consideration in the choice of the material was that it should be as far as representative of the type of material that is normally analysed, in respect of composition of matrix and the concentration range of analytes. According to the users requirements it was agreed to use soil and compost as test samples with radionuclides ( $^{137}\text{Cs}$ ,  $^{40}\text{K}$  and  $^{238}\text{U}$ ) and trace elements with concentrations at environmental level. The agricultural soil and compost materials were provided by the Environmental Metrology Service of the Italian Environmental Protection Agency (APAT). It is known that compost is a common name for humus, which is the result of the decomposition of organic matter. Generally, compost is the raw material obtained by the aerobic decomposition of the organic residues of the municipal waste or of the vegetable market waste. Full technical details of both materials are reported in Appendix C. The experience gained in the earlier intercomparison exercises showed that two different types of matrices would allow to check if the methods perform equally well when applied to two different materials (matrix effect), eliminating thereby one of the important sources of bias in measurement process.

The following proficiency test design was applied:

- for trace elements analysis the test samples set consisted of 5 samples:
  - one soil reference material IAEA-375 to check the trueness of the participating laboratories results;
  - duplicate soil samples;
  - duplicate compost samples. The soil and compost samples were duplicated to evaluate the repeatability of analysis results.
- for gamma-spectrometric measurements the test set consisted of 4 samples:
  - one soil reference material IAEA-375,
  - duplicate soil samples;
  - one soil sample with low activity concentration.

The set of test samples distributed to the participating laboratories is shown in Table 1.



Sample code	Description	Mass (g)	Analytes
01	Soil IAEA-375	125	Radionuclides
02	Soil APAT-RM-05	250	
03	Soil IAEA - 401	250	
04	Soil APAT- RM-05-Duplicate	250	
01	Soil IAEA - 375	40	Trace elements
05	Compost APAT - RM-04	30	
06	Compost APAT - RM-04-Duplicate	30	
07	Soil APAT - RM-05	30	
08	Soil APAT - RM-05-Duplicate	30	

**Table 1**  
The sets of test samples

The participating laboratories were asked to choose which set of samples (or both) they are willing to analyse. They received the chosen set, together with handling instructions and the reporting forms. The deadline for data return was initially set to 30<sup>th</sup> November 2005 and then prolonged up to 15<sup>th</sup> of December 2005.

### ***3.1 Description of the test samples***

The compost and agricultural soil test samples (Compost APAT-RM-04 and Soil APAT-RM-05) were prepared, tested for homogeneity and characterised for trace elements by the Environmental Metrology Service of the Italian Environmental Protection Agency (APAT) [3]. Full technical details on the production and characterization of these materials are reported on Appendix C. According to the APAT report [3], the property values and associated total combined uncertainties were assigned by characterization in expert laboratories using aqua regia digestion method. Aqua regia is considered adequate for dissolving most base metal sulphates, sulphides, oxides and carbonates but only provides a “partial” digestion for most rock forming elements and elements of a refractory nature. For example, aqua regia digestion might give reliable results for the levels of polluting metals such as Cd, Cu, Pb and Zn while it is known to provide unsatisfactory results for metals like Cr, Ni and Ba which can only be efficiently recovered by using hydrofluoric acid (total digestion method) [4]. Considering that the current proficiency test (IAEA-CU-2006-01) was addressed to the determination of the total content of trace elements in soil and compost, the target values for the same test samples were also characterized by instrumental neutron activation analysis (INAA). This technique is immune from the potential problem of poor recovery due to incomplete digestion because it does not require sample dissolution. To this end the test samples 05 and 06 (Compost APAT-RM-04) were analyzed by INAA at the Italian National Metrological Institute (Istituto di Metrologia Gustavo Colonnetti) [5], while in the test samples 07 and 08 (Soil APAT-RM-05), the trace elements values were determined by INAA, at the Jožef Stefan Institute, Ljubljana, Slovenia [6]. In addition, the trace element values for both Compost APAT-RM-04 and Soil APAT-RM-05 reference materials were also confirmed with another non-destructive analytical technique (X ray fluorescence spectrometry) at the IAEA Instrumentation Unit, Seibersdorf, Austria [7] and at the Environmental Metrology Service of APAT [8].

The Soil APAT-RM-05 was characterised for radionuclides by the following additional three laboratories:

- the Hungarian National Food Investigation Centre (NFII), Budapest, Hungary (IAEA collaborating Centre);
- the Korea Institute of Nuclear Safety (KINS), Daejeon, Korea;
- the University of Roma Tre, Department of Physics, Roma, Italy.

The soil APAT-RM-05 was found also homogenous for the radionuclides of interest ( $^{137}\text{Cs}$ ,  $^{40}\text{K}$  and  $^{238}\text{U}$ ). Appendix C-2 contains the information on the radionuclides property values and their associated uncertainties.

### 3.2 Target values and uncertainties for trace elements

According to the certification report of the IAEA-375 reference material [9] the target values of As, and Ni in the test sample 01 are showed in table 2.

Analyte	Target value ( $\text{mg.kg}^{-1}$ ) dw
As	$2.56 \pm 0.32$
Ni	$9.7 \pm 1.85$

**Table 2**

Target values of the trace elements total concentrations  
in sample 01 (Soil IAEA-375)

The uncertainty is expressed as  $1 \sigma$  ( $k = 1$ )  
dw = based on dry weight

Considering that the current proficiency test (IAEA-CU-2006-01) was addressed to the determination of the total content of trace elements in soil and compost, only the target values determined by instrumental neutron activation analysis (INAA) were used for the evaluation of the analytical performance of the participant laboratories. As above reported, this technique is immune from the potential problem of poor recovery due to incomplete digestion because it does not require sample dissolution.

The target values for the test samples 05, 06, 07 and 08 are reported in tables 3 and 4.

<b>Analyte</b>	<b>(INAA) mg kg<sup>-1</sup> d.w.</b>
As	6.90 ± 0.09
Co	8.97 ± 0.15
Cr	505.0 ± 9.6
Mo	8.05 ± 0.40
Ni	248.1 ± 9.4
Se	0.61 ± 0.04
Zn	228.9 ± 14.6

**Table 3**

Target values of the trace elements concentrations  
in the test sample 05 and 06 (Compost APAT-RM-04)  
The uncertainty is expressed as 1  $\sigma$  (k=1)  
dw = based on dry weight

<b>Analyte</b>	<b>(INAA) mg kg<sup>-1</sup> d.w.</b>
As	11 ± 1
Cr	1030 ± 30
Fe	25570 ± 2827
Zn	91.8 ± 10.5

**Table 4**

Target values of the trace elements concentrations  
in the test samples 07 and 08 (Soil APAT-RM-05)  
Uncertainty is expressed as 1  $\sigma$  (k=1)  
dw = based on dry weight

Considering that in the trace element analysis, the acid digestion procedures applied to soil samples could represent a significant source of uncertainty in the final analytical data, the present document reports also on Appendix C, the property values of the trace elements concentrations and associated total combined uncertainties, assigned by characterization in expert laboratories using aqua regia

digestion method. This will permit the participating laboratories, which in the current proficiency test have used a “partial” digestion method, to make a self-evaluation (self-scoring) of their analytical performance.

### 3.3 Target values and uncertainties for radionuclides

The target values for the test samples 01, 02, 03 and 04 are reported in tables 5, 6 and 7.

*Reference date: 1- July -2005*

Analyte	Target value (Bq kg <sup>-1</sup> ) dw
<sup>40</sup> K	424 ± 8
<sup>137</sup> Cs	3850 ± 58
<sup>238</sup> U	24.4 ± 5.4

**Table 5**

Target values of the radionuclides in sample 01 (Soil IAEA-375) [9]

Analyte	Target value (Bq kg <sup>-1</sup> ) dw
<sup>40</sup> K	307 ± 17
<sup>137</sup> Cs	12.1 ± 0.47
<sup>238</sup> U	39.2 ± 1.09

**Table 6**

Target values of the radionuclides in sample 02 and 04  
(Soil APAT-RM-05)

Analyte	Target value (Bq kg <sup>-1</sup> ) dw
<sup>40</sup> K	716 ± 36
<sup>137</sup> Cs	2.6 ± 0.2

**Table 7**

Target values of the gamma emitting radionuclides in samples 03  
Uncertainty is expressed as 1 σ (k=1)  
dw = based on dry weight

#### **4. Analytical techniques used by the participating laboratories**

The participating laboratories were request 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 assess the real performance of the laboratory. Each laboratory was given a confidential code to assure the anonymity of the evaluation results. The technical information provided by the participants on the analytical procedures used in their own laboratory is compiled in Appendix E and coded with the same laboratory code used in data evaluation. The participants can benefit from the information exchange without revealing the laboratories identity.

#### **5. Performance criteria**

Currently most of laboratories produce test results accompanied, at best, with an indication of their repeatability only and provide no indication of their analytical uncertainty. However, new requirements coming into force (ISO/IEC 17025:2005) [2] require that laboratories have to express their measurement uncertainty.

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 uncertainty of the participant's measurement result is not taken into account for the evaluation of performance. In the case of U-scores, the evaluation includes uncertainties of the participant measurements and the uncertainty of the assigned value. Laboratories performing well in classical proficiency testing (Z-Scores) will not necessarily exhibit the same level of performance when their analytical uncertainties are considered in the evaluation.

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

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

##### **5.1 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:

$$\text{Relative bias} = \frac{\text{Value}_{\text{Analyst}} - \text{Value}_{\text{IAEA}}}{\text{Value}_{\text{IAEA}}} \times 100\% \quad (1)$$

## 5.2 The Z-score value

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

$$Z_{\text{Score}} = \frac{\text{Value}_{\text{Analyst}} - \text{Value}_{\text{IAEA}}}{\sigma} \quad (2)$$

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

$$0.10 \times \text{Value}_{\text{IAEA}}$$

The laboratory performance is evaluated as satisfactory if  $|z_{\text{Score}}| \leq 2$ ; questionable for  $2 < |z_{\text{Score}}| < 3$ , and unsatisfactory for  $|z_{\text{Score}}| \geq 3$ .

## 5.3 The U score value

The value of the  $U_{\text{test}}$  score calculated according to the following equation [10]

$$u_{\text{test}} = \frac{|\text{Value}_{\text{IAEA}} - \text{Value}_{\text{Analyst}}|}{\sqrt{\text{Unc}_{\text{IAEA}}^2 + \text{Unc}_{\text{Analyst}}^2}} \quad (3)$$

The calculated  $U_{\text{test}}$  value is compared with the critical values 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  $U_{\text{test}}$  that it takes into consideration the propagation of measurement uncertainties when defining the normalised error, this is especially useful when evaluating results, which 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 level of probability at 99% to determine if a result passes the test ( $u \leq 2.58$ ).

## 5.4 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.

## 5.5 Trueness

The participant result is assigned “Acceptable” status if:

$$A1 \leq A2$$

where:

$$A1 = |Value_{IAEA} - Value_{Analyst}|$$

$$A2 = 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Analyst}^2}$$

## 5.6 Precision

The participant result is assigned “Acceptable” status if:

$$P = \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Analyst}}{Value_{Analyst}}\right)^2} \times 100\%$$

The acceptance criterion for precision is dependent on the concentration or activity concentrations of the considered analytes. Applying the above reported equation the participant result is assigned “Acceptable” status if P is:

- $\leq 10\%$  for  $^{40}\text{K}$
- $\leq 10\%$  for  $^{137}\text{Cs}$
- $\leq 30\%$  for  $^{238}\text{U}$
- $\leq 22\%$  for As
- $\leq 10\%$  for Cr
- $\leq 22\%$  for Ni
- $\leq 10\%$  for Zn

A result must obtain “Acceptable” status in both criteria to be assigned final status of “Acceptable”. If a result obtained a “Not Acceptable” status for trueness or precision, then the relative bias is compared to a predetermined limit (20% for all analytes and 25% for  $^{238}\text{U}$ ), and if a result bias is below this limit then the status “Warning” is assigned as a final score, otherwise the status “Not Acceptable” is assigned as a final score. Obviously, if a result obtained “Not Acceptable” status for both trueness and precision the final score will be assigned as “Not Acceptable”.

## **6. Results and discussions**

### ***6.1 General***

13 laboratories from the 19 initially registered reported to the IAEA their results. Altogether 265 results were submitted. The participants' data along with the performance evaluation criteria and evaluation scores were compiled and presented in tables which constitute an integral part of this report. Performance evaluation for the radionuclides measurements is reported in Appendix A, while the performance evaluation for trace elements analysis is presented in Appendix B.

The performance evaluation results showed that the laboratories involved in radionuclides measurements had a higher final score than those related to trace elements determination. In the case of the radionuclides, the analytical results were satisfactory for  $^{137}\text{Cs}$  and  $^{40}\text{K}$ , while the  $^{238}\text{U}$  analysis indicated the need of corrective actions in the analysis process, to improve the quality of the results. The analytical uncertainties associated with the results were, in general appropriate, for the analytes and matrices considered in the current proficiency test. In the case of trace elements, the laboratory performances are satisfactory when the uncertainty of the participant measurement results is not taken into account for the evaluation of performance.

The different results observed between the radionuclides determination and trace elements analysis could be partially attributed to the fact that the trace element analysis presents more source of uncertainty in the final analytical data. The subject of the evaluation of measurement uncertainty in analytical laboratories is of relevant interest and although several guides have been published, analytical scientists frequently regard the estimation of uncertainty as too theoretical and not suitable for the estimation of uncertainties of complex techniques. This is because identifying and quantifying all sources of uncertainty is difficult and laborious, for procedures consisting of numerous steps, many of which are not clearly distinguishable. In addition, to find objective techniques for deciding how much uncertainty is acceptable in measurements intended for particular purposes is a difficult task. To this end it is recommended a training effort for the laboratory staff to estimate measurement uncertainty on the analytical techniques used.

### ***6.2 Recommendations to the participating laboratories***

The results submitted by the laboratories were evaluated against the reference values, the uncertainties claimed by the laboratories were revised and taken into consideration during the evaluation and when possible. Due to the limited technical information provided by the participants about the details of their analytical procedure, it was not possible to define the detailed root causes of discrepancies. Based on the results of this proficiency test, analysts could investigate their problems and take necessary remedial actions. Upon a request for assistance on a specific issue, the proficiency test organiser could give technical advice which might help in resolving remaining issues. Therefore, it is recommended, later on, to confirm whether the participating laboratories have resolved the problem through another proficiency test.



### ***6.2.1 Participating laboratories in trace elements analysis***

#### ***Laboratory 01***

The laboratory 01 sent results of the following 5 trace elements: Cr, Cu, Ni, Pb and Zn. Analysis were performed by flame AAS, digestion with HNO<sub>3</sub>, HF and HClO<sub>4</sub>. No method validation has been done. The uncertainty of the measurement is expressed as a standard deviation of three replicate measurements.

The laboratory results showed satisfactory performance for Cr, Ni and Zn, i.e., the digestion and the measurement procedure were performed in a systematic manner.

The measurement uncertainty was underestimated in Ni result for sample 06 and caused “Warning” status.

The Z-score evaluation was satisfactory for all analytes in all samples.

#### ***Laboratory 02***

The laboratory 02 submitted results of 6 trace elements: As, Cr, Cu, Hg, Ni and Zn. Analysis were performed by INAA. Method validation has been done for As and Cr and presented in Appendix E. The uncertainty of the measurement is expressed as combined uncertainty.

According to the reported individual results the method proves to have a satisfactory repeatability.

The negative bias in the compost and soil test samples was significant. In this case root cause should be investigated, .

According to Z-score, the laboratory obtained acceptable scores for As, Cr and Zn in the test samples.

#### ***Laboratory 03***

The laboratory 03 sent results of 5 trace elements: Cr, Cu, Ni, Pb and Zn. Analysis were performed by Total X-ray Fluorescence of digested samples in Teflon bomb with concentrated HNO<sub>3</sub>, HCl, and HF.

No method validation has been done. The uncertainty of the measurement is expressed as a standard deviation of three replicate measurements.

The analytical technique demonstrated a satisfactory repeatability.

In Z-score system the laboratory obtained satisfactory scores for all results.

#### ***Laboratory 04***

The laboratory 04 sent results of 5 trace elements: Cd, Cu, Ni, Pb and Zn. Analysis were performed by flame AAS. No method validation parameters were submitted. The uncertainty of the measurement is expressed as a standard deviation of three measurements.

The repeatability of the analytical technique was acceptable. Corrective actions should be implemented to correct the bias observed in the results.

#### ***Laboratory 05***

The laboratory 05 sent results of 6 trace elements: As, Cr, Cu, Ni, Pb and Zn. Analysis were performed by PIXE. Method validation parameters were submitted.

The laboratory had satisfactory results for As, Cr, Ni and Zn in samples 07 and 08. The As in samples 05, 06 obtained: "Warning" status due to measurement high uncertainty. Corrective actions to reduce the bias should be investigated.

#### ***Laboratory 06***

The laboratory 06 sent results of 5 trace elements: Cr, Cu, Ni, Pb and Zn. Analysis were performed by ICP-MS. Method validation parameters were submitted.

The laboratory had satisfactory results for Cr, Ni and Zn in samples 05 and 06. The validation data reported that Zn has a positive bias of 50% comparing to RM Soil-7, no correction was applied and consequently the Zn results obtained "Warning" status test due to underestimated uncertainty.

In Z-score system the laboratory performed satisfactory for all elements in samples 05 and 06 and Cr in samples 07 and 08.

#### ***Laboratory 07***

The laboratory 07 sent results of 18 trace elements, however due to the limitation of the availability of the target values of the test materials used in the proficiency test, only As, Cr, Ni, and Zn were evaluated. Analysis was performed by INAA. Method validation parameters were submitted on only Minimum detection limit.

The laboratory systematically overestimated the combined uncertainty up to 150%, which make the result of less quality. Corrective actions to reduce the bias and to report realistic uncertainty should be investigated and applied.

### ***6.2.2 Participating laboratories in radionuclides analysis***

#### ***General comment:***

Data evaluation of  $^{238}\text{U}$  results in this proficiency test shows that most of participants have not yet established the analytical capability of  $^{238}\text{U}$  using gamma spectrometry. Therefore, it is recommended to confirm whether the participating laboratories have the capability of analysis of uranium by alpha-spectrometry through other proficiency test.

#### ***Laboratory 08***

The laboratory 08 sent results of 3 radionuclides. Method validation parameters were submitted on only Minimum detection limit. Uncertainty sources were listed.

The laboratory demonstrated satisfactory performance for all analytes and for all samples, except for  $^{238}\text{U}$  in all samples, the results suffered of a bias up to 99%. Corrective actions to improve the accuracy of  $^{238}\text{U}$  should be investigated.

In Z-score system the laboratory performed satisfactory for  $^{40}\text{K}$  and  $^{137}\text{Cs}$  for all samples, but  $^{238}\text{U}$  failed the Z-Score.

#### ***Laboratory 09***

The laboratory 09 submitted results of 3 radionuclides. Information on Minimum detection limit was submitted. Uncertainty sources were listed.

The laboratory demonstrated satisfactory performance for  $^{40}\text{K}$  and  $^{137}\text{Cs}$  for all samples.  $^{238}\text{U}$  results in sample 01 were satisfactory, but in samples 02 and 04 had a bias up to 90%, Corrective actions to improve the accuracy of  $^{238}\text{U}$  should be implemented.

The laboratory 09 obtained acceptable Z-Score for all of the results except for  $^{238}\text{U}$  in samples 02 and 04.

### ***Laboratory 10***

The laboratory 10 sent results of 3 radionuclides. Method validation parameters were submitted on Minimum detection limit, repeatability limit, reproducibility limit and accuracy. Uncertainty sources were listed. Control charts were also submitted.

The laboratory demonstrated satisfactory performance for  $^{40}\text{K}$ ,  $^{137}\text{Cs}$  and  $^{238}\text{U}$  for all samples except  $^{238}\text{U}$  result in sample 01.

The laboratory obtained acceptable Z-score for all of the results in all samples.

### ***Laboratory 11***

The laboratory 11 sent results of 3 radionuclides. Method validation parameters were submitted on Minimum detection limit, repeatability limit, reproducibility limit and accuracy. Uncertainty sources were listed. Control charts were also submitted.

The laboratory demonstrated satisfactory performance for  $^{40}\text{K}$  and  $^{137}\text{Cs}$  for all samples.  $^{238}\text{U}$  results in sample 01 were satisfactory, but in samples 02 and 04 had a bias up to 70%. Corrective actions to improve the accuracy of  $^{238}\text{U}$  should be implemented.

The laboratory 11 obtained acceptable Z-score for all of the results except for  $^{238}\text{U}$  in samples 02 and 04.

### ***Laboratory 12***

The laboratory 12 sent results of 3 radionuclides. Method validation parameters were submitted on Minimum detection limit, repeatability limit, reproducibility limit and accuracy. Uncertainty sources were listed.

The laboratory demonstrated satisfactory performance for  $^{40}\text{K}$  and  $^{137}\text{Cs}$  for all samples.  $^{137}\text{Cs}$  result in sample 03 was rejected due to its high uncertainty (42%). This result would had been accepted if the uncertainty was estimated in the same way as for sample 01 (around 10%).  $^{238}\text{U}$  results in sample 01 were satisfactory, but in samples 02 a difference between samples 02 and 04 was observed up to 40%, although samples 02 and 04 are duplicate and should have the same value. The analyst should investigate the cause of discrepancy in the results of the duplicate test sample.

The laboratory 11 obtained acceptable Z-score for all of the results except for  $^{238}\text{U}$  in samples 02 and 04.

### ***Laboratory 13***

The laboratory 13 sent results of 3 radionuclides in triplicate. Method validation parameters were submitted on Minimum detection limit, repeatability limit, reproducibility limit and accuracy. Uncertainty sources were listed. Control charts were also submitted.

The laboratory presented a complete set of quality assurance documents which indicate that it applies and implements an effective quality assurance system.

The laboratory demonstrated satisfactory performance for  $^{40}\text{K}$ ,  $^{137}\text{Cs}$  and  $^{238}\text{U}$  for all samples.

The laboratory 13 obtained acceptable Z-score for all of the results in all samples.

## 7. Conclusions

In the frame of the IAEA Technical Cooperation project RAS/2/011 “Quality Assurance and Control of Nuclear Analytical Techniques (formerly RAW/2/005)”, aimed to introduce and implement quality management systems for nuclear analytical techniques in Member State laboratories, in accordance with internationally accepted standards, a proficiency test was organized and conducted by the Chemistry Unit of the IAEA's Seibersdorf Laboratories (Austria).

102 test samples were distributed by the Chemistry Unit to the participating laboratories in July 2005. The participating laboratories were request 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 assess the real performance of the laboratory. Each laboratory was given a confidential code to assure the anonymity of the evaluation results. 13 laboratories from the 19 initially registered reported to the IAEA their results. The analytical results of the participating laboratories were compared with the reference values assigned to the reference materials and a rating system was applied for determining the laboratories performance.

The analytical data evaluation of this proficiency test indicates that 85% of the laboratories reported “acceptable” results for the radionuclides and 52% for the trace elements.

In the case of radionuclides, the analytical results were satisfactory for  $^{137}\text{Cs}$  and  $^{40}\text{K}$ , while the  $^{238}\text{U}$  analysis indicated the need of corrective actions in the analysis process, to improve the quality of the results. The analytical uncertainties associated with the results were, in general appropriate, for the analytes and matrices considered in the current proficiency test.

Through this proficiency test it was found that many participants did not have a proper estimation of the uncertainty budget of their analytical results, which led to a “Warning” score in precision criteria. Failure in reporting well estimated combined uncertainty might lead to misinterpretation and false impression about the quality of the results, which consequently could mislead the decision maker who use these results. On the other hand, overestimation of the combined uncertainty might render the result of poor information due to very high and unrealistic claimed uncertainty. Therefore, training of laboratory staff in the field of uncertainty estimation is recommended. Furthermore, corrective actions should be applied to reduce the bias observed in the results and to improve the accuracy of the measurements.

It is worthy to note that proficiency testing has to be carried out within the context of an application of a complete system for quality assurance in each laboratory to provide a participant laboratory with an indication of problems if they are present, and it is clear that successful performance in a proficiency test for one analyte does not indicate that a laboratory is equally competent in determining an unrelated analyte.

## 8. References

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## **Appendix A: Performance evaluation of radionuclides analysis**

Target value:		424.0	[Bq/kg]	Data Evaluation of <sup>40</sup> K in soil, sample code 01									
Uncertainty:		3.00											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	434.00	29.70	6.8%	2.4%	0.24	0.33	1.02	10.00	77.02	Acceptable	6.9%	Acceptable	Acceptable
09	488.00	30.00	6.1%	15.1%	1.51	2.12	1.15	64.00	77.79	Acceptable	6.2%	Acceptable	Acceptable
10	413.10	11.40	2.8%	-2.6%	-0.26	-0.92	0.97	10.90	30.41	Acceptable	2.8%	Acceptable	Acceptable
11	444.56	23.65	5.3%	4.8%	0.48	0.86	1.05	20.56	61.51	Acceptable	5.4%	Acceptable	Acceptable
12	400.00	50.00	12.5%	-5.7%	-0.57	-0.48	0.94	24.00	129.23	Acceptable	12.5%	Acceptable	Acceptable
13	375.00	19.00	5.1%	-11.6%	-1.16	-2.55	0.88	49.00	49.63	Acceptable	5.1%	Acceptable	Acceptable

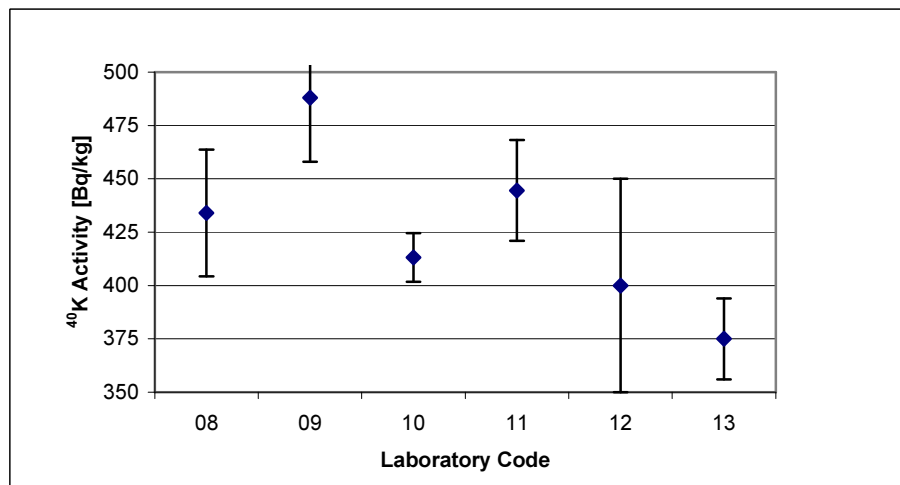


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$



Target value:		3850.0	[Bq/kg]	Data Evaluation of <sup>137</sup> Cs in soil, sample code 01									
Uncertainty:		72.00											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	[Bq/kg]					%	A1	A2	Score	P	
08	4035.00	55.00	1.4%	4.8%	0.48	2.04	1.05	185.00	233.76	Acceptable	2.3%	Acceptable	Acceptable
09	4406.00	232.00	5.3%	14.4%	1.44	2.29	1.14	556.00	626.72	Acceptable	5.6%	Acceptable	Acceptable
10	3675.20	35.20	1.0%	-4.5%	-0.45	-2.18	0.95	174.80	206.77	Acceptable	2.1%	Acceptable	Acceptable
11	3936.50	145.05	3.7%	2.2%	0.22	0.53	1.02	86.50	417.80	Acceptable	4.1%	Acceptable	Acceptable
12	3600.00	360.00	10.0%	-6.5%	-0.65	-0.68	0.94	250.00	947.19	Acceptable	10.2%	Acceptable	Acceptable
13	3771.00	120.00	3.2%	-2.1%	-0.21	-0.56	0.98	79.00	361.05	Acceptable	3.7%	Acceptable	Acceptable

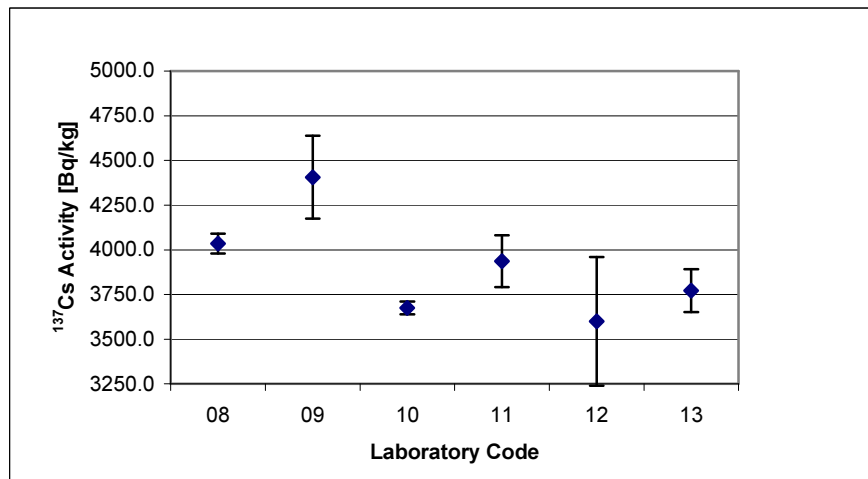


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		24.4	[Bq/kg]	Data Evaluation of <sup>238</sup> U in soil, sample code 01									
Uncertainty:		5.40											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	0.20	0.10	50.0%	-99.2%	-9.92	-4.48	0.01	24.20	13.93	Not Acceptable	54.7%	Not Acceptable	Not Acceptable
09	22.78	2.11	9.3%	-6.6%	-0.66	-0.28	0.93	1.62	14.96	Acceptable	24.0%	Acceptable	Acceptable
10	16.50	4.10	24.8%	-32.4%	-3.24	-1.17	0.68	7.90	17.49	Acceptable	33.3%	Not Acceptable	Not Acceptable
11	19.01	2.64	13.9%	-22.1%	-2.21	-0.90	0.78	5.39	15.51	Acceptable	26.1%	Acceptable	Acceptable
12	20.00	3.00	15.0%	-18.0%	-1.80	-0.71	0.82	4.40	15.94	Acceptable	26.7%	Acceptable	Acceptable
13	34.40	6.90	20.1%	41.0%	4.10	1.14	1.41	10.00	22.61	Acceptable	29.9%	Acceptable	Acceptable

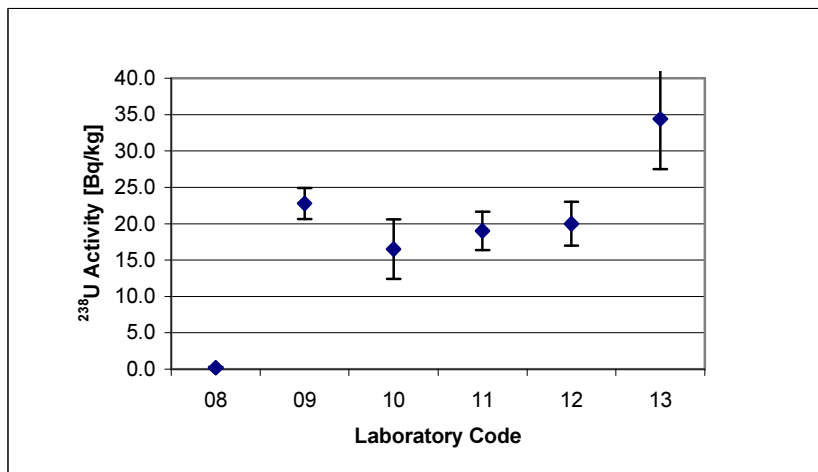


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		307.0	[Bq/kg]	Data Evaluation of <sup>40</sup> K in agricultural soil, sample code 02										
Uncertainty:		17.00												
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score	
	Value [Bq/kg]	Unc.						Trueness			Precision			
		[Bq/kg]	%					A1	A2	Score	P	Score		
08	325.42	17.25	5.3%	6.0%	0.60	0.76	1.06	18.42	62.49	Acceptable	7.7%	Acceptable	Acceptable	
09	345.00	16.00	4.6%	12.4%	1.24	1.63	1.12	38.00	60.23	Acceptable	7.2%	Acceptable	Acceptable	
10	308.60	7.20	2.3%	0.5%	0.05	0.09	1.01	1.60	47.63	Acceptable	6.0%	Acceptable	Acceptable	
11	333.05	19.61	5.9%	8.5%	0.85	1.00	1.08	26.05	66.96	Acceptable	8.1%	Acceptable	Acceptable	
12	290.00	30.00	10.3%	-5.5%	-0.55	-0.49	0.94	17.00	88.96	Acceptable	11.7%	Acceptable	Acceptable	
13	295.00	16.00	5.4%	-3.9%	-0.39	-0.51	0.96	12.00	60.23	Acceptable	7.8%	Acceptable	Acceptable	

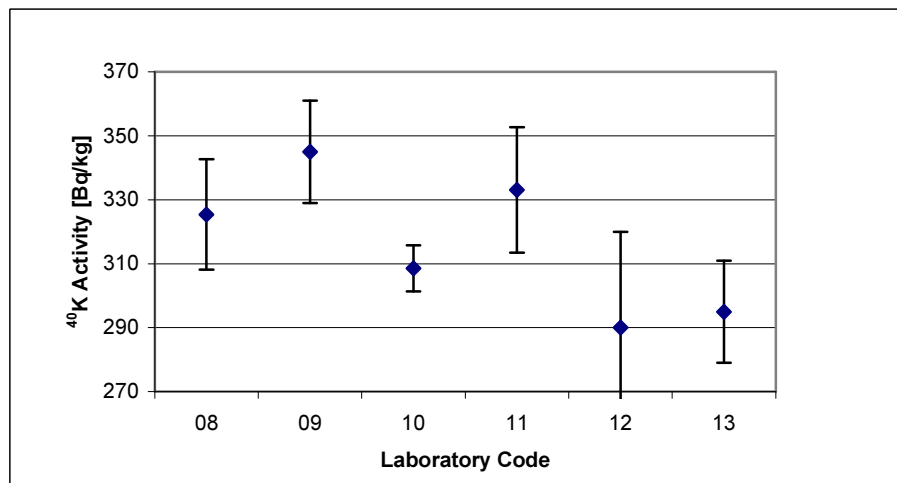


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		12.1	[Bq/kg]	Data Evaluation of <sup>137</sup> Cs in agricultural soil, sample code 02									
Uncertainty:		0.47											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	12.81	0.60	4.7%	5.9%	0.59	0.93	1.06	0.71	1.97	Acceptable	6.1%	Acceptable	Acceptable
09	13.02	0.75	5.8%	7.6%	0.76	1.04	1.08	0.92	2.28	Acceptable	6.9%	Acceptable	Acceptable
10	12.10	0.50	4.1%	0.0%	0.00	0.00	1.00	0.00	1.77	Acceptable	5.7%	Acceptable	Acceptable
11	13.84	0.93	6.7%	14.4%	1.44	1.67	1.14	1.74	2.69	Acceptable	7.8%	Acceptable	Acceptable
12	13.00	2.00	15.4%	7.4%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable
13	13.00	2.00	15.4%	7.4%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable

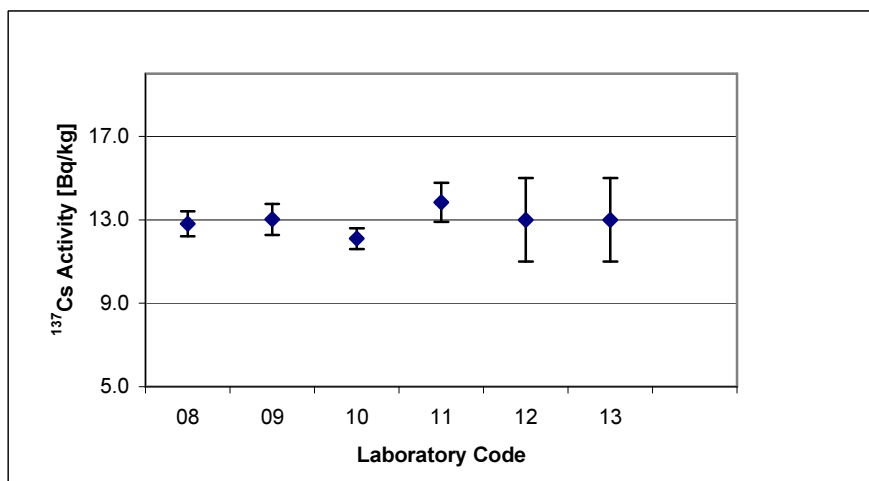


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		39.2	[Bq/kg]	Data Evaluation of <sup>238</sup> U in agricultural soil, sample code 02									
Uncertainty:		3.90											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	83.58	7.00	8.4%	113.2%	11.32	5.54	2.13	44.38	20.67	Not Acceptable	13.0%	Acceptable	Not Acceptable
09	66.68	2.81	4.2%	70.1%	7.01	5.72	1.70	27.48	12.40	Not Acceptable	10.8%	Acceptable	Not Acceptable
10	38.50	3.10	8.1%	-1.8%	-0.18	-0.14	0.98	0.70	12.85	Acceptable	12.8%	Acceptable	Acceptable
11	60.96	2.49	4.1%	55.5%	5.55	4.70	1.56	21.76	11.94	Not Acceptable	10.8%	Acceptable	Not Acceptable
12	30.00	12.00	40.0%	-23.5%	-2.35	-0.73	0.77	9.20	32.55	Acceptable	41.2%	Not Acceptable	Warning
13	22.70	5.30	23.3%	-42.1%	-4.21	-2.51	0.58	16.50	16.98	Acceptable	25.4%	Acceptable	Acceptable

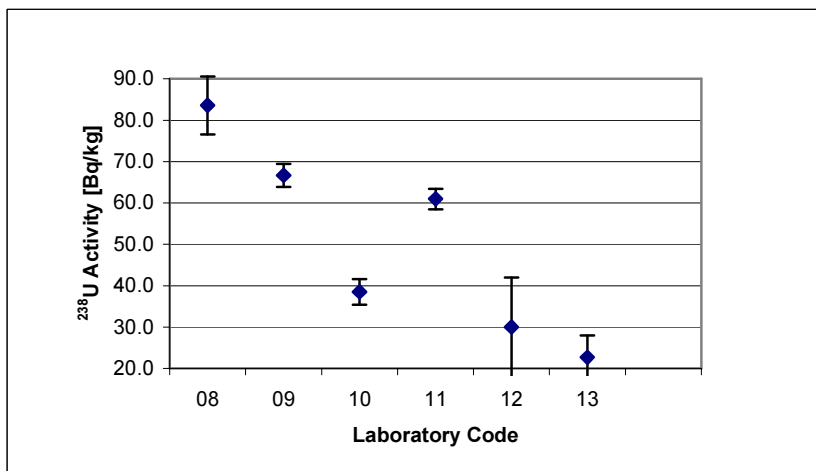


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		716.0	[Bq/kg]	Data Evaluation of <sup>40</sup> K in soil, sample code 03										
Uncertainty:		36.00												
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score	
	Value [Bq/kg]	Unc.						Trueness			Precision			
		[Bq/kg]	%					A1	A2	Score	P	Score		
08	742.22	37.20	5.0%	3.7%	0.37	0.51	1.04	26.22	133.56	Acceptable	7.1%	Acceptable	Acceptable	
09	761.00	31.00	4.1%	6.3%	0.63	0.95	1.06	45.00	122.57	Acceptable	6.5%	Acceptable	Acceptable	
10	703.90	13.30	1.9%	-1.7%	-0.17	-0.32	0.98	12.10	99.02	Acceptable	5.4%	Acceptable	Acceptable	
11	774.56	35.93	4.6%	8.2%	0.82	1.15	1.08	58.56	131.22	Acceptable	6.8%	Acceptable	Acceptable	
12	680.00	70.00	10.3%	-5.0%	-0.50	-0.46	0.95	36.00	203.08	Acceptable	11.5%	Acceptable	Acceptable	
13	683.00	32.00	4.7%	-4.6%	-0.46	-0.69	0.95	33.00	124.27	Acceptable	6.9%	Acceptable	Acceptable	

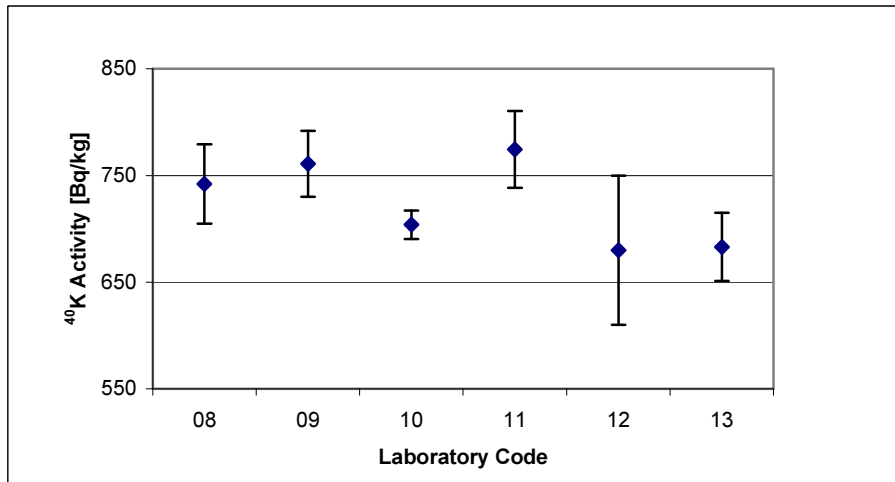


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$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		2.6	[Bq/kg]	Data Evaluation of <sup>137</sup> Cs in soil, sample code 03									
Uncertainty:		0.20											
Laboratory Code	Laboratories Results							Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	2.93	0.30	10.2%	12.7%	1.27	0.92	1.13	0.33	0.93	Acceptable	12.8%	Acceptable	Acceptable
09	2.95	0.29	9.8%	13.5%	1.35	0.99	1.13	0.35	0.91	Acceptable	12.5%	Acceptable	Acceptable
10	2.80	0.30	10.7%	7.7%	0.77	0.55	1.08	0.20	0.93	Acceptable	13.2%	Acceptable	Acceptable
11	3.73	0.54	14.5%	43.5%	4.35	1.96	1.43	1.13	1.49	Acceptable	16.4%	Acceptable	Acceptable
12	2.10	0.90	42.9%	-19.2%	-1.92	-0.54	0.81	0.50	2.38	Acceptable	43.5%	Not Acceptable	Warning
13	3.10	0.34	11.0%	19.2%	1.92	1.27	1.19	0.50	1.02	Acceptable	13.4%	Acceptable	Acceptable

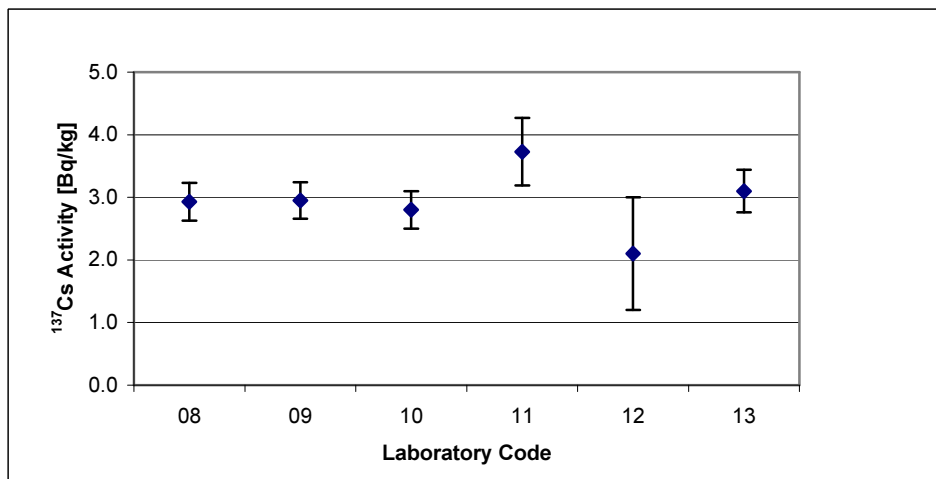


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		307.0	[Bq/kg]	Data Evaluation of <sup>40</sup> K in agricultural soil, sample code 04									
Uncertainty:		17.00											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [Bq/kg]	Unc.						Trueness			Precision		
		[Bq/kg]	%					A1	A2	Score	P	Score	
08	320.30	17.60	5.5%	4.3%	0.43	0.54	1.04	13.30	63.13	Acceptable	7.8%	Acceptable	Acceptable
09	322.00	16.00	5.0%	4.9%	0.49	0.64	1.05	15.00	60.23	Acceptable	7.4%	Acceptable	Acceptable
10	312.70	7.00	2.2%	1.9%	0.19	0.31	1.02	5.70	47.43	Acceptable	6.0%	Acceptable	Acceptable
11	367.80	20.64	5.6%	19.8%	1.98	2.27	1.20	60.80	68.99	Acceptable	7.9%	Acceptable	Acceptable
12	300.00	30.00	10.0%	-2.3%	-0.23	-0.20	0.98	7.00	88.96	Acceptable	11.4%	Acceptable	Acceptable
13	297.00	16.00	5.4%	-3.3%	-0.33	-0.43	0.97	10.00	60.23	Acceptable	7.7%	Acceptable	Acceptable

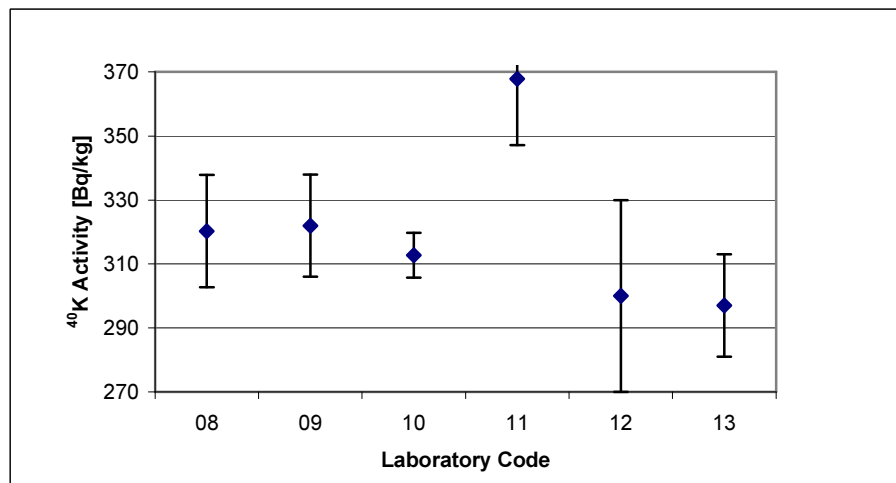


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$



Target value:		12.1	[Bq/kg]	Data Evaluation of <sup>137</sup> Cs in agricultural soil, sample code 04										
Uncertainty:		0.47												
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score	
	Value [Bq/kg]	Unc.						Trueness			Precision			
		[Bq/kg]	%					A1	A2	Score	P	Score		
08	12.75	0.60	4.7%	5.4%	0.54	0.85	1.05	0.65	1.97	Acceptable	6.1%	Acceptable	Acceptable	
09	13.54	0.75	5.5%	11.9%	1.19	1.63	1.12	1.44	2.28	Acceptable	6.8%	Acceptable	Acceptable	
10	11.90	0.30	2.5%	-1.7%	-0.17	-0.36	0.98	0.20	1.44	Acceptable	4.6%	Acceptable	Acceptable	
11	13.86	0.93	6.7%	14.5%	1.45	1.69	1.15	1.76	2.69	Acceptable	7.8%	Acceptable	Acceptable	
12	13.00	2.00	15.4%	7.4%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable	
13	13.90	0.80	5.8%	14.9%	1.49	1.94	1.15	1.80	2.39	Acceptable	6.9%	Acceptable	Acceptable	

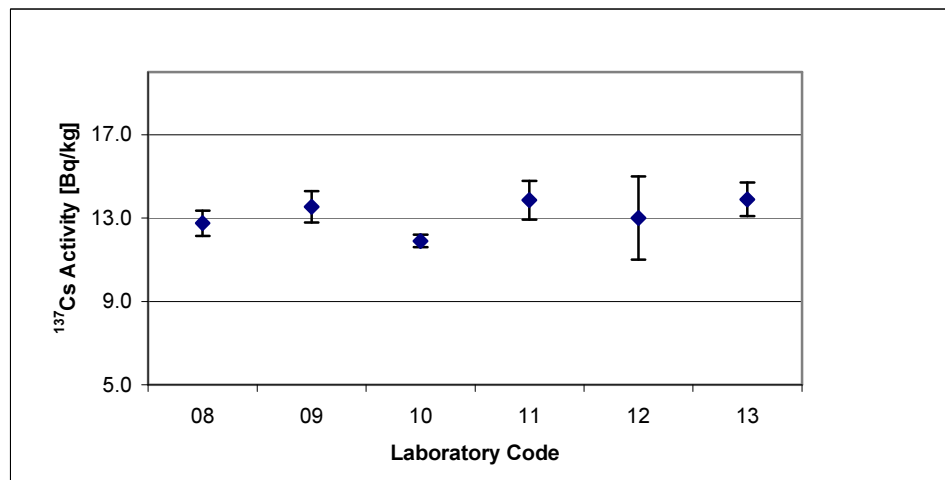


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		39.2	[Bq/kg]	Data Evaluation of <sup>238</sup> U in agricultural soil, sample code 04									
Uncertainty:		3.90		Laboratories Results				Acceptance criteria					Final Score
Laboratory Code	Value	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
08	77.20	6.70	8.7%	96.9%	9.69	4.90	1.97	38.00	20.00	Not Acceptable	13.2%	Acceptable	Not Acceptable
09	65.87	3.78	5.7%	68.0%	6.80	4.91	1.68	26.67	14.01	Not Acceptable	11.5%	Acceptable	Not Acceptable
10	30.80	2.70	8.8%	-21.4%	-2.14	-1.77	0.79	8.40	12.24	Acceptable	13.3%	Acceptable	Acceptable
11	58.82	2.38	4.0%	50.1%	5.01	4.29	1.50	19.62	11.79	Not Acceptable	10.7%	Acceptable	Not Acceptable
12	43.00	12.00	27.9%	9.7%	0.97	0.30	1.10	3.80	32.55	Acceptable	29.6%	Acceptable	Acceptable
13	24.70	7.10	28.7%	-37.0%	-3.70	-1.79	0.63	14.50	20.90	Acceptable	30.4%	Acceptable	Acceptable

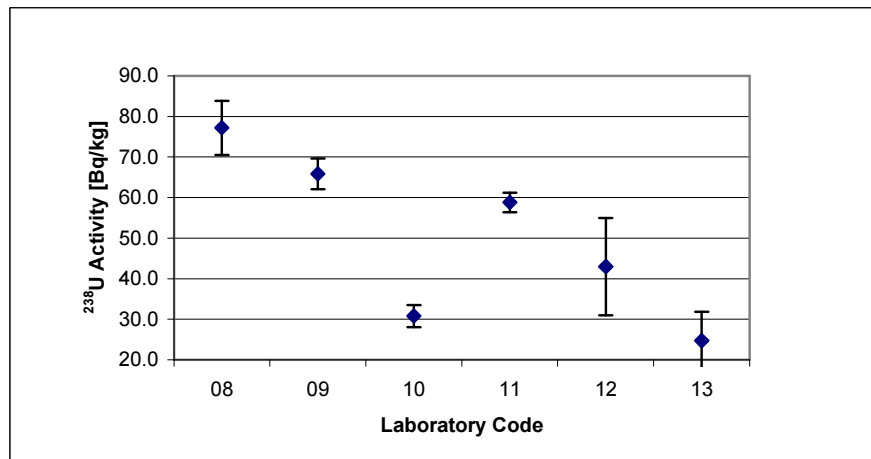


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

## Analytical Performance Evaluation of Laboratory 08

**Sample code 01**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	434.00	29.70	6.8%	2%	0.24	0.33	1.02	10.00	77.02	Acceptable	6.9%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	4035.00	55.00	1.4%	5%	0.48	2.04	1.05	185.00	233.76	Acceptable	2.3%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	0.20	0.10	50.0%	-99%	-9.92	-4.48	0.01	24.20	13.93	Not Acceptable	54.7%	Not Acceptable	Not Acceptable

**Sample code 03**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	742.22	37.20	5.0%	4%	0.37	0.51	1.04	26.22	133.56	Acceptable	7.1%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	2.93	0.30	10.2%	13%	1.27	0.92	1.13	0.33	0.93	Acceptable	12.8%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 08

### Sample code 02

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	325.42	17.25	5.3%	6%	0.60	0.76	1.06	18.42	62.49	Acceptable	7.7%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	12.81	0.60	4.7%	6%	0.59	0.93	1.06	0.71	1.97	Acceptable	6.1%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	83.58	7.00	8.4%	113%	11.32	5.54	2.13	44.38	20.67	Not Acceptable	13.0%	Acceptable	Not Acceptable

### Sample code 04

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	320.30	17.60	5.5%	4%	0.43	0.54	1.04	13.30	63.13	Acceptable	7.8%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	12.75	0.60	4.7%	5%	0.54	0.85	1.05	0.65	1.97	Acceptable	6.1%	Acceptable	Acceptable
<sup>238</sup> U	39.2	5.37	77.20	6.70	8.7%	97%	9.69	4.43	1.97	38.00	22.15	Not Acceptable	16.2%	Acceptable	Not Acceptable

## Analytical Performance Evaluation of Laboratory 09

**Sample code 01**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	488.00	30.00	6.1%	15%	1.51	2.12	1.15	64.00	77.79	Acceptable	6.2%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	4406.00	232.00	5.3%	14%	1.44	2.29	1.14	556.00	626.72	Acceptable	5.6%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	22.78	2.11	9.3%	-7%	-0.66	-0.28	0.93	1.62	14.96	Acceptable	24.0%	Acceptable	Acceptable

**Sample code 03**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	761.00	31.00	4.1%	6%	0.63	0.95	1.06	45.00	122.57	Acceptable	6.5%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	2.95	0.29	9.8%	13%	1.35	0.99	1.13	0.35	0.91	Acceptable	12.5%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 09

Sample code 02

Reference date: 1 - 07- 2005

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	345.00	16.00	4.6%	12%	1.24	1.63	1.12	38.00	60.23	Acceptable	7.2%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.02	0.75	5.8%	8%	0.76	1.04	1.08	0.92	2.28	Acceptable	6.9%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	66.68	2.81	4.2%	70%	7.01	5.72	1.70	27.48	12.40	Not Acceptable	10.8%	Acceptable	Not Acceptable

Sample code 04

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	322.00	16.00	5.0%	5%	0.49	0.64	1.05	15.00	60.23	Acceptable	7.4%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.54	0.75	5.5%	12%	1.19	1.63	1.12	1.44	2.28	Acceptable	6.8%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	65.87	3.78	5.7%	68%	6.80	4.91	1.68	26.67	14.01	Not Acceptable	11.5%	Acceptable	Not Acceptable

## Analytical Performance Evaluation of Laboratory 10

**Sample code 01**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	413.10	11.40	2.8%	-3%	-0.26	-0.92	0.97	10.90	30.41	Acceptable	2.8%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	3675.20	35.20	1.0%	-5%	-0.45	-2.18	0.95	174.80	206.77	Acceptable	2.1%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	16.50	4.10	24.8%	-32%	-3.24	-1.17	0.68	7.90	17.49	Acceptable	33.3%	Not Acceptable	Not Acceptable

**Sample code 03**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[Bq/kg]	[Bq/kg]	[Bq/kg]	[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	703.90	13.30	1.9%	-2%	-0.17	-0.32	0.98	12.10	99.02	Acceptable	5.4%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	2.80	0.30	10.7%	8%	0.77	0.55	1.08	0.20	0.93	Acceptable	13.2%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 10

**Sample code 02**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	308.60	7.20	2.3%	1%	0.05	0.09	1.01	1.60	47.63	Acceptable	6.0%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	12.10	0.50	4.1%	0%	0.00	0.00	1.00	0.00	1.77	Acceptable	5.7%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	38.50	3.10	8.1%	-2%	-0.18	-0.14	0.98	0.70	12.85	Acceptable	12.8%	Acceptable	Acceptable

**Sample code 04**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	312.70	7.00	2.2%	2%	0.19	0.31	1.02	5.70	47.43	Acceptable	6.0%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	11.90	0.30	2.5%	-2%	-0.17	-0.36	0.98	0.20	1.44	Acceptable	4.6%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	30.80	2.70	8.8%	-21%	-2.14	-1.77	0.79	8.40	12.24	Acceptable	13.3%	Acceptable	Acceptable



## Analytical Performance Evaluation of Laboratory 11

Sample code 01

Reference date: 1 - 07- 2005

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	444.56	23.65	5.3%	5%	0.48	0.86	1.05	20.56	61.51	Acceptable	5.4%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	3936.50	145.05	3.7%	2%	0.22	0.53	1.02	86.50	417.80	Acceptable	4.1%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	19.01	2.64	13.9%	-22%	-2.21	-0.90	0.78	5.39	15.51	Acceptable	26.1%	Acceptable	Acceptable

Sample code 03

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	774.56	35.93	4.6%	8%	0.82	1.15	1.08	58.56	131.22	Acceptable	6.8%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	3.73	0.54	14.5%	43%	4.35	1.96	1.43	1.13	1.49	Acceptable	16.4%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 11

**Sample code 02**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	333.05	19.61	5.9%	8%	0.85	1.00	1.08	26.05	66.96	Acceptable	8.1%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.84	0.93	6.7%	14%	1.44	1.67	1.14	1.74	2.69	Acceptable	7.8%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	60.96	2.49	4.1%	56%	5.55	4.70	1.56	21.76	11.94	Not Acceptable	10.8%	Acceptable	Not Acceptable

**Sample code 04**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	367.80	20.64	5.6%	20%	1.98	2.27	1.20	60.80	68.99	Acceptable	7.9%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.86	0.93	6.7%	15%	1.45	1.69	1.15	1.76	2.69	Acceptable	7.8%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	58.82	2.38	4.0%	50%	5.01	4.29	1.50	19.62	11.79	Not Acceptable	10.7%	Acceptable	Not Acceptable

## Analytical Performance Evaluation of Laboratory 12

**Sample code 01**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	400.00	50.00	12.5%	-6%	-0.57	-0.48	0.94	24.00	129.23	Acceptable	12.5%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	3600.00	360.00	10.0%	-6%	-0.65	-0.68	0.94	250.00	947.19	Acceptable	10.2%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	20.00	3.00	15.0%	-18%	-1.80	-0.71	0.82	4.40	15.94	Acceptable	26.7%	Acceptable	Acceptable

**Sample code 03**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	680.00	70.00	10.3%	-5%	-0.50	-0.46	0.95	36.00	203.08	Acceptable	11.5%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	2.10	0.90	42.9%	-19%	-1.92	-0.54	0.81	0.50	2.38	Acceptable	43.5%	Not Acceptable	Warning

## Analytical Performance Evaluation of Laboratory 12

**Sample code 02**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	290.00	30.00	10.3%	-6%	-0.55	-0.49	0.94	17.00	88.96	Acceptable	11.7%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.00	2.00	15.4%	7%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	30.00	12.00	40.0%	-23%	-2.35	-0.73	0.77	9.20	32.55	Acceptable	41.2%	Not Acceptable	Warning

**Sample code 04**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	300.00	30.00	10.0%	-2%	-0.23	-0.20	0.98	7.00	88.96	Acceptable	11.4%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.00	2.00	15.4%	7%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	43.00	12.00	27.9%	10%	0.97	0.30	1.10	3.80	32.55	Acceptable	29.6%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 13

**Sample code 01**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	424.00	3.00	375.00	19.00	5.1%	-12%	-1.16	-2.55	0.88	49.00	49.63	Acceptable	5.1%	Acceptable	Acceptable
<sup>137</sup> Cs	3850.0	72.00	3771.00	120.00	3.2%	-2%	-0.21	-0.56	0.98	79.00	361.05	Acceptable	3.7%	Acceptable	Acceptable
<sup>238</sup> U	24.4	5.40	34.40	6.90	20.1%	41%	4.10	1.14	1.41	10.00	22.61	Acceptable	29.9%	Acceptable	Acceptable

**Sample code 03**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	716.00	36.00	683.00	32.00	4.7%	-5%	-0.46	-0.69	0.95	33.00	124.27	Acceptable	6.9%	Acceptable	Acceptable
<sup>137</sup> Cs	2.6	0.20	3.10	0.34	11.0%	19%	1.92	1.27	1.19	0.50	1.02	Acceptable	13.4%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 13

**Sample code 02**

*Reference date: 1 - 07- 2005*

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	295.00	16.00	5.4%	-4%	-0.39	-0.51	0.96	12.00	60.23	Acceptable	7.8%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.00	2.00	15.4%	7%	0.74	0.44	1.07	0.90	5.30	Acceptable	15.9%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	22.70	5.30	23.3%	-42%	-4.21	-2.51	0.58	16.50	16.98	Acceptable	25.4%	Acceptable	Acceptable

**Sample code 04**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [Bq/kg]	Unc. [Bq/kg]	Value [Bq/kg]	Unc.						Trueness			Precision		
				[Bq/kg]	%					A1	A2	Score	P	Score	
<sup>40</sup> K	307.00	17.00	297.00	16.00	5.4%	-3%	-0.33	-0.43	0.97	10.00	60.23	Acceptable	7.7%	Acceptable	Acceptable
<sup>137</sup> Cs	12.1	0.47	13.90	0.80	5.8%	15%	1.49	1.94	1.15	1.80	2.39	Acceptable	6.9%	Acceptable	Acceptable
<sup>238</sup> U	39.2	3.90	24.70	7.10	28.7%	-37%	-3.70	-1.79	0.63	14.50	20.90	Acceptable	30.4%	Acceptable	Acceptable

## **Appendix B: Performance evaluation of trace elements analysis**

<b>Target value:</b>		2.56	[mg/kg]	<b>Data evaluation of As in soil, sample code 01</b>										
<b>Uncertainty:</b>		0.32												
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score	
	Value [mg/kg]	Unc.						Trueness			Precision			
		[mg/kg]	%					A1	A2	Score	P	Score		
02	2.23	0.01	0.4%	-12.9%	-1.29	-1.03	0.87	0.330	0.826	acceptable	12.5%	Acceptable	Acceptable	
05	4.30	1.20	27.9%	68.0%	6.80	1.40	1.68	1.74	3.20	acceptable	30.6%	Not Acceptable	Not Acceptable	
07	2.34	3.71	158.5%	-8.6%	-0.86	-0.06	0.91	0.22	9.61	acceptable	159%	Not Acceptable	Warning	

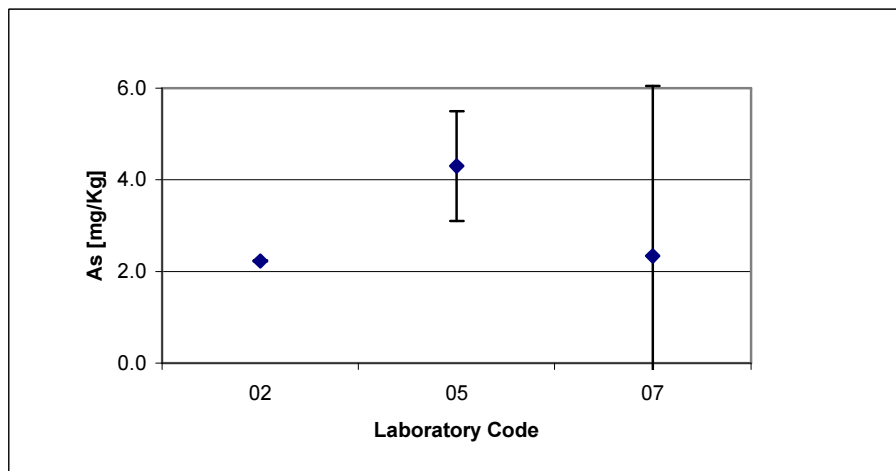


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$



Target value:		9.7	[mg/kg]	Data evaluation of Ni in soil, sample code 01									
Uncertainty:		1.85											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	12.25	0.42	3.4%	26.3%	2.63	1.34	1.26	2.55	4.89	Acceptable	19.4%	Acceptable	Acceptable
02	7.80	0.80	10.3%	-19.6%	-1.96	-0.94	0.80	1.90	5.20	Acceptable	21.7%	Not Acceptable	Warning
03	12.76	1.61	12.6%	31.5%	3.15	1.25	1.32	3.06	6.33	Acceptable	22.9%	Not Acceptable	Not Acceptable
04	356.70	3.50	1.0%	3577.3%	357.73	87.65	36.77	347.00	10.21	Not Acceptable	19.1%	Acceptable	Not Acceptable
05	14.37	3.00	20.9%	48.1%	4.81	1.32	1.48	4.67	9.09	Acceptable	28.3%	Not Acceptable	Not Acceptable
06	9.05	1.06	11.7%	-6.7%	-0.67	-0.30	0.93	0.65	5.50	Acceptable	22.4%	Not Acceptable	Warning
07	<50												Acceptable

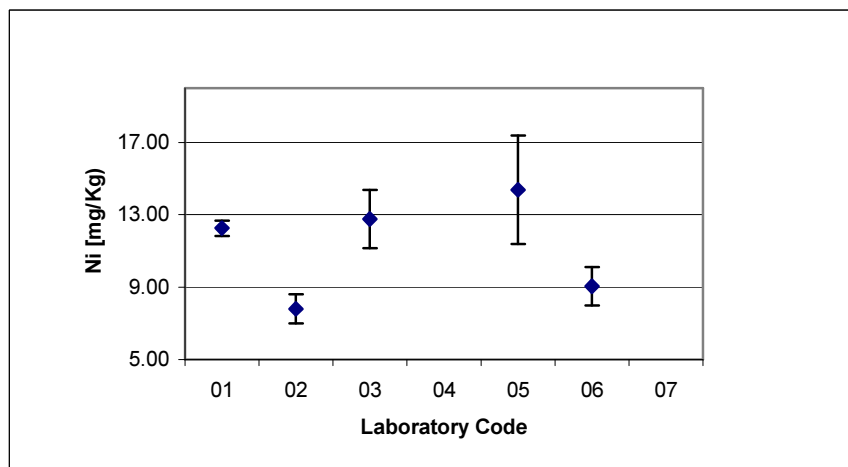


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		6.90	[mg/kg]	Data evaluation of As in compost, sample code 05									
Uncertainty:		0.09											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
02	6.100	0.01	0.2%	-11.6%	-1.16	-8.83	0.88	0.800	0.234	Not Acceptable	1.3%	Acceptable	Warning
05	9.30	2.90	31.2%	34.8%	3.48	0.83	1.35	2.40	7.49	Acceptable	31.2%	Not Acceptable	Not Acceptable
07	3.80	2.82	74.2%	-44.9%	-4.49	-1.10	0.55	3.10	7.28	Acceptable	74.2%	Not Acceptable	Not Acceptable

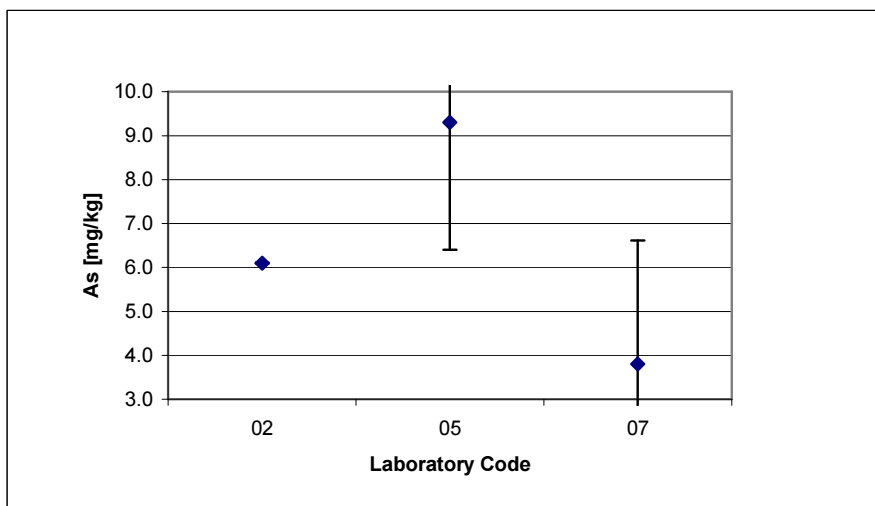


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		505	[mg/kg]	Data evaluation of Cr in compost, sample code 05									
Uncertainty:		9.6		Laboratories Results				Acceptance criteria					Final Score
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	430.10	10.10	2.3%	-14.8%	-1.48	-5.38	0.85	74.90	35.95	Not Acceptable	3.0%	Acceptable	Warning
02	454.00	26.00	5.7%	-10.1%	-1.01	-1.84	0.90	51.00	71.51	Acceptable	6.0%	Acceptable	Acceptable
03	457.80	20.90	4.6%	-9.3%	-0.93	-2.05	0.91	47.20	59.34	Acceptable	4.9%	Acceptable	Acceptable
05	488.50	10.20	2.1%	-3.3%	-0.33	-1.18	0.97	16.50	36.14	Acceptable	2.8%	Acceptable	Acceptable
06	518.73	7.60	1.5%	2.7%	0.27	1.12	1.03	13.73	31.59	Acceptable	2.4%	Acceptable	Acceptable
07	312.80	82.10	26.2%	-38.1%	-3.81	-2.33	0.62	192.20	213.26	Acceptable	26.3%	Not Acceptable	Not Acceptable

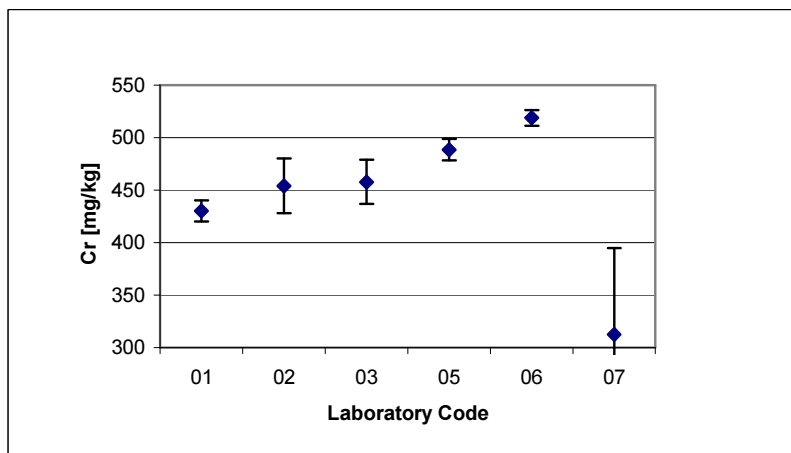


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		248	[mg/kg]	Data evaluation of Ni in compost, sample code 05									
Uncertainty:		9.4		Laboratories Results				Acceptance criteria					Final Score
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	250.00	6.20	2.5%	0.8%	0.08	0.17	1.01	1.90	29.05	Acceptable	4.5%	Acceptable	Acceptable
02	150.00	9.00	6.0%	-39.5%	-3.95	-7.54	0.60	98.10	33.58	Not Acceptable	7.1%	Acceptable	Not Acceptable
03	254.60	7.20	2.8%	2.6%	0.26	0.55	1.03	6.50	30.55	Acceptable	4.7%	Acceptable	Acceptable
04	186.70	1.90	1.0%	-24.7%	-2.47	-6.40	0.75	61.40	24.74	Not Acceptable	3.9%	Acceptable	Not Acceptable
05	228.00	7.80	3.4%	-8.1%	-0.81	-1.65	0.92	20.10	31.51	Acceptable	5.1%	Acceptable	Acceptable
06	231.96	5.04	2.2%	-6.5%	-0.65	-1.51	0.93	16.14	27.52	Acceptable	4.4%	Acceptable	Acceptable
07	293.10	171.00	58.3%	18.1%	1.81	0.26	1.18	45.00	441.85	Acceptable	58.5%	Not Acceptable	Warning

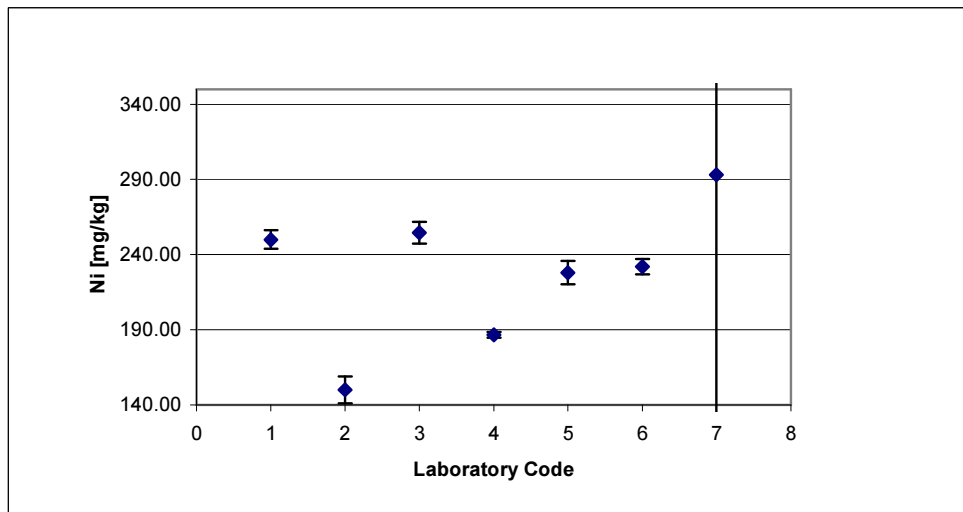


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		228.9	[mg/kg]	Data evaluation of Zn in compost, sample code 05									
Uncertainty:		14.60											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	208.00	10.10	4.9%	-9.1%	-0.91	-1.18	0.91	20.90	45.80	Acceptable	8.0%	Acceptable	Acceptable
02	248.00	5.00	2.0%	8.3%	0.83	1.24	1.08	19.10	39.82	Acceptable	6.7%	Acceptable	Acceptable
03	219.00	12.70	5.8%	-4.3%	-0.43	-0.51	0.96	9.90	49.92	Acceptable	8.6%	Acceptable	Acceptable
04	141.33	1.40	1.0%	-38.3%	-3.83	-5.97	0.62	87.57	37.84	Not Acceptable	6.5%	Acceptable	Not Acceptable
05	243.20	5.10	2.1%	6.2%	0.62	0.92	1.06	14.30	39.90	Acceptable	6.7%	Acceptable	Acceptable
06	215.27	3.49	1.6%	-6.0%	-0.60	-0.91	0.94	13.63	38.73	Acceptable	6.6%	Acceptable	Acceptable
07	138.90	61.40	44.2%	-39.3%	-3.93	-1.43	0.61	90.00	162.83	Acceptable	44.7%	Not Acceptable	Not Acceptable

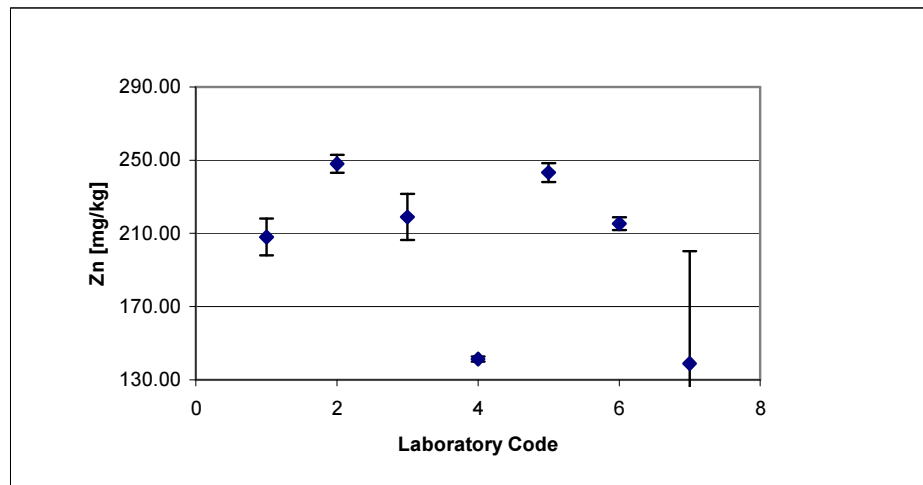


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		6.90	[mg/kg]	Data evaluation of As in compost, sample code 06									
Uncertainty:		0.1		Laboratories Results			Acceptance criteria					Final Score	
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P		Score
02	5.95	0.01	0.2%	-13.8%	-1.38	-10.49	0.86	0.950	0.234	Not Acceptable	1.3%	Acceptable	Warning
05	9.30	2.90	31.2%	34.8%	3.48	0.83	1.35	2.40	7.49	Acceptable	31.2%	Not Acceptable	Not Acceptable
07	3.96	2.84	71.7%	-42.6%	-4.26	-1.03	0.57	2.94	7.33	Acceptable	71.7%	Not Acceptable	Not Acceptable

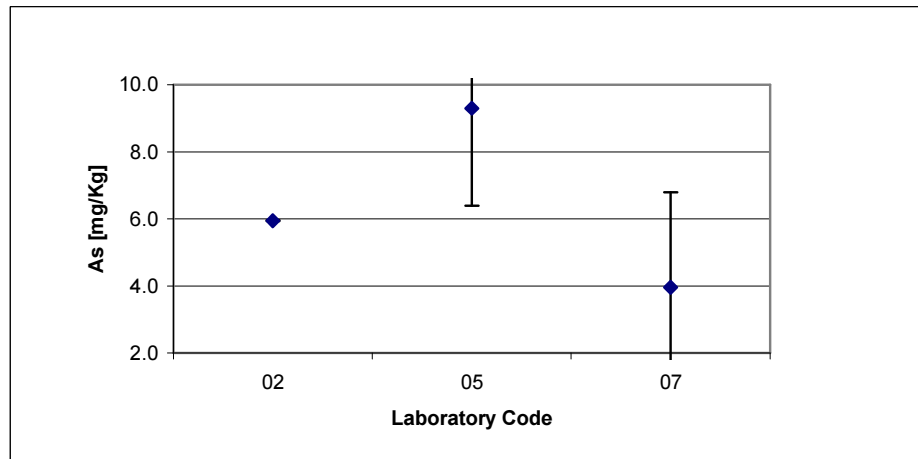


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		505	[mg/kg]	Data evaluation of Cr in compost, sample code 06											
Uncertainty:		9.6		Laboratories Results							Acceptance criteria				
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision				
		[mg/kg]	%					A1	A2	Score	P	Score			
01	411.50	19.70	4.8%	-18.5%	-1.85	-4.27	0.81	93.50	59.17	Not Acceptable	5.2%	Acceptable	Warning		
02	443.00	25.00	5.6%	-12.3%	-1.23	-2.32	0.88	62.00	72.31	Acceptable	6.0%	Acceptable	Acceptable		
03	439.90	2.60	0.6%	-12.9%	-1.29	-6.55	0.87	65.10	26.85	Not Acceptable	2.0%	Acceptable	Warning		
05	466.20	9.10	2.0%	-7.7%	-0.77	-2.93	0.92	38.80	35.71	Not Acceptable	2.7%	Acceptable	Warning		
06	502.86	16.20	3.2%	-0.4%	-0.04	-0.11	1.00	2.14	50.84	Acceptable	3.7%	Acceptable	Acceptable		
07	349.50	134.90	38.6%	-30.8%	-3.08	-1.15	0.69	155.50	365.15	Acceptable	38.6%	Not Acceptable	Not Acceptable		

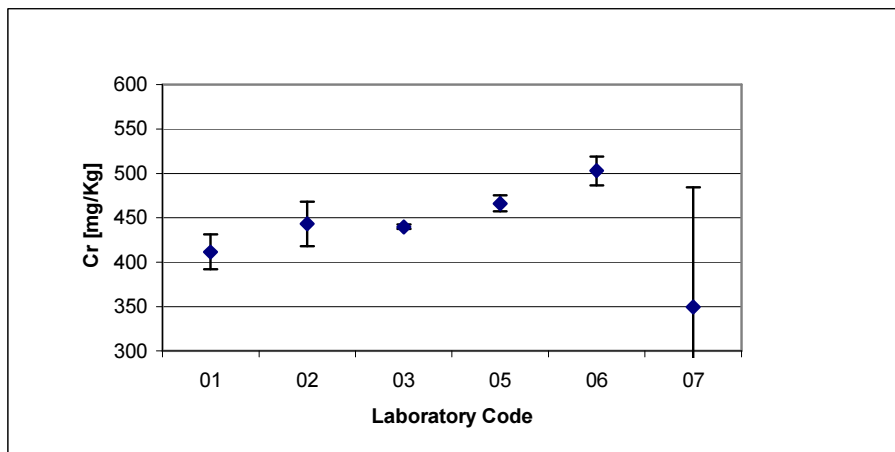


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		248.1	[mg/kg]	Data evaluation of Ni in compost, sample code 06									
Uncertainty:		9.4		Laboratories Results			Acceptance criteria				Final Score		
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness				Precision	
		[mg/kg]	%					A1	A2	Score		P	Score
01	247.80	5.80	2.3%	-0.1%	-0.01	-0.03	1.00	0.30	28.50	Acceptable	4.5%	Acceptable	Acceptable
02	150.00	9.00	6.0%	-39.5%	-3.95	-7.54	0.60	98.10	33.58	Not Acceptable	7.1%	Acceptable	Not Acceptable
03	249.10	6.70	2.7%	0.4%	0.04	0.09	1.00	1.00	29.78	Acceptable	4.6%	Acceptable	Acceptable
04	183.30	1.80	1.0%	-26.1%	-2.61	-6.77	0.74	64.80	24.69	Not Acceptable	3.9%	Acceptable	Not Acceptable
05	218.00	6.30	2.9%	-12.1%	-1.21	-2.66	0.88	30.10	29.20	Not Acceptable	4.8%	Acceptable	Warning
06	233.15	2.61	1.1%	-6.0%	-0.60	-1.53	0.94	14.95	25.17	Acceptable	4.0%	Acceptable	Acceptable
07	345.30	181.40	52.5%	39.2%	3.92	0.54	1.39	97.20	468.64	Acceptable	52.7%	Not Acceptable	Not Acceptable

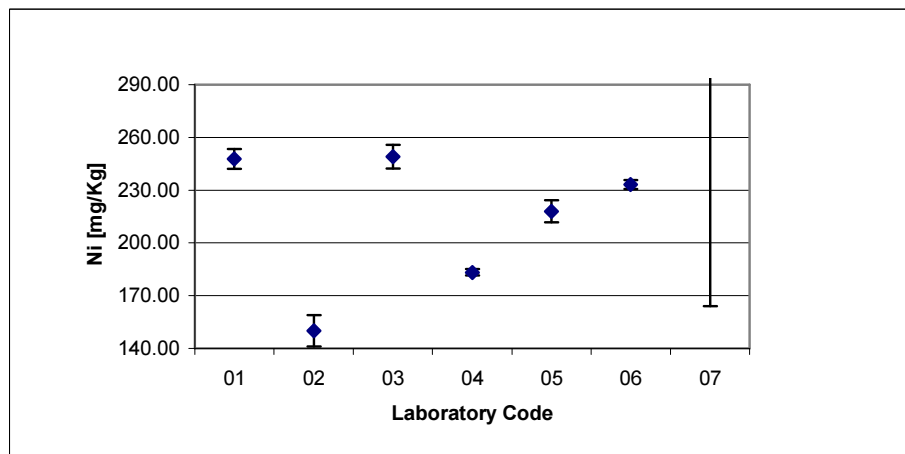


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$



Target value:		228.9	[mg/kg]	Data evaluation of Zn in compost, sample code 06									
Uncertainty:		14.60											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	209.60	2.50	1.2%	-8.4%	-0.84	-1.30	0.92	19.30	38.22	Acceptable	6.5%	Acceptable	Acceptable
02	242.00	5.00	2.1%	5.7%	0.57	0.85	1.06	13.10	39.82	Acceptable	6.7%	Acceptable	Acceptable
03	213.60	10.00	4.7%	-6.7%	-0.67	-0.86	0.93	15.30	45.66	Acceptable	7.9%	Acceptable	Acceptable
04	140.67	1.40	1.0%	-38.5%	-3.85	-6.02	0.61	88.23	37.84	Not Acceptable	6.5%	Acceptable	Not Acceptable
05	243.17	7.40	3.0%	6.2%	0.62	0.87	1.06	14.27	42.23	Acceptable	7.1%	Acceptable	Acceptable
06	210.08	2.78	1.3%	-8.2%	-0.82	-1.27	0.92	18.82	38.34	Acceptable	6.5%	Acceptable	Acceptable
07	154.60	59.60	38.6%	-32.5%	-3.25	-1.21	0.68	74.30	158.31	Acceptable	39.1%	Not Acceptable	Not Acceptable

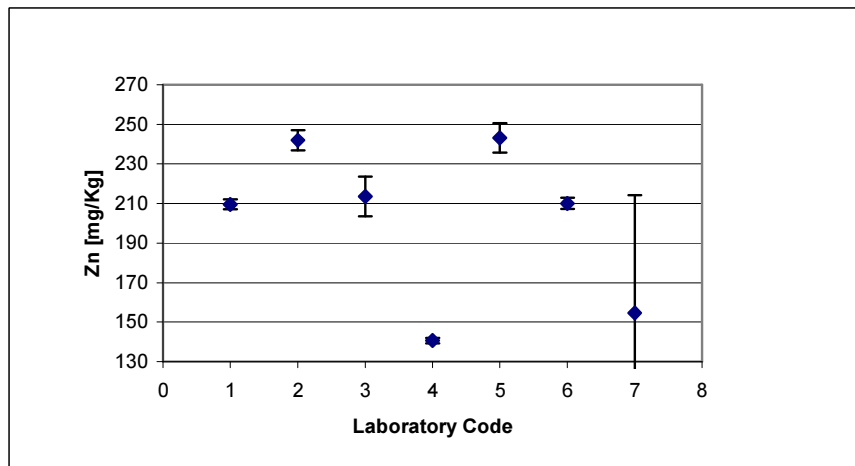


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		11.0	[mg/kg]	Data evaluation of As in soil, sample code 07									
Uncertainty:		1.0		Laboratories Results			Acceptance criteria					Final Score	
Laboratory Code	Value [mg/kg]	Unc.		Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
02	10.19	0.02	0.2%	-7.4%	-0.74	-0.81	0.93	0.810	2.581	Acceptable	9.1%	Acceptable	Acceptable
05	13.90	1.40	10.1%	26.4%	2.64	1.69	1.26	2.90	4.44	Acceptable	13.6%	Acceptable	Acceptable
07	6.79	5.05	74.4%	-38.3%	-3.83	-0.82	0.62	4.21	13.28	Acceptable	74.9%	Not Acceptable	Not Acceptable

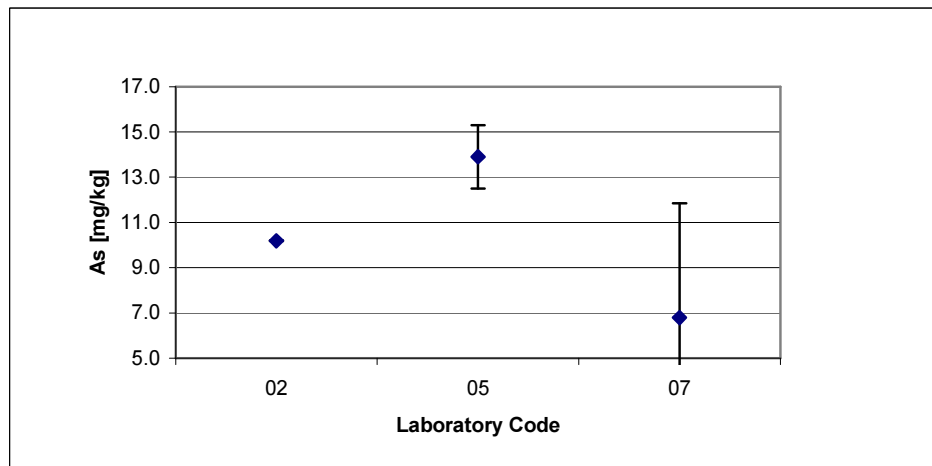


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		1030	[mg/kg]	Data evaluation of Cr in soil, sample code 07									
Uncertainty:		30											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value	Unc.						Trueness			Precision		
		[mg/kg]	[mg/kg]					%	A1	A2	Score	P	
01	892.6	43.0	4.8%	-13.3%	-1.33	-2.62	0.87	137.40	141.56	Acceptable	5.6%	Acceptable	Acceptable
02	812.0	48.0	5.9%	-21.2%	-2.12	-3.85	0.79	218.00	152.83	Not Acceptable	6.6%	Acceptable	Not Acceptable
03	773.4	24.4	3.2%	-24.9%	-2.49	-6.64	0.75	256.60	104.41	Not Acceptable	4.3%	Acceptable	Not Acceptable
05	1027.0	20.0	1.9%	-0.3%	-0.03	-0.08	1.00	3.00	97.35	Acceptable	3.5%	Acceptable	Acceptable
06	877.6	12.2	1.4%	-14.8%	-1.48	-4.71	0.85	152.40	87.44	Not Acceptable	3.2%	Acceptable	Warning
07	683.4	263.6	38.6%	-33.7%	-3.37	-1.31	0.66	346.60	716.31	Acceptable	38.7%	Not Acceptable	Not Acceptable

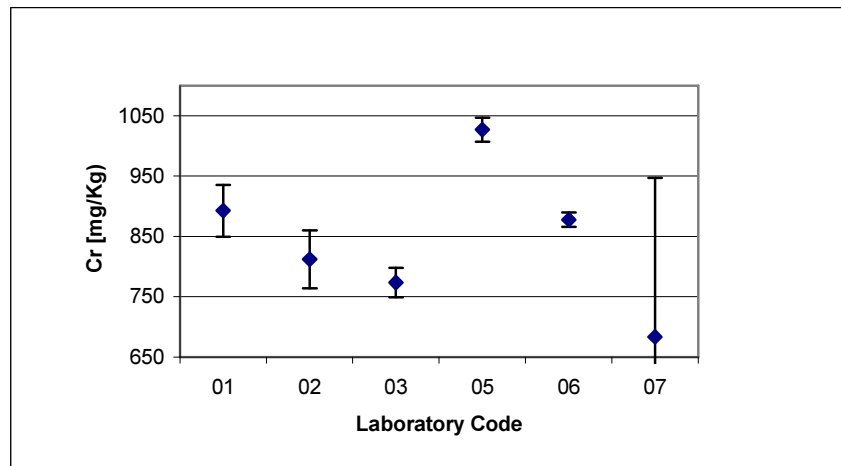


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		91.8	[mg/kg]	Data evaluation of Zn in soil, sample code 07									
Uncertainty:		10.5											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	102.2	0.9	0.9%	11.3%	1.13	0.99	1.11	10.40	27.19	Acceptable	11.5%	Acceptable	Acceptable
02	101.0	1.0	1.0%	10.0%	1.00	0.87	1.10	9.20	27.21	Acceptable	11.5%	Acceptable	Acceptable
03	96.4	4.2	4.4%	5.0%	0.50	0.40	1.05	4.55	29.19	Acceptable	12.2%	Acceptable	Acceptable
04	87.3	0.9	1.0%	-4.9%	-0.49	-0.42	0.95	4.47	27.18	Acceptable	11.5%	Acceptable	Acceptable
05	109.5	2.2	2.0%	19.3%	1.93	1.65	1.19	17.70	27.68	Acceptable	11.6%	Acceptable	Acceptable
06	144.3	4.1	2.9%	57.2%	5.72	4.65	1.57	52.50	29.11	Not Acceptable	11.8%	Acceptable	Not Acceptable
07	76.3	30.3	39.8%	-16.9%	-1.69	-0.48	0.83	15.49	82.83	Acceptable	41.4%	Not Acceptable	Warning

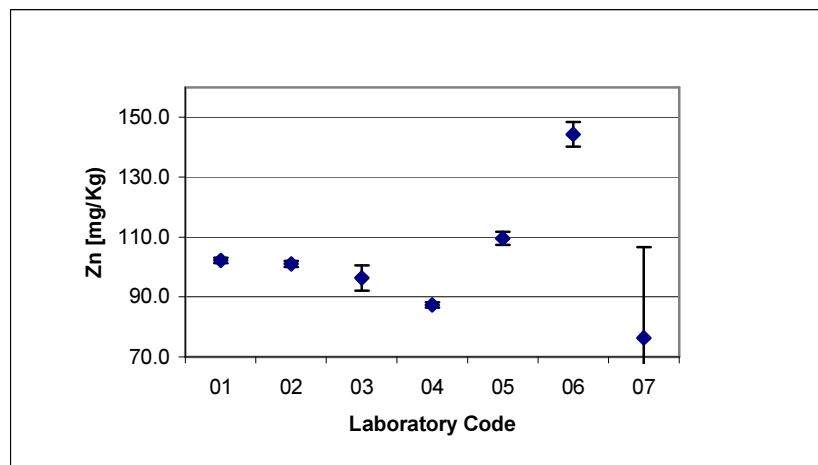


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		11.0	[mg/kg]	Data evaluation of As in soil, sample code 08									
Uncertainty:		1.0											
Laboratory Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
02	9.96	0.02	0.2%	-9.5%	-0.95	-1.04	0.91	1.040	2.581	Acceptable	9.1%	Acceptable	Acceptable
05	14.80	1.30	8.8%	34.5%	3.45	2.32	1.35	3.80	4.23	Acceptable	12.6%	Acceptable	Acceptable
07	7.15	5.45	76.2%	-35.0%	-3.50	-0.69	0.65	3.85	14.30	Acceptable	76.8%	Not Acceptable	Not Acceptable

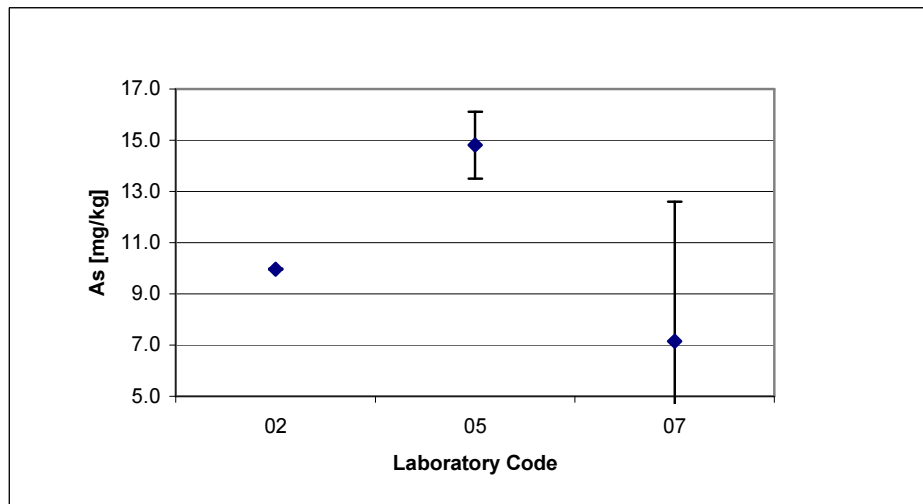


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{lab}}{Value_{lab}}\right)^2} \times 100\%$$

Target value:		1030	[mg/kg]	Data evaluation of Cr in soil, sample code 08									
Uncertainty:		30											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	903.2	42.9	4.7%	-12.3%	-1.23	-2.42	0.88	126.80	135.06	Acceptable	5.6%	Acceptable	Acceptable
02	824.0	49.0	5.9%	-20.0%	-2.00	-3.59	0.80	206.00	148.23	Not Acceptable	6.6%	Acceptable	Not Acceptable
03	774.5	14.8	1.9%	-24.8%	-2.48	-7.64	0.75	255.50	86.31	Not Acceptable	3.5%	Acceptable	Not Acceptable
05	1071.0	23.0	2.1%	4.0%	0.40	1.08	1.04	41.00	97.53	Acceptable	3.6%	Acceptable	Acceptable
06	905.9	8.0	0.9%	-12.0%	-1.20	-4.00	0.88	124.10	80.10	Not Acceptable	3.0%	Acceptable	Warning
07	730.1	281.2	38.5%	-29.1%	-2.91	-1.06	0.71	299.90	729.61	Acceptable	38.6%	Not Acceptable	Not Acceptable

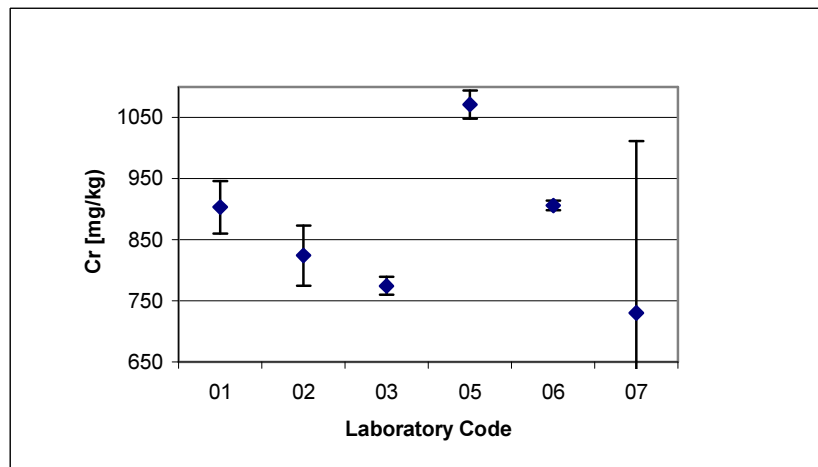


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

Target value:		91.8	[mg/kg]	Data evaluation of Zn in soil, sample code 08									
Uncertainty:		10.5											
Lab. Code	Laboratories Results			Bias(%)	Z-Score	U-Score	Laboratory/IAEA	Acceptance criteria					Final Score
	Value [mg/kg]	Unc.						Trueness			Precision		
		[mg/kg]	%					A1	A2	Score	P	Score	
01	104.4	2.5	2.4%	13.7%	1.37	1.17	1.14	12.60	27.85	Acceptable	11.7%	Acceptable	Acceptable
02	101.0	1.0	1.0%	10.0%	1.00	0.87	1.10	9.20	27.21	Acceptable	11.5%	Acceptable	Acceptable
03	94.4	2.2	2.3%	2.8%	0.28	0.24	1.03	2.61	27.68	Acceptable	11.7%	Acceptable	Acceptable
04	85.7	0.9	1.0%	-6.7%	-0.67	-0.58	0.93	6.13	27.18	Acceptable	11.5%	Acceptable	Acceptable
05	108.5	2.3	2.1%	18.2%	1.82	1.55	1.18	16.70	27.73	Acceptable	11.6%	Acceptable	Acceptable
06	146.6	3.3	2.3%	59.7%	5.97	4.97	1.60	54.80	28.43	Not Acceptable	11.7%	Acceptable	Not Acceptable
07	81.2	32.9	40.5%	-11.5%	-1.15	-0.31	0.88	10.60	89.03	Acceptable	42.1%	Not Acceptable	Warning

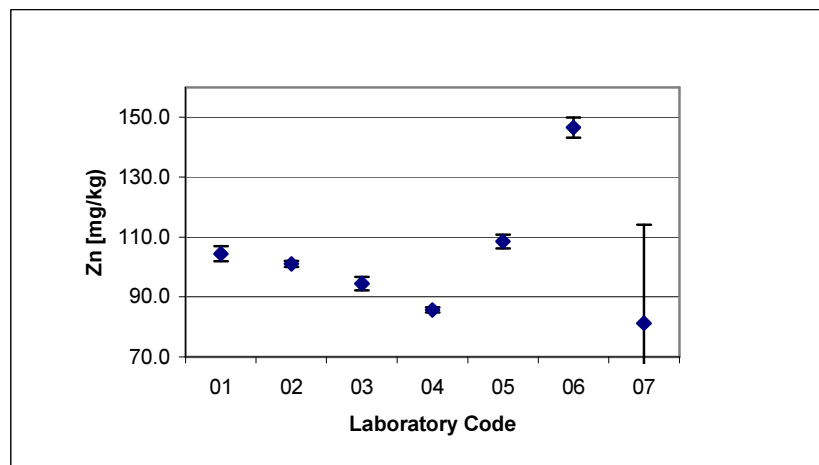


Table legend:

$$A1: |Value_{IAEA} - Value_{Laboratory}|$$

$$A2: 2.58 \times \sqrt{Unc_{IAEA}^2 + Unc_{Laboratory}^2}$$

$$P: \sqrt{\left(\frac{Unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{Unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

**Analytical Performance Evaluation of Laboratory 01**  
Trace elements in soil and compost

**Sample code 01**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Ni	9.70	1.85	12.25	0.42	3.4%	26%	2.63	1.34	1.26	2.55	5.12	Acceptable	19.4%	Acceptable	Acceptable

**Sample code 05**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.00	9.60	430.10	10.10	2.3%	-15%	-1.48	-5.38	0.85	74.90	37.62	Not Acceptable	3.0%	Acceptable	Warning
Ni	248.10	9.40	250.00	6.20	2.5%	1%	0.08	0.17	1.01	1.90	30.40	Acceptable	4.5%	Acceptable	Acceptable
Zn	228.90	14.60	208.00	10.10	4.9%	-9%	-0.91	-1.18	0.91	20.90	47.93	Acceptable	8.0%	Acceptable	Acceptable

**Sample code 06**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.00	9.60	411.50	19.70	4.8%	-19%	-1.85	-4.27	0.81	93.50	59.17	Not Acceptable	5.2%	Acceptable	Warning
Ni	248.10	9.40	247.80	5.80	2.3%	0%	-0.01	-0.03	1.00	0.30	29.82	Acceptable	4.5%	Acceptable	Acceptable
Zn	228.90	14.60	209.60	2.50	1.2%	-8%	-0.84	-1.30	0.92	19.30	39.99	Acceptable	6.5%	Acceptable	Acceptable



**Analytical Performance Evaluation of Laboratory 01**  
Trace elements in soil

**Sample code 07**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.0	30.0	892.6	43.0	4.8%	-13%	-1.33	-2.62	0.87	137.40	141.56	Acceptable	5.6%	Acceptable	Acceptable
Zn	91.8	10.5	102.2	0.9	0.9%	11%	1.13	0.99	1.11	10.40	28.45	Acceptable	11.5%	Acceptable	Acceptable

**Sample code 08**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.00	30.00	903.2	42.9	4.7%	-12%	-1.23	-2.42	0.88	126.80	141.34	Acceptable	5.6%	Acceptable	Acceptable
Zn	91.8	10.5	104.4	2.5	2.4%	14%	1.37	1.17	1.14	12.60	29.14	Acceptable	11.7%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 02

### Trace elements in soil and compost

#### Sample code 01

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	2.56	0.32	2.23	0.01	0.4%	-13%	-1.29	-1.03	0.87	0.33	0.83	Acceptable	12.5%	Acceptable	Acceptable
Ni	9.70	1.85	7.80	0.80	10.3%	-20%	-1.96	-0.94	0.80	1.90	5.20	Acceptable	21.7%	Acceptable	Acceptable

#### Sample code 05

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	6.90	0.09	6.10	0.01	0.2%	-12%	-1.16	-8.83	0.88	0.80	0.23	Not Acceptable	1.3%	Acceptable	Warning
Cr	505.00	9.60	454.00	26.00	5.7%	-10%	-1.01	-1.84	0.90	51.00	71.51	Acceptable	6.0%	Acceptable	Acceptable
Ni	248.1	9.4	150.00	9.00	6.0%	-40%	-3.95	-7.54	0.60	98.10	33.58	Not Acceptable	7.1%	Acceptable	Not Acceptable
Zn	228.9	14.6	248.00	5.00	2.0%	8%	0.83	1.24	1.08	19.10	39.82	Acceptable	6.7%	Acceptable	Acceptable

#### Sample code 06

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	6.90	0.09	5.95	0.01	0.2%	-14%	-1.38	-10.49	0.86	0.95	0.23	Not Acceptable	1.3%	Acceptable	Warning
Cr	505.0	9.60	443.00	25.00	5.6%	-12%	-1.23	-2.32	0.88	62.00	69.09	Acceptable	6.0%	Acceptable	Acceptable
Ni	248.1	9.4	150.00	9.00	6.0%	-40%	-3.95	-7.54	0.60	98.10	33.58	Not Acceptable	7.1%	Acceptable	Not Acceptable
Zn	228.9	14.6	242.00	5.00	2.1%	6%	0.57	0.85	1.06	13.10	39.82	Acceptable	6.7%	Acceptable	Acceptable

**Analytical Performance Evaluation of Laboratory 02**  
Trace elements in soil

**Sample code 07**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	11.00	1.00	10.19	0.02	0.2%	-7%	<b>-0.74</b>	-0.81	0.93	0.81	2.58	<b>Acceptable</b>	9.1%	<b>Acceptable</b>	<b>Acceptable</b>
Cr	1030.00	30.00	812.00	48.00	5.9%	-21%	<b>-2.12</b>	-3.85	0.79	218.00	146.04	<b>Not Acceptable</b>	6.6%	<b>Acceptable</b>	<b>Not Acceptable</b>
Zn	91.8	10.5	101.00	1.00	1.0%	10%	<b>1.00</b>	0.87	1.10	9.20	27.21	<b>Acceptable</b>	11.5%	<b>Acceptable</b>	<b>Acceptable</b>

**Sample code 08**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	11.00	1.00	9.96	0.02	0.2%	-9%	<b>-0.95</b>	-1.04	0.91	1.04	2.58	<b>Acceptable</b>	9.1%	<b>Acceptable</b>	<b>Acceptable</b>
Cr	1030.00	30.00	824.00	49.00	5.9%	-20%	<b>-2.00</b>	-3.59	0.80	206.00	148.23	<b>Not Acceptable</b>	6.6%	<b>Acceptable</b>	<b>Not Acceptable</b>
Zn	91.8	10.5	101.00	1.00	1.0%	10%	<b>1.00</b>	0.87	1.10	9.20	27.21	<b>Acceptable</b>	11.5%	<b>Acceptable</b>	<b>Acceptable</b>

## Analytical Performance Evaluation of Laboratory 03

### Trace elements in soil and compost

#### Sample code 01

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	%					A1	A2	Score	P	Score	
Ni	9.70	1.85	12.76	1.61	12.6%	32%	3.15	1.25	1.32	3.06	6.33	Acceptable	22.9%	Not Acceptable	Not Acceptable

#### Sample code 05

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.0	9.60	457.80	20.90	4.6%	-9%	-0.93	-2.05	0.91	47.20	59.34	Acceptable	4.9%	Acceptable	Acceptable
Ni	248.1	9.4	254.60	7.20	2.8%	3%	0.26	0.55	1.03	6.50	30.55	Acceptable	4.7%	Acceptable	Acceptable
Zn	228.9	14.6	219.00	12.70	5.8%	-4%	-0.43	-0.51	0.96	9.90	49.92	Acceptable	8.6%	Acceptable	Acceptable

#### Sample code 06

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value	Unc.	Value	Unc.						Trueness			Precision		
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.0	9.60	439.90	2.60	0.6%	-13%	-1.29	-6.55	0.87	65.10	25.66	Not Acceptable	2.0%	Acceptable	Warning
Ni	248.1	9.4	249.10	6.70	2.7%	0%	0.04	0.09	1.00	1.00	29.78	Acceptable	4.6%	Acceptable	Acceptable
Zn	228.9	14.6	213.60	10.00	4.7%	-7%	-0.67	-0.86	0.93	15.30	45.66	Acceptable	7.9%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 03

### Trace elements in soil

#### Sample code 07

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.0	30.0	773.4	24.4	3.2%	-25%	<b>-2.49</b>	-6.64	0.75	256.60	99.77	<b>Not Acceptable</b>	4.3%	<b>Acceptable</b>	<b>Not Acceptable</b>
Zn	91.8	10.5	96.4	4.2	4.4%	5%	<b>0.50</b>	0.40	1.05	4.55	29.19	<b>Acceptable</b>	12.2%	<b>Acceptable</b>	<b>Acceptable</b>

#### Sample code 08

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.0	30.0	774.5	14.8	1.9%	-25%	<b>-2.48</b>	-7.64	0.75	255.50	86.31	<b>Not Acceptable</b>	3.5%	<b>Acceptable</b>	<b>Not Acceptable</b>
Zn	91.8	10.5	94.4	2.2	2.3%	3%	<b>0.28</b>	0.24	1.03	2.61	27.68	<b>Acceptable</b>	11.7%	<b>Acceptable</b>	<b>Acceptable</b>

**Analytical Performance Evaluation of Laboratory 04**  
Trace elements in soil and compost

**Sample code 01**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Ni	9.70	1.85	356.70	3.50	1.0%	3577%	357.73	87.65	36.77	347.00	10.21	Not Acceptable	19.1%	Acceptable	Not Acceptable

**Sample code 05**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Ni	248.1	9.4	186.70	1.90	1.0%	-25%	-2.47	-6.40	0.75	61.40	24.74	Not Acceptable	3.9%	Acceptable	Not Acceptable
Zn	228.9	14.6	141.33	1.40	1.0%	-38%	-3.83	-5.97	0.62	87.57	37.84	Not Acceptable	6.5%	Acceptable	Not Acceptable

**Sample code 06**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Laboratory/IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Ni	248.1	9.4	183.30	1.80	1.0%	-26%	-2.61	-6.77	0.74	64.80	24.69	Not Acceptable	3.9%	Acceptable	Not Acceptable
Zn	228.9	14.6	140.67	1.40	1.0%	-39%	-3.85	-6.02	0.61	88.23	37.84	Not Acceptable	6.5%	Acceptable	Not Acceptable

**Analytical Performance Evaluation of Laboratory 04**  
Trace elements in soil

**Sample code 07**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Zn	91.8	10.5	87.3	0.88	1.0%	-5%	<b>-0.49</b>	-0.42	0.95	4.47	27.18	<b>Acceptable</b>	11.5%	<b>Acceptable</b>	<b>Acceptable</b>

**Sample code 08**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Zn	91.8	10.5	85.67	0.86	1.0%	-7%	<b>-0.67</b>	-0.58	0.93	6.13	27.18	<b>Acceptable</b>	11.5%	<b>Acceptable</b>	<b>Acceptable</b>

## Analytical Performance Evaluation of Laboratory 05

### Trace elements in soil and compost

#### Sample code 01

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	2.56	0.32	4.30	1.20	27.9%	68%	6.80	1.40	1.68	1.74	3.20	Acceptable	30.6%	Not Acceptable	Not Acceptable
Ni	9.70	1.85	14.37	3.00	20.9%	48%	4.81	1.32	1.48	4.67	9.09	Acceptable	28.3%	Not Acceptable	Not Acceptable

#### Sample code 05

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	6.90	0.09	9.30	2.90	31.2%	35%	3.48	0.83	1.35	2.40	7.49	Acceptable	31.2%	Not Acceptable	Not Acceptable
Cr	505.0	9.60	488.50	10.20	2.1%	-3%	-0.33	-1.18	0.97	16.50	36.14	Acceptable	2.8%	Acceptable	Acceptable
Ni	248.1	9.4	228.00	7.80	3.4%	-8%	-0.81	-1.65	0.92	20.10	31.51	Acceptable	5.1%	Acceptable	Acceptable
Zn	228.9	14.6	243.20	5.10	2.1%	6%	0.62	0.92	1.06	14.30	39.90	Acceptable	6.7%	Acceptable	Acceptable

#### Sample code 06

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.0	9.60	466.20	9.10	2.0%	-8%	-0.77	-2.93	0.92	38.80	34.13	Not Acceptable	2.7%	Acceptable	Warning
Ni	248.1	9.4	218.00	6.30	2.9%	-12%	-1.21	-2.66	0.88	30.10	29.20	Not Acceptable	4.8%	Acceptable	Warning
Zn	228.9	14.6	243.17	7.40	3.0%	6%	0.62	0.87	1.06	14.27	42.23	Acceptable	7.1%	Acceptable	Acceptable



**Analytical Performance Evaluation of Laboratory 05**  
**Trace elements in soil**

**Sample code 07**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	11.00	1.00	13.90	1.40	10.1%	26%	<b>2.64</b>	1.69	1.26	2.90	4.44	Acceptable	13.6%	Acceptable	Acceptable
Cr	1030.00	30.00	1027.00	20.00	1.9%	0%	<b>-0.03</b>	-0.08	1.00	3.00	93.02	Acceptable	3.5%	Acceptable	Acceptable
Zn	91.8	10.5	109.50	2.20	2.0%	19%	<b>1.93</b>	1.65	1.19	17.70	27.68	Acceptable	11.6%	Acceptable	Acceptable

**Sample code 08**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	10.60	1.00	14.80	1.30	8.8%	40%	<b>3.96</b>	2.56	1.40	4.20	4.23	Acceptable	12.9%	Acceptable	Acceptable
Cr	1030.00	30.00	1071.00	23.00	2.1%	4%	<b>0.40</b>	1.08	1.04	41.00	97.53	Acceptable	3.6%	Acceptable	Acceptable
Zn	91.8	10.5	108.50	2.30	2.1%	18%	<b>1.82</b>	1.55	1.18	16.70	27.73	Acceptable	11.6%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 06

### Trace elements in soil and compost

#### Sample code 01

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Ni	9.70	1.85	9.05	1.06	11.7%	-7%	-0.67	-0.30	0.93	0.65	5.50	Acceptable	22.4%	Not Acceptable	Warning

#### Sample code 05

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.0	9.60	518.73	7.60	1.5%	3%	0.27	1.12	1.03	13.73	31.59	Acceptable	2.4%	Acceptable	Acceptable
Ni	248.1	9.4	231.96	5.04	2.2%	-7%	-0.65	-1.51	0.93	16.14	27.52	Acceptable	4.4%	Acceptable	Acceptable
Zn	228.9	14.6	215.27	3.49	1.6%	-6%	-0.60	-0.91	0.94	13.63	38.73	Acceptable	6.6%	Acceptable	Acceptable

#### Sample code 06

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	505.0	9.60	502.86	16.20	3.2%	0%	-0.04	-0.11	1.00	2.14	48.58	Acceptable	3.7%	Acceptable	Acceptable
Ni	248.1	9.4	233.15	2.61	1.1%	-6%	-0.60	-1.53	0.94	14.95	25.17	Acceptable	4.0%	Acceptable	Acceptable
Zn	228.9	14.6	210.08	2.78	1.3%	-8%	-0.82	-1.27	0.92	18.82	38.34	Acceptable	6.5%	Acceptable	Acceptable

## Analytical Performance Evaluation of Laboratory 06

### Trace elements in soil

#### Sample code 07

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.00	30.00	877.60	12.20	1.4%	-15%	<b>-1.48</b>	-4.71	0.85	152.40	83.56	<b>Not Acceptable</b>	3.2%	<b>Acceptable</b>	<b>Warning</b>
Zn	91.8	10.5	144.30	4.13	2.9%	57%	<b>5.72</b>	4.65	1.57	52.50	29.11	<b>Not Acceptable</b>	11.8%	<b>Acceptable</b>	<b>Not Acceptable</b>

#### Sample code 08

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
Cr	1030.00	30.00	905.9	8.0	0.9%	-12%	<b>-1.20</b>	-4.00	0.88	124.10	80.10	<b>Not Acceptable</b>	3.0%	<b>Acceptable</b>	<b>Warning</b>
Zn	91.8	10.5	146.60	3.34	2.3%	60%	<b>5.97</b>	4.97	1.60	54.80	28.43	<b>Not Acceptable</b>	11.7%	<b>Acceptable</b>	<b>Not Acceptable</b>

**Analytical Performance Evaluation of Laboratory 07**  
Trace elements in soil and compost

**Sample code 01**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	2.56	0.32	2.34	3.71	158.5	-9%	-0.86	-0.06	0.91	0.22	9.61	Acceptable	159.0%	Not Acceptable	Warning
Ni	9.70	1.85	<50												Acceptable

**Sample code 05**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	6.90	0.09	3.80	2.82	74.2	-45%	-4.49	-1.10	0.55	3.10	7.28	Acceptable	74.2%	Not Acceptable	Not Acceptable
Cr	505.0	9.60	312.80	82.10	26.2	-38%	-3.81	-2.33	0.62	192.20	213.26	Acceptable	26.3%	Not Acceptable	Not Acceptable
Ni	248.1	9.4	293.10	171.00	58.3	18%	1.81	0.26	1.18	45.00	441.85	Acceptable	58.5%	Not Acceptable	Warning
Zn	228.9	14.6	138.90	61.40	44.2	-39%	-3.93	-1.43	0.61	90.00	162.83	Acceptable	44.7%	Not Acceptable	Not Acceptable

**Sample code 06**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	6.90	0.09	3.96	2.84	71.7	-43%	-4.26	-1.03	0.57	2.94	7.33	Acceptable	71.7%	Not Acceptable	Not Acceptable
Cr	505.0	9.60	349.50	134.90	38.6	-31%	-3.08	-1.15	0.69	155.50	348.92	Acceptable	38.6%	Not Acceptable	Not Acceptable
Ni	248.1	9.4	345.30	181.40	52.5	39%	3.92	0.54	1.39	97.20	468.64	Acceptable	52.7%	Not Acceptable	Not Acceptable
Zn	228.9	14.6	154.60	59.60	38.6	-32%	-3.25	-1.21	0.68	74.30	158.31	Acceptable	39.1%	Not Acceptable	Not Acceptable

**Analytical Performance Evaluation of Laboratory 07**  
**Trace elements in soil**

**Sample code 07**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	11.00	1.00	6.79	5.05	74.4%	-38%	<b>-3.83</b>	-0.82	0.62	4.21	13.28	Acceptable	74.9%	Not Acceptable	Not Acceptable
Cr	1030.00	30.00	683.40	263.60	38.6%	-34%	<b>-3.37</b>	-1.31	0.66	346.60	684.48	Acceptable	38.7%	Not Acceptable	Not Acceptable
Zn	91.8	10.5	76.31	30.34	39.8%	-17%	<b>-1.69</b>	-0.48	0.83	15.49	82.83	Acceptable	41.4%	Not Acceptable	Warning

**Sample code 08**

Analyte	IAEA		Laboratory			R. bias %	Z-score	U-Test	Lab./IAEA	Acceptance criteria					Final score
	Value [mg/kg]	Unc. [mg/kg]	Value [mg/kg]	Unc.						Trueness			Precision		
				[mg/kg]	%					A1	A2	Score	P	Score	
As	10.60	1.00	7.15	5.45	76.2%	-33%	<b>-3.25</b>	-0.62	0.67	3.45	14.30	Acceptable	76.8%	Not Acceptable	Not Acceptable
Cr	1030.00	30.00	730.10	281.20	38.5%	-29%	<b>-2.91</b>	-1.06	0.71	299.90	729.61	Acceptable	38.6%	Not Acceptable	Not Acceptable
Zn	91.8	10.5	81.20	32.87	40.5%	-12%	<b>-1.15</b>	-0.31	0.88	10.60	89.03	Acceptable	42.1%	Not Acceptable	Warning

## **Appendix C: Characterisation report of compost and soil test samples**

### **PRODUCTION AND CHARACTERIZATION OF APAT-RM004 (COMPOST) AND APAT-RM005 (AGRICULTURAL SOIL) MATRIX REFERENCE MATERIALS**

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#### **Production of the matrix reference materials**

##### **APAT-RM004 (compost)**

Compost is the decomposed remnants of organic materials (usually those with plant origins) and it is used in gardening and agriculture, mixed in with the soil. It improves soil structure, increases the amount of organic matter, and provides nutrients. Compost is a common name for humus, which is the result of the decomposition of organic matter. Generally, compost is the raw material obtained by the aerobic decomposition of the organic residues of the municipal waste or of the vegetable market waste. Composting is the industrial operation to produce compost on a large scale and it is the controlled decomposition technique of organic matter. Rather than allowing nature to take its slow course, a composter provides an optimal environment in which decomposer can thrive. The compost raw material used to prepare the APAT-RM004 reference material has been obtained from an aerobic composting plant located near Rome (Italy). This plant produces compost from organic waste originating from municipal routine plant trimmings, pruning, lawn mowing and wastes deriving from vegetable markets.

About 100 kg of stabilized compost were collected in 2003 from the compost piles, directly by the Environmental Metrology Service of the Italian Environmental Protection Agency (APAT). The material was then transported to the APAT laboratories in Rome (Italy) for processing. Drying was done at a constant temperature of +40°C in a ventilated oven. About 30 kg of compost were then sieved through a 2 mm mesh sieve and the resulting fraction >2mm, composed of barks, stems or extraneous materials was discarded. The fraction <2mm was milled into powder (<90 micrometer) and about 22 kg were homogenized over two weeks by mixing into a cylindrical drum placed on a roll-bed.

The bulk homogeneity of the sample was checked by measuring the C and N concentrations on 10 sub-samples (10-15 g each), taken directly from the cylindrical drum. The samples were analyzed by CHN-S elemental analyzer [1]. As the data of C and N content did not show any heterogeneity in the material (coefficient of variation were below 1% for C and N), the samples were bottled. The bottling has been carried out in one day and to prevent the possible segregation of fine particles, 10 samples, each of about 30 g, were taken from the center of the cylindrical drum immediately after stopping the rotation and placed into 10 pre-cleaned brown glass bottles. The drum was again rotated for a further 2

minutes and again 10 samples were taken in the same way and bottled. The sampling from the cylindrical drum and the bottling of the samples continued following this procedure until the material was finished (about 700 bottles of compost reference material were obtained). The between-bottle homogeneity and stability testing was verified on bottles selected sequentially over the whole bottling process.

#### *APAT-RM005 (agricultural soil)*

The raw material was collected in an agricultural area qualified as a “reference site” in the frame of the SOILSAMP international project, funded and coordinated by APAT [2] [3] and [4]. The area is located in a research field belonging to a public scientific institution (Ente Regionale per lo Sviluppo Agricolo del Friuli Venezia Giulia, ERSA, Italy), in Pozzuolo del Friuli, Udine, in the north-eastern part of Italy. The reference site (10000 m<sup>2</sup>) is flat and regular shaped, with three sub-areas of different gravel content. On average the fraction above 2mm represents only 13% of the sampled soil. Crop production did not take place on the site over the last six years. The soil has a quite balanced grain size distribution with a slight dominance of the silt fraction (47%) and a low percentage of clay (below 16%). Relatively high pH values (about 7.7) are observed as well as a low percentage of organic carbon content. The CEC reveals low values (in average below 16 cmol<sub>(+)</sub>/kg) [4]. The soil samples were collected in 2001 and before processing, stones, roots and other extraneous material were removed. Drying was done at a constant temperature of +40°C in a ventilated oven. The soil was then sieved through a 2 mm mesh sieve and the resulting fraction >2mm was discarded. The fraction <2mm was milled into powder (<90 micrometer) and about 20 kg were homogenized over two weeks by mixing into a cylindrical drum placed on a roll-bed. The bottling has been carried out in one day and to prevent the possible segregation of fine particles, 10 samples, each of about 30 g, were taken from the center of the cylindrical drum immediately after stopping the rotation and placed into 10 pre-cleaned brown glass bottles. The drum was again rotated for a further 2 minutes and again 20 samples were taken in the same way and bottled. The sampling from the cylindrical drum and the bottling of the samples continued following this procedure until the material was finished (about 1000 bottles of soil reference material were obtained). The between-bottle homogeneity and stability testing was verified on bottles selected sequentially over the whole bottling process.

## **Homogeneity tests**

### **APAT-RM004 (compost)**

The homogeneity test was carried out on 10 different units (bottles) sequentially selected over the whole bottling process. This study has been carried by measuring the total contents of C by CHN-S [1] considering a sample intake of 0.02g and by the determination of the Hg content by direct mercury analyzer (DMA-80) [5] considering a sample intake of 0.5g. Both techniques achieve high precision levels and require little or no sample processing prior to analysis. This analytical technique also eliminates uncertainty associated with sample processing. The within-bottle homogeneity was assessed by replicate determinations on the content of one bottle: the analytes (C and Hg) were determined by analyzing 30 sub-samples taken from one bottle. The homogeneity determinations were also performed to define the variations between bottles: 3 sub-samples were taken in each of 10 bottles selected during the bottling procedure [6] [7] [8] [9]. The results of the homogeneity tests are reported in Table 1. The homogeneity was verified using the analysis of variance test ANOVA [10]. The differences were considered significant at p<0.05. The “between bottles” showed no significant differences from the “within-bottles” tests for the analytes considered. The material was thus considered suitable to be used for external and internal quality control in the analytical laboratories, at the sample intakes considered for the tested analytes.

	Hg (mg kg <sup>-1</sup> )		C (g kg <sup>-1</sup> )	
	APAT RM004		APAT RM004	
	<i>within</i>	<i>between</i>	<i>within</i>	<i>between</i>
Weight (g)	0.5	0.5	0.02	0.02
Mean	0.236	-	359.38	-
Grand mean	-	0.236	-	366.01
Standard deviation	0.010	0.004	0.97	1.08
CV (%)	4.3	1.7	0.3	0.3
Number of samples	30	30	30	30
Number of bottles	1	10	1	10

**Table 1.**

APAT-RM004 reference material (compost).  
Homogeneity test for total carbon and mercury

Analyte	Robust standard deviations ( $\hat{\sigma}$ ) (mg kg <sup>-1</sup> d.w.)
As	1.5
Cd	0.2
Co	2.5
Cr	131
Cu	13
Hg	0.16
Mo	4.1
Ni	47
Pb	22
Se	0.51
Zn	25

**Table 2.**

APAT-RM004 reference material (compost).  
Robust standard deviations from the proficiency test



The APAT-RM004 reference material was used in a Proficiency Test (PT) with a number of laboratories participating ranging from 30 to 63 per analyte. The following metals were determined: As, Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Se, Zn. After the PT, the homogeneity of the material was checked for these analytes using the procedure suggested in the Appendix B of ISO 13528 [11]. The “between bottles” standard deviation ( $S_{bb}$ ) was compared with the robust standard deviation of the PT ( $\hat{\sigma}$ ). The “between-bottles” standard deviation should satisfy  $S_{bb}/\hat{\sigma} < 0.3$ .  $S_{bb}$  was determined using a one-way ANOVA test on As, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Zn concentrations measured in 30 sub-samples taken from one bottle (“within-bottles” homogeneity test) and three sub-samples in each of ten different bottles selected during the bottling procedure (“between-bottles” homogeneity test). The metal concentrations in the sub-samples were measured by inductively coupled plasma mass spectrometry (ICP-MS) after *aqua regia* digestion assisted by microwave with the control of temperature and pressure [12]. Table 2 reports the robust standard deviations of the PT ( $\hat{\sigma}$ ). The ratios reported in Table 3 range between 0.01 and 0.2. This confirms the fitness for purpose of the homogeneity of the material.

Analyte	$S_{bb}/\hat{\sigma}$
As	0.06
Cd	0.2
Co	0.02
Cr	0.04
Cu	0.1
Mo	0.02
Ni	0.01
Pb	0.2
Zn	0.2

**Table 3.**

APAT-RM004 reference material (compost).  
Confirmation of the “between-bottles” homogeneity

*APAT-RM005 (agricultural soil)*

The homogeneity for APAT-RM005 (agricultural soil) was carried out using the same procedures reported for APAT-RM004. Sample intake for Hg determination was 0.25g, while for total C was 0.02g. The metal concentrations in the sub-samples were measured by inductively coupled plasma mass spectrometry (ICP-MS) after *aqua regia* and hydrogen peroxide digestion assisted by microwave with the control of temperature and pressure. Tables 4 and 5 report the results of homogeneity test.

Table 6 reports the results of the “between-bottles” homogeneity confirmation assessed after the proficiency test.

	Hg (mg kg <sup>-1</sup> )		C (g kg <sup>-1</sup> )	
	APAT RM005		APAT RM005	
	<i>within</i>	<i>between</i>	<i>within</i>	<i>between</i>
Weight (g)	0.25	0.25	0.02	0.02
Mean	0.102	-	23.2	-
Grand mean	-	0.100	-	23.2
Standard deviation	0.005	0.002	0.10	0.06
CV (%)	5.1	1.8	0.5	0.3
Number of samples	30	30	30	30
Number of bottles	1	10	1	10

**Table 4.**

APAT-RM005 reference material (agricultural soil).  
Homogeneity test for total carbon and mercury

Analyte	Robust standard deviations ( $\hat{\sigma}$ ) (mg kg <sup>-1</sup> d.w.)
Cd	0.18
Cr	125
Cu	7
Fe	3200
Hg	0.04
Mn	112
Ni	72
Pb	6
Zn	12

**Table 5.**

APAT-RM005 reference material (agricultural soil).  
Robust standard deviations from the proficiency test

Analyte	$S_{bb}/\hat{\sigma}$
Cd	0.06
Cr	0.09
Cu	0.1
Hg	0.03
Ni	0.1
Pb	0.1
Zn	0.2

**Table 6.**

APAT-RM005 reference material (agricultural soil).  
Confirmation of the “between-bottles” homogeneity

## Stability tests

### APAT-RM004 (compost)

The short term stability studies of APAT-RM004 reference material was monitored for all the time in which the Proficiency Test was running (3 months), by measuring the C and Hg content. The isochronous method [13] was applied. The method requires that the short-term stability studies are run usually at two different storage temperatures (20 and 40 °C) at least up to the 3 months of production that all measurements be carried out under repeatability condition: i.e. in one run with one calibration, to avoid that the estimated uncertainty due to instability is unnecessarily enlarged due to the reproducibility effects in the results during stability testing.

At the start of the stability test, 25 bottles were stored at a reference temperature (-18 °C) at which it is assumed that no instability is encountered. Additional 5 bottles were stored at +20 °C and 5 bottles at +40 °C. After 1, 2 and 3 months, 5 bottles were transferred from -18 °C to +20 °C and 5 bottles from -18 °C to +40 °C. After three months 3 sub-samples collected from each bottle have been measured in one run. For each temperature (+20 and +40 °C), the following parameters were assessed:

- standard deviations between the bottles stored at the same temperature for the same time interval, the mean value of concentration and the coefficient of variation (CV %);
- standard deviations between the mean values of concentration of bottles stored for different time periods, the mean values and the coefficient of variation (CV %);
- ratios of the mean values of measurements on bottles stored at +20 °C and +40 °C, respectively, and the mean values of measurements on samples stored at -18 °C for the same period [7];
- linear regression of the above mentioned ratios, the uncertainty contribution due to the material stability [7];
- analysis of variance (ANOVA) to assess the influence of the storage period at +20 °C and +40 °C on the stability of the material.

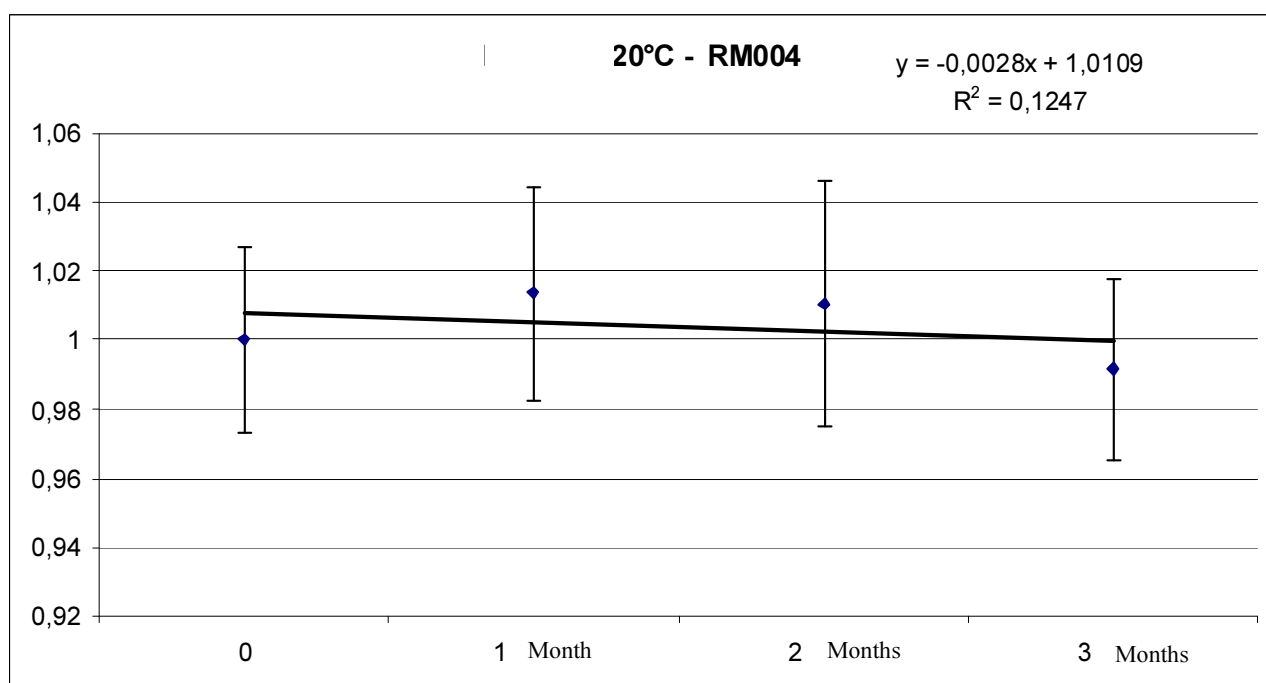
Table 7 reports the results of the stability study for APAT-RM004 reference material. The coefficient of variations are comparable with the repeatability of C and Hg measurements. On the basis of these results the material can be considered stable.

	Hg		C
	APAT RM004		APAT RM004
Weight (g)	0.3	Weight (g)	0.02
Regression 20°C (R <sup>2</sup> )	0.12	Regression 20°C (R <sup>2</sup> )	0.57
S <sub>stab</sub> 20°C (mg kg <sup>-1</sup> )	0.004	S <sub>stab</sub> 20°C (g kg <sup>-1</sup> )	2.6
CV% 20°C	1.0	CV% 20°C	0.7
Regression 40°C (R <sup>2</sup> )	0.5	Regression 40°C (R <sup>2</sup> )	0.9
S <sub>stab</sub> 40°C (mg kg <sup>-1</sup> )	0.003	S <sub>stab</sub> 40°C (g kg <sup>-1</sup> )	0.8
CV% 40°C	1.2	CV% 40°C	0.4

**Table 7**

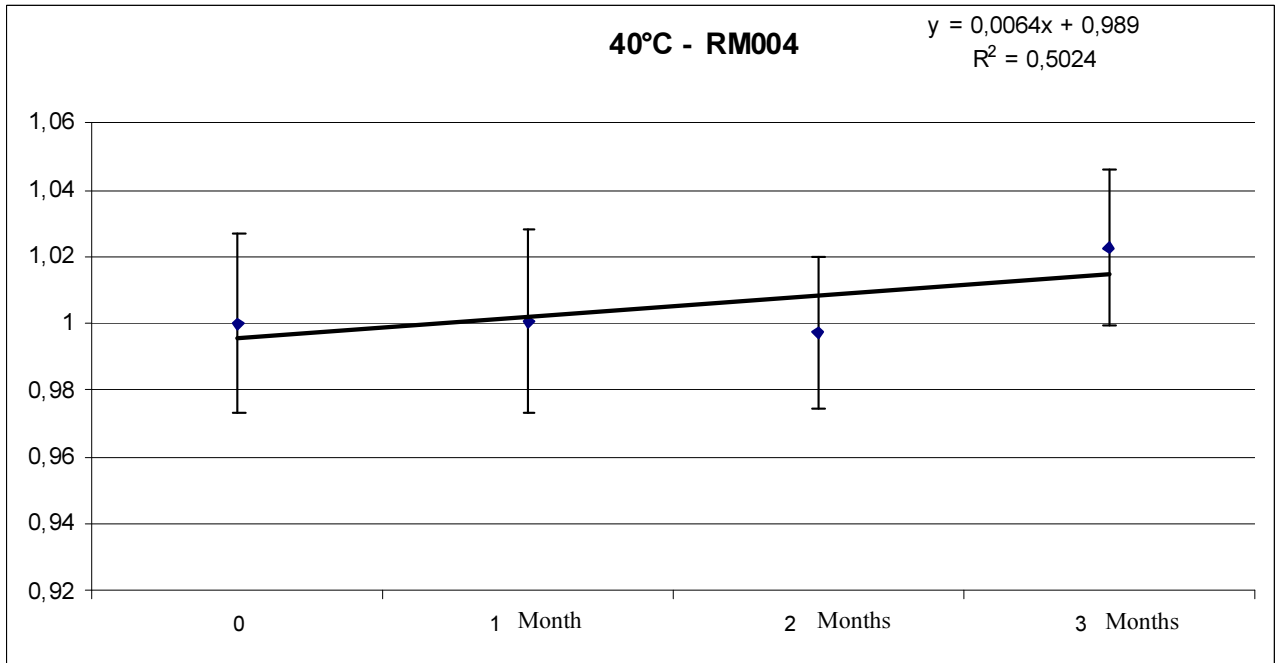
APAT-RM004 reference material (compost).  
Results of the short term stability test

Figures 1 and 2 show the regression for Hg at 20 and 40 °C APAT-RM004 reference material (compost).



**Figure 1.**

APAT-RM004 reference material (compost).  
Mercury stability regression line at 20°C



**Figure 2.**

APAT-RM004 reference material (compost).  
Mercury stability regression line at 40°C

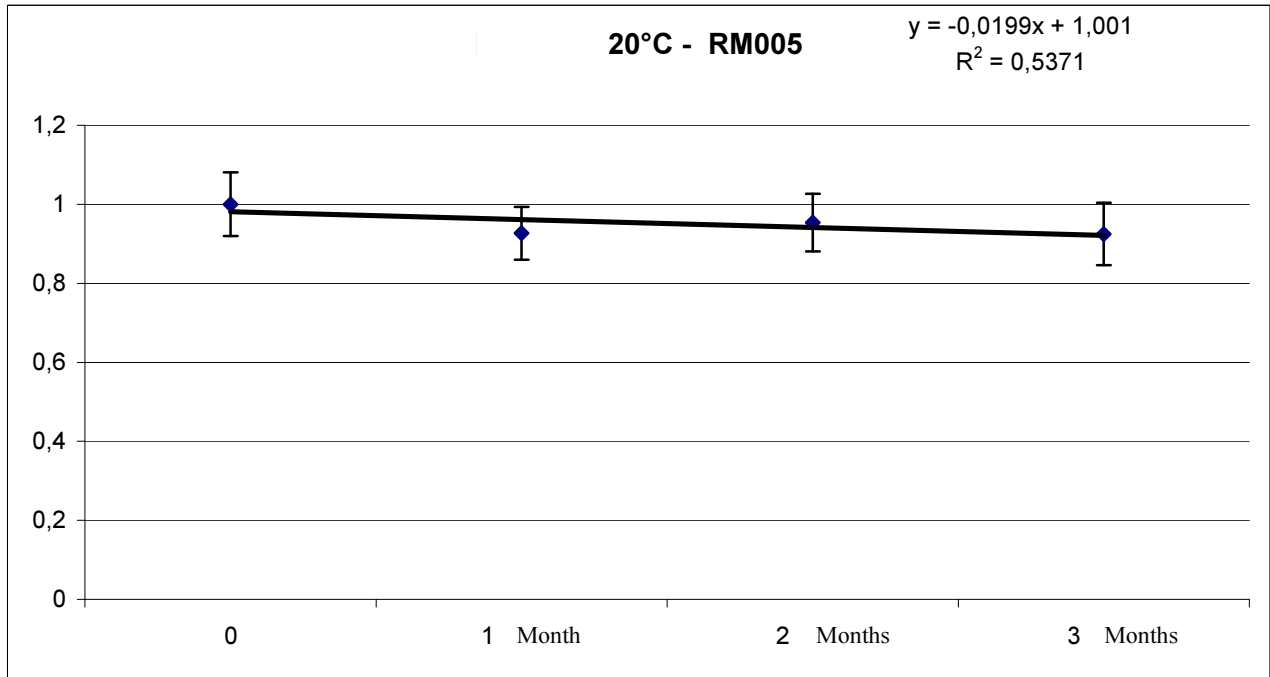
*APAT-RM005 (agricultural soil)*

The stability for APAT-RM005 (agricultural soil) was carried out using the same procedures reported for APAT-RM004. Table 8 reports the results of the stability study for APAT-RM005. The coefficient of variations are comparable with the repeatability of C and Hg measurements. On the basis of this results the material can be considered stable. Figure 3 and 4 show the regression for Hg at 20 and 40 °C.

	Hg		C
	APAT RM005		APAT RM005
Weight (g)	0.3	Weight (g)	0.02
Regression 20°C (R <sup>2</sup> )	0.54	Regression 20°C (R <sup>2</sup> )	0.14
S <sub>stab</sub> 20°C (mg kg <sup>-1</sup> )	0.004	S <sub>stab</sub> 20°C (g kg <sup>-1</sup> )	0.132
CV% 20°C	3.7	CV% 20°C	0.4
Regression 40°C (R <sup>2</sup> )	0.053	Regression 40°C (R <sup>2</sup> )	0.05
S <sub>stab</sub> 40°C (mg kg <sup>-1</sup> )	0.006	S <sub>stab</sub> 40°C (g kg <sup>-1</sup> )	0.15
CV% 40°C	3.6	CV% 40°C	0.4

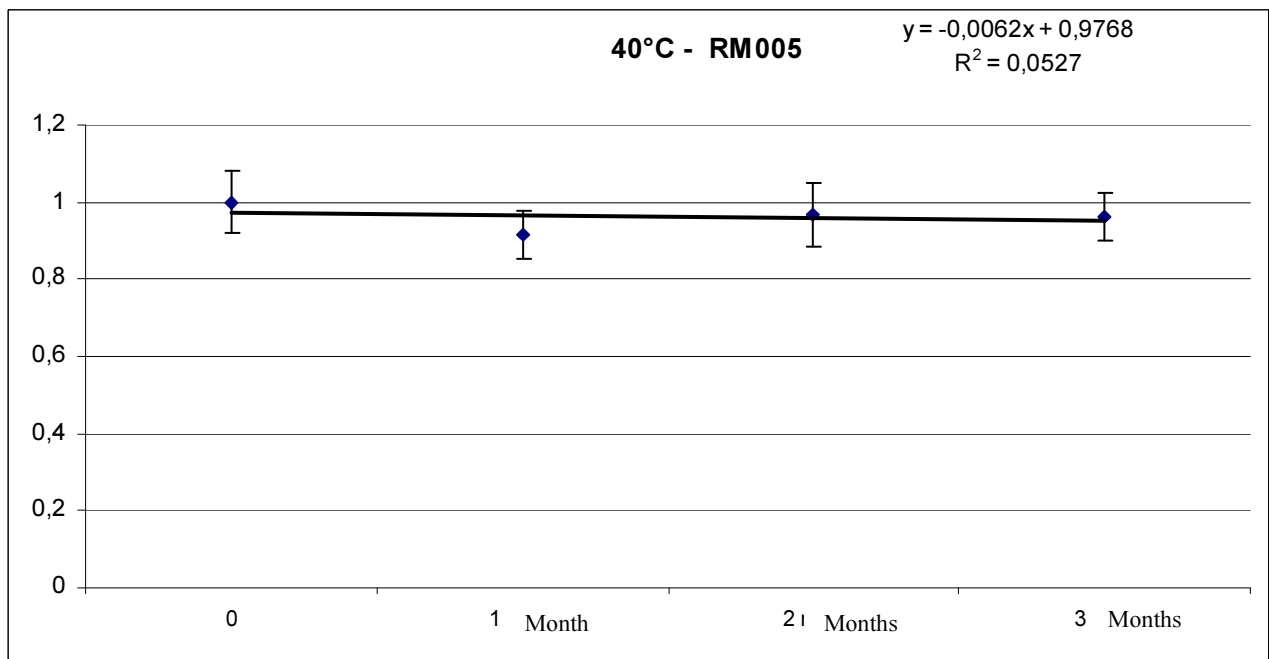
**Table 8**

APAT-RM005 reference material (agricultural soil).  
Results of the short term stability test



**Figure 3.**

APAT-RM005 reference material (agricultural soil).  
Mercury stability regression line at 20°C



**Figure 4.**

APAT-RM005 reference material (agricultural soil).  
Mercury stability regression line at 40°C

## Assignment of the property values

### APAT-RM004 (compost)

The property values and associated total combined uncertainties were assigned according to ISO Guides 35 [7] for the different analytes, by characterization in expert laboratories. Expert laboratories having demonstrable competence in the determination of the measurands were selected. The laboratories used a given method for digestion of samples (*aqua regia*) [13]: 1g of dried sample was extracted with a hydrochloric/nitric acid mixture by standing for at least 12 h at room temperature, followed by boiling for 2 h. The extract was clarified and the extracted elements determined. The property values for each analyte were assessed from the results of at least 12 laboratories (Se) and at most 29 laboratories (Pb and Zn) using a robust statistics method (Algorithm A) [9]. Table 9 reports the assigned values for each analyte and the associated uncertainty with a cover factor  $k=1$

Analyte	Property values mg kg <sup>-1</sup> d.w.
As	5.8 ± 0.4
Cd	0.45 ± 0.04
Co	8 ± 1
Cr	426 ± 28
Cu	96 ± 3
Hg	0.35 ± 0.06
Mo	8.2 ± 0.8
Ni	217 ± 7
Pb	106 ± 9
Se	0.5 ± 0.1
Zn	188 ± 7

**Table 9.**

APAT-RM004 reference material (compost).

Property values assigned by expert laboratories using *aqua regia* digestion method

The uncertainty expressed as  $1 \sigma$  ( $k=1$ )

*d.w.* = dry weight

Low recoveries for Cr are well known and are associated with the presence of insoluble refractory Cr minerals such as chromospinel and chromite ( $\text{FeCr}_2\text{O}_7$ ). These minerals, frequently occurring in geological materials, are very difficult to dissolve. Cr determined by INAA on RM004 is  $505 \pm 9.6$ .

### APAT-RM005 (agricultural soil)

The property values stability for APAT-RM005 (agricultural soil) was assigned using *aqua regia* digestion method by expert laboratories as reported for APAT-RM004. The number of expert laboratories involved ranges from 15 (Hg) to 30 (Ni, Cu). Table 10 reports the assigned values for each analyte and the associated uncertainty with a cover factor  $k=1$ .

The properties value for As was derived from the results of INAA carried out on 100 sub-samples of the same bulk material used for APAT-RM005 production.

Element	Property values mg kg <sup>-1</sup> d.w.
As (*)	11 ± 1
Cd	0.63 ± 0.07
Cr	659 ± 19
Cu	48 ± 1
Fe	25500 ± 939
Hg	0.18 ± 0.03
Mn	1174 ± 35
Ni	376 ± 10
Pb	33 ± 2
Zn	89 ± 3

**Table 10.**

APAT-RM005 reference material (agricultural soil).  
 Property values assigned by expert laboratories using *aqua regia* digestion method  
 The uncertainty expressed as 1  $\sigma$  (k=1)  
*d.w.* = dry weight  
 (\*) Values assigned by INAA measurement

Low recoveries for Cr are well known and are associated with the presence of insoluble refractory Cr minerals such as chromospinel and chromite (FeCr<sub>2</sub>O<sub>7</sub>). These minerals, frequently occurring in geological materials, are very difficult to dissolve. For RM005 the bias due to *acqua regia* method has been calculated on the basis of INAA measurements on soil used for the preparation of RM005. The assigned value for chromium corrected for the bias of *aqua regia* method is 1030±30.

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## Homogeneity test and characterisation of radionuclides in PT samples

### 1- Test sample 01

The test sample 01 is an IAEA reference material, its homogeneity test and characterization is described in reference [4] activities were corrected to the reference date 01-07-2005.

### 2- Test sample 03

The test sample 03 is a soil collected in China with very low activity level of man made radionuclides. The material had been milled and passed through a sieve of 0.2 mm, then homogenised before bottling. A homogeneity test was performed on the raw material, by measuring  $^{40}\text{K}$ ,  $^{137}\text{Cs}$  and  $^{238}\text{U}$  in 10 sub-samples taken from the bulk material. The measurements were performed in the gamma-spectrometry laboratory of the IAEA Laboratories Seibersdorf [6] and Hungarian National Institute for Food Inspection. The homogeneity of the bulk material was checked for a sample mass of 50g, the relative standard deviations were 6.6% and 0.9% for  $^{137}\text{Cs}$  and  $^{40}\text{K}$ , respectively. This proves that the level of the homogeneity of the bulk material fits for the purpose of this proficiency test.

The Within-bottle homogeneity test results are shown in table 1.

Within-bottle homogeneity check Soil sample 03 RAS/2/011						
Results are in form of cps $\pm$ 1 $\sigma$ RSD (%) for 50g aliquots taken from bottle no. 6						
Sample code	Count rate cps $\pm$ 1 $\sigma$ (%)					
	$^{40}\text{K}$		$^{137}\text{Cs}$		$^{238}\text{U}$	
	x100	%	x100	%	x1000	%
1/6	1.92	3	4.24	2	6.46	19
2/6	2	3	4.23	2	6.88	13
3/6	1.9	3	4.4	2	6.65	24
4/6	1.85	3	4.31	2	6.19	19
5/6	1.85	3	4.25	2	7.55	19
Average $\pm$ 1 $\sigma$ (%)	1.9	3	4.29	2	6.746	8
Relative standard deviation (%)	1.9		1.2		7.6%	

**Table 1.**

#### Within-bottle homogeneity test results

The target values were estimated according to measurements which were performed in the gamma-spectrometry laboratory of the IAEA Laboratories Seibersdorf [6], the material was found to contain  $2.6 \pm 0.2$  Bq/kg d.w. of  $^{137}\text{Cs}$  (Ref. date: 2005-07-01) and  $716 \pm 36$  Bq/kg d.w. of  $^{40}\text{K}$ . The moisture content determined at 105°C. was found to be  $2.4 \pm 0.2$  %.

### 3- Test samples 02 and 04

Test samples 02 and 04 are originated from Italian reference soil material APAT-RM-05 prepared by the Italian Agency for Environmental Protection (APAT). The material preparation is described elsewhere [1].

The radionuclides target values were determined according to the analysis performed in three laboratories:

- the Hungarian National Food Investigation Centre (NFII), Budapest, Hungary (IAEA collaborating centre);
- the Korea Institute of Nuclear Safety (KINS), Korea;
- the University of Roma Tre, Department of Physics, Roma, Italy.

$^{40}\text{K}$  and  $^{137}\text{Cs}$  were analysed by gamma-spectrometry, while  $^{238}\text{U}$  was determined by alpha-spectrometry and ICP-MS. Each laboratory was asked to perform at least 4 measurements for each radionuclide.

Target values and associated combined uncertainties were estimated by the method of mean of the means as described in ISO Guide 35. The target values and associated combined uncertainties of  $^{40}\text{K}$ ,  $^{137}\text{Cs}$  and  $^{238}\text{U}$  in the test samples 02 and 04 are listed in table 2.

Target values and associated combined uncertainty Bq/kg dry weight			
	$^{40}\text{K}$	$^{137}\text{Cs}$	$^{238}\text{U}$
Mean of Laboratory 01	301±5.3	12.9±0.45	38.4±0.91
Mean of Laboratory 02	295±13.1	11.27±0.28	39.9±1.02
Mean of Laboratory 03	335±12.3	11.97±0.75	-
Mean of means	307±17	12.1±0.82	39.2±1.09
Combined uncertainty	9.87	0.47	0.77
Combined uncertainty (%)	3.2	3.9	2.0

**Table 2.**

The target values and associated combined uncertainties of  $^{40}\text{K}$ ,  $^{137}\text{Cs}$  and  $^{238}\text{U}$  in the test samples 02 and 04

## Reference

1. Maria Belli, Stefania Balzamo, Sabrina Barbizzi, Damiano Centioli, Paolo de Zorzi, Chiara Galas, Stefania Gaudino, Teresa Guagnini, Alessandra Pati, Cristiano Ravaioli, Silvia Rosamilia, Giovanna Sentina, Production and characterization of APAT-RM004 (compost) and APAT-RM005 (agricultural soil) matrix reference materials, Italian Environmental Protection Agency (APAT) - Environmental Metrology Service, Via di Castel Romano, 100 - 00128 Roma (Italy).

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## **Appendix E: Technical information provided by the participants**

The technical information provided by the participants on the analytical procedures used in their own laboratory is compiled in this Appendix 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 technical information provided by the participants was scanned in the same format as it was received without any modification or editing.