PINSTECH - 195

Determination of Natural Levels of Radionuclides In Proposed Mushroom Reference Material (A Proficiency Test Exercise)

SHAHIDA WAHEED
ASMA RAHMAN
NAILA SIDDIQUE
SHUJAAT AHMAD
JAMSHED HUSSAIN ZAIDI

Nuclear Chemistry Division
Pakistan Institute of Nuclear Science and Technology
P.O. Nilore, Islamabad

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Abstract

A proficiency test (PT) was organized within the framework of International Atomic Energy Agency (IAEA) project INT/1/054, entitled "Preparation of Reference Materials and Organization of Proficiency Test Rounds". This exercise served to estimate the proficiency of the analytical laboratories from participating countries.

This report presents the results of the proficiency test exercise on the proposed Mushroom Reference Material for the determination of natural levels of radionuclides. Laboratories from 6 different countries submitted data on the following three radionuclides:

^{134}Cs, ^{137}Cs, ^{40}K

Results for ^{134}Cs, ^{137}Cs and ^{40}K in the mushroom reference material were reported by three or more participating laboratories and could be subjected to statistical evaluation. The original data of these radionuclides was subjected to a computer program "Histo Version 2.1" provided by IAEA. The four outlier tests i.e. Dixon, Grubbs, Skewness and Kurtosis were applied to the data sets. All values for these three radionuclides were accepted by the software. Consensus (overall) mean values, absolute standard deviation, relative standard deviation, standard error, median and range of values for these three radionuclides have been are obtained (at significance level 0.05). The consensus mean values and confidence intervals are given below.

^{134}Cs: 4.4 Bq/kg	(3.4-5.3 Bq/kg)
^{137}Cs: 2899 Bq/kg	(2740-3058 Bq/kg)
^{40}K: 1136 Bq/kg	(1046-1226 Bq/kg)

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1. Introduction

Proficiency tests are primarily designed to assess the accuracy and precision measurement through the use of inter-laboratory comparisons. These test schemes are organized individually for the analysis of a certain analyte(s) in a specific matrix. These schemes provide laboratories with an objective means of assessing and demonstrating the reliability of the data they are producing.

A proficiency test (PT) was organized within the framework of the International Atomic Energy Agency (IAEA) project INT/1/054, entitled "Preparation of Reference Materials and Organization of Proficiency Test Rounds". The aim of this project was to prepare, homogenize and characterize a reference material for proficiency tests which will serve as a pilot study for the participant countries for future organization of such tests in their respective countries. This exercise would also serve to estimate the proficiency of the analytical laboratories of the countries participating in this project for the determination of various constituents such as radionuclides and heavy metals.

After various considerations mushrooms were selected as a suitable material for this purpose. Mushrooms have a well-established place in European cuisine and are also gaining popularity at the international level as an important source of dietary protein. These not only accumulate heavy and alkaline metals but also contain a significant amount of ^{137}Cs. Mushrooms were collected from Poland where they are easily available in the forests. Candidate mushroom species were assessed on basis of appropriate content of ^{137}Cs for preparation of the reference material.

Six countries viz. Brazil, Hungary, Korea, Pakistan, Poland and Syria participated and submitted their data for the determination of radionuclides in edible mushroom material in this proficiency test exercise. From Pakistan the Neutron Activation Analysis (NAA) Laboratory of Nuclear Chemistry Division (NCD), Pakistan Institute of Nuclear Science and Technology (PINSTECH) participated in this activity.

2. Scope of the study

The participants from selected laboratories were supplied bottles (5 or 10 on request) containing samples of 20 g each and were asked to perform analysis of the samples in their countries. It was decided that the participating laboratories would report data to the Agency and to Poland for evaluation and compilation of the report. However in the final project coordinators meeting in Pretoria, South Africa, Pakistan was given the assignment to prepare the final proficiency report for radionuclides in the mushroom reference material.

The NAA Laboratory at PARR-2, PINSTECH is one of the few laboratories in Pakistan which have been formally accredited by the Pakistan National Accreditation Council (PNAC), under ISO/IEC-17025 quality standards. This laboratory has been regularly participating in IAEA Analytical Quality Control exercises to maintain confidence in our analytical capabilities On the basis of its outstanding, performance in different intercomparison exercises and proficiency test rounds, IAEA designated this laboratory a Regional Resource Unit (RRU) for Asia and the Pacific region.

For proficiency test exercise, the participants were requested to make at least three, but preferably six independent determinations and to report mean values, uncertainties and limit of detection for each radionuclide. The results from 6 different countries were received. Three laboratories determined ^{134}Cs, all 6 determined ^{137}Cs and 6 determined ^{40}K.

3. Description of the material

The samples usually used for PT have an average weight of 10-20 g. Approximately 100-200 g of fresh mushrooms are needed for this weight. As it was not possible to supply the participants with bulk samples, therefore selection of mushrooms for this exercise was made considering appropriate accumulation of ^{137}Cs as a critical feature, which depends on kind and place

of growth. For this ^{137}Cs was determined in different varieties of wild mushrooms.

The results of ^{137} Cs in wild edible mushrooms is as follows:

Mushroom type	Units	Activity concentration ± uncertainty (1s)
• Xerocomus Badius	Bq/kg dry mass	2700 ± 70
• Boletus Luteus	Bq/kg dry mass	1300 ± 60
• Boletus Edulis	Bq/kg dry mass	90 ± 4
• Psaliota Campestris	Bq/kg dry mass	40 ± 2

On the basis of high ^{137}Cs contents in wild mushrooms, Xerocomus Badius, family Boletaceae was selected for the study. This particular mushroom type was collected by a group of trained people from a forest in a non-contaminated rural area in northern part of Poland. More than 200 kg of fresh mushrooms were collected. The mushrooms were first cleaned to remove dust, soil and attached mosses. The feet part of mushrooms which is embedded in soil were cut and removed. Mushrooms were cut into smaller pieces and air dried in a dryer at a temperature of 25-60°C according to standard procedure used by food concentrate producers. Dried mushrooms were milled in a centrifugal mill and sieved. Particles approximately below 1mm diameter were collected.

The mushroom material was then sent to the Institute for Reference Materials and Measurements (IRMM) Laboratory, Geel, Belgium for final preparation. Further processing of this material was performed at this institute by the participants of the project under IAEA and IRMM experts in a "Workshop on Preparation of In-house Reference Materials) from 07-11 July 2003.

- The results were of questionable quality when: $2<|Z|<2$
- The measurement was regarded out of acceptable range when: $|Z| \geq 3$

This type of score represents a simple method of giving each participant a normalized performance score for bias. This method of assessing laboratories has been accepted as a standard for ISO/IUPAC.

4.1.3. U-test

The value of U-test score was calculated according to the following equation

$$u_{test} = \frac{|Value_{mean} - Value_{analyst}|}{\sqrt{Unc^2_{mean} + Unc^2_{analyst}}} \qquad \text{Eq. 3}$$

where Unc is the uncertainty

The calculated u-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:

Condition	Probability	Status
$u < 1.64$	Greater than 0.1	The reported result does not differ significantly from the expected value
$1.95 > u > 1.64$	Between 0.1 and 0.05	The reported result probably does not differ significantly from the expected value
$2.58 > u > 1.95$	Between 0.05 and 0.01	It is not clear whether the reported result differs significantly from the expected value

3.29 > u > 2.58	Between 0.01 and 0.001	The reported result is probably significantly different from the expected value
u > 3.29	Less than 0.001	The reported result is significantly different from the expected value

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 1.95 to determine if a result passes the test (u < 1.95).

4.2. Acceptance criteria

The results were evaluated against the following acceptance criteria for accuracy and precision and assigned the status "passed" or "rejected" accordingly. A result must pass both criteria to be assigned the final status of "passed".

4.2.1 Accuracy: result passes at 95% confidence level if:

$$\left|Value_{mean} - Value_{analyst}\right| \leq 1.95 \times \sqrt{Unc^2_{mean} + Unc^2_{analyst}} \qquad \text{Eq. 4}$$

where Unc is the uncertainty

4.2.2 Precision: for the purpose of this study the result passes if:

$$\sqrt{\left(\frac{Unc_{mean}}{Value_{mean}}\right)^2 + \left(\frac{Unc_{analyst}}{Value_{analyst}}\right)^2} \times 100\% \qquad \text{Eq. 5}$$

is less than, or equal to twice the reproducibility standard deviation as given in the table for z-Scores using 95% confidence statistics.

5. Explanation of tables

5.1. Data tables

- Laboratory code number: Each laboratory was assigned a code number, which is the same throughout the whole report. These do not correspond to the sequence of the laboratories in the list of participants given at the end of the report and thus anonymity is secured.

- Laboratory mean: The arithmetic mean computed from all the individual results supplied by the participating laboratory.

- Laboratory standard deviation: The absolute and relative standard deviations were calculated if at least three results were reported by the participating laboratory.

- No. of determinations: The number of individual results for a given radionuclide as supplied by the participating laboratories.

5.2. Summary of results tables

The summary of the results for consensus radionuclides in mushroom reference material is given in Table 4. Most of the terms used in the summary tables have been already defined. The standard error (S. E.) is defined as the standard deviation of the mean values divided by the square root of the number of laboratory means.

5.3. Description of figures

Figures 1 to 3 present the distribution of results expressed as the analyst/consensus value ratio for ^{134}Cs, ^{137}Cs and ^{40}K respectively in the mushroom reference material. The results are organized according to the laboratory code in ascending order. The error bars used in the figures

represent the laboratory ratio ± combined standard uncertainty for the ratio. The horizontal dashed lines represent ± two standard deviation of the overall mean of these ratios.

6. Results and conclusions

Analysts were requested to report their result together with the corresponding combined standard uncertainty. It is the responsibility of the analyst to report an accurate and precise value and to provide a reliable estimate of the uncertainty. For calculation of critical values for accuracy and precision, the evaluation procedure used involves the reported value and its corresponding combined standard uncertainty. To determine the acceptance range for a result to pass the accuracy criterion for this proficiency test, we have set the two-tailed value for Students t distribution to the 0.05 significance level. The second criterion (precision) defines the maximum acceptable uncertainty which could be assigned to the reported value and was set as twice the reproducibility standard deviation (which expresses the inter-laboratory precision) for this exercise, again using 95% confidence statistics. These fairly large limits were applied keeping in view the small number of participating laboratories reporting small number of values.

As the material used was being analyzed for the first time no certified or reference property values were available and the target values were derived from the consensus means obtained from this proficiency test exercise. Hence only those three radionuclides which were reported by more than one laboratory i.e. ^{134}Cs, ^{137}Cs and ^{40}K could be assigned property values.

Only three laboratories reported results of analysis for ^{134}Cs. All seven values provided were accepted on the basis of outlier tests, resulting in a relatively large uncertainty. As a consequence of this large uncertainty all laboratory mean results passed the z-score, u-test score (Tables 5.a. to 5.c) and accuracy criteria. However the individual large uncertainty of laboratory C5 (39.62%) is reflected in the failed precision test (Tables 5.a to 6.a).

All six participating laboratories provided results for ^{137}Cs and 22 values were available. Concentration of this radionuclide was the highest and overall standard deviation of laboratory means was the lowest amongst the three selected radionuclides, reflecting best analytical results of this radionuclide. Using 95% confidence criteria all results passed in terms of accuracy and precision (Tables 5.b to 6.b).

A similar number of results were received for ^{40}K from all six participating laboratories. The uncertainties were slightly higher as compared to ^{137}Cs as was the range of values. However all reported values were acceptable on the basis of outlier tests. Results of accuracy and precision tests were acceptable for all laboratory means using 95% confidence statistics (Tables 5.c to 6.c).

The final summary of consensus means and associated confidence intervals (at significance level 0.05) for ^{134}Cs, ^{137}Cs and ^{40}K are presented in Table 7. Recommended values could not be formulated because of the very limited data.

Acknowledgements

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Table 1

Data of individual laboratory results of ^{134}Cs in Mushroom Reference Material

Lab. code	Mean value (Bq/kg)	Uncertainty (Bq/kg)	Relative Uncertainty (%)	Mass (g)	Dry/wet ratio
C1	3.70	0.90	24.32	NR	NR
C2	-	-	-	-	-
C3	-	-	-	-	-
C4	4.14	0.93	22.37	3.75	0.934
C5	5.30	2.10	39.62	9.63	0.930
C6	-	-	-	-	-

NR – Not reported

Table 2

Data of individual laboratory results of ^{137}Cs in Mushroom Reference Material

Lab. code	Mean value (Bq/kg)	Uncertainty (Bq/kg)	Relative Uncertainty (%)	Mass (g)	Dry/wet ratio
C1	2680.00	170.00	6.34	NR	NR
C2	2710.00	140.00	5.17	NR	NR
C3	3192.00	27.00	0.85	37.50	0.929
C4	3039.46	30.29	1.00	3.75	0.934
C5	2948.67	98.33	3.33	9.80	0.932
C6	2823.21	36.56	1.29	2.20	NR

NR – Not reported

Table 3

Data of individual laboratory results of ^{40}K in Mushroom Reference Material

Lab. code	Mean value (Bq/kg)	Uncertainty (Bq/kg)	Relative Uncertainty (%)	Mass (g)	Dry/wet ratio
C1	1130.0	80.0	7.1	NR	NR
C2	1001.0	50.0	5.0	NR	NR
C3	1140.0	22.0	1.9	37.50	0.929
C4	1319.3	53.4	4.0	3.75	0.934
C5	1183.7	95.5	8.1	9.80	0.932
C6	1040.0	136.0	13.1		

NR – Not reported

Table 4

Summarized results of the radionuclides ^{134}Cs, ^{137}Cs and ^{40}K in Mushroom Reference Material

Radionuclide	^{134}Cs	^{137}Cs	^{40}K
Overall mean (Bq/kg)	4.38	2898.9	1135.7
Abs. std. dev. (Bq/kg)	0.83	198.7	112.6
Rel. std. dev (%)	18.9	6.9	9.9
Standard error (abs)	0.50	81.1	46
Standard error (%)	10.9	2.8	4.0
Median (Bq/kg)	4.14	2885.9	1135.0
Range of values (Bq/kg)	3.70 - 5.30	2680.0 - 3192.0	1001.0 - 1319.3
Number of laboratories	3	6	6
No. of results averaged	7	22	22

Table 5a

Comparison of ^{134}Cs results against the consensus values in Mushroom Reference Material

Lab. Code	Mean (Bq/kg)	Uncertainty (Bq/kg)	Rel. uncertainty (%)	Analyst/consensus ratio	Rel. bias (%)	z-score	Status	u-score	Status
C1	3.70	0.90	24.32	0.84	-15.5	-0.8	Pass	0.6	Pass
C4	4.14	0.93	22.37	0.95	-5.5	-0.3	Pass	0.2	Pass
C5	5.30	2.10	39.62	1.21	21.0	1.1	Pass	0.4	Pass

Table 5b

Comparison of ^{137}Cs results against the consensus values in Mushroom Reference Material

Lab.Code	Mean (Bq/kg)	Uncertainty (Bq/kg)	Rel. uncertainty (%)	Analyst/consensus ratio	Rel. bias (%)	z-score	Status	u-score	Status
C1	2680.0	170.0	6.3	0.92	-7.6	-1.1	Pass	0.8	Pass
C2	2710.0	140.0	5.2	0.93	-6.5	-1.0	Pass	0.8	Pass
C3	3192.0	27.0	0.8	1.10	10.1	1.5	Pass	1.5	Fail
C4	3039.5	30.3	1.0	1.05	4.8	0.7	Pas	0.7	Pass
C5	2948.7	98.3	3.3	1.02	1.7	0.3	Pass	0.2	Pass
C6	2823.2	36.5	1.3	0.97	-2.6	-0.4	Pass	0.4	Pass

Table 5c

Comparison of ^{40}K results against the consensus values in Mushroom Reference Material

Lab. Code	Mean (Bq/kg)	Uncertainty (Bq/kg)	Rel. uncertainty (%)	Analyst/consensus ratio	Rel. bias (%)	z-score	Status	u-score	Status
C1	1130.0	80.0	7.1	1.00	-0.5	-0.1	Pass	0.0	Pass
C2	1001.0	50.0	5.0	0.88	-11.9	-1.2	Pass	1.1	Pass
C3	1140.0	22.0	1.9	1.00	0.4	0.0	Pass	0.0	Pass
C4	1319.3	53.4	4.0	1.16	16.2	1.6	Pass	1.5	Fail
C5	1183.7	95.5	8.1	1.04	4.2	0.4	Pass	0.3	Pass
C6	1040.0	136.0	13.1	0.92	-8.4	-0.8	Pass	0.5	Pass

Table 6a

Acceptance tests for accuracy and precision of ^{134}Cs results in Mushroom Reference Material

Lab. Code	Accuracy Criteria			Precision Criteria		Final status
	$\lvert Value_{mean} - Value_{analyst} \rvert$	$1.95 \times \sqrt{Unc^2_{mean} + Unc^2_{analyst}}$	Status	[%]	Status	
C1	0.7	2.2	Pass	30.8	Pass	Pass
C4	0.2	2.2	Pass	29.3	Pass	Pass
C5	0.9	3.97	Pass	43.9	Fail	Fail

Table 6b

Acceptance tests for accuracy and precision of ^{137}Cs results in Mushroom Reference Material

Lab. Code	Accuracy Criteria			Precision Criteria		Final status
	$\lvert Value_{mean} - Value_{analyst} \rvert$	$1.95 \times \sqrt{Unc_{mean}^2 + Unc_{analyst}^2}$	Status	[%]	Status	
C1	218.9	461.6	Pass	9.3	Pass	Pass
C2	188.9	429.1	Pass	8.6	Pass	Pass
C3	293.1	354.0	Pass	6.9	Pass	Pass
C4	140.6	354.8	Pass	6.9	Pass	Pass
C5	49.8	391.4	Pass	7.6	Pass	Pass
C6	75.7	356.7	Pass	7.0	Pass	Pass

Table 6c

Acceptance tests for accuracy and precision of ^{40}K results in Mushroom Reference Material

Lab. Code	Accuracy Criteria			Precision Criteria		Final status
	$\lvert Value_{mean} - Value_{analyst} \rvert$	$1.95 \times \sqrt{Unc^2_{mean} + Unc^2_{analyst}}$	Status	[%]	Status	
C1	5.7	243.8	Pass	12.2	Pass	Pass
C2	134.7	217.4	Pass	11.1	Pass	Pass
C3	4.3	202.4	Pass	10.1	Pass	Pass
C4	183.6	220.0	Pass	10.7	Pass	Pass
C5	48.0	260.5	Pass	12.8	Pass	Pass
C6	95.7	311.7	Pass	16.4	Pass	Pass

Table 6c

Acceptance tests for accuracy and precision of ^{40}K results in Mushroom Reference Material

Lab. Code	Accuracy Criteria			Precision Criteria		Final status
	$\lvert Value_{mean} - Value_{analyst} \rvert$	$1.95 \times \sqrt{Unc^2_{mean} + Unc^2_{analyst}}$	Status	[%]	Status	
C1	5.7	243.8	Pass	12.2	Pass	Pass
C2	134.7	217.4	Pass	11.1	Pass	Pass
C3	4.3	202.4	Pass	10.1	Pass	Pass
C4	183.6	220.0	Pass	10.7	Pass	Pass
C5	48.0	260.5	Pass	12.8	Pass	Pass
C6	95.7	311.7	Pass	16.4	Pass	Pass

Figure 1

Results for ^{134}Cs expressed as Analyst mean/Consensus mean ratio in Mushroom Reference Material

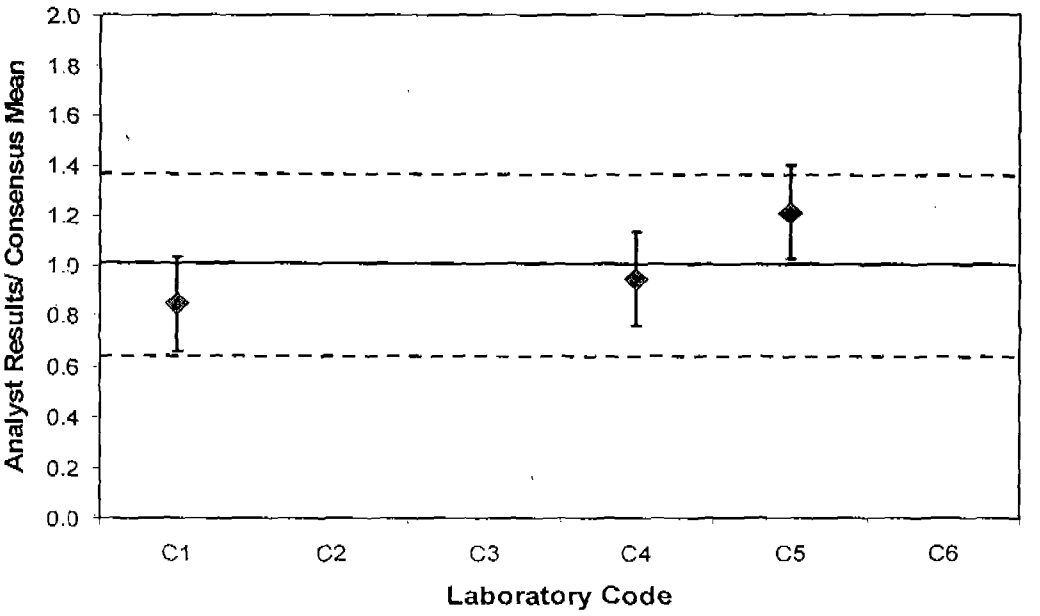

Figure 2

Results for ^{137}Cs expressed as Analyst mean/Consensus mean ratio in Mushroom Reference Material

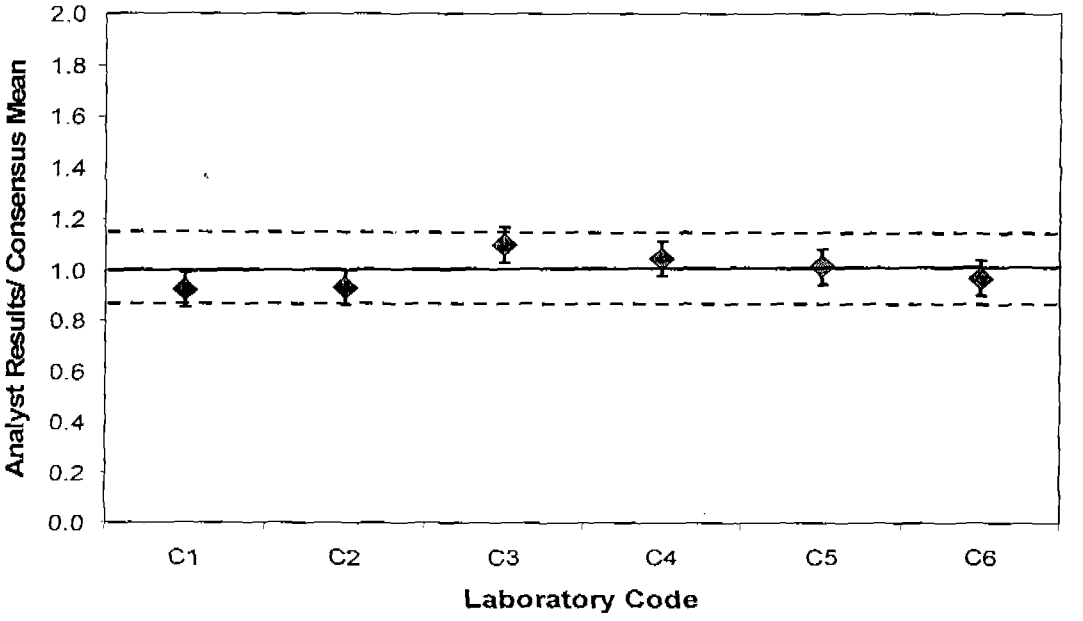

Table 7

Summary of mean and confidence interval for radionuclides in Mushroom Reference Material

Radionuclide	Mean Value (Bq/kg)	Confidence Interval (Bq/kg)
^{134}Cs	4.4	3.4-5.3
^{137}Cs	2899	2740-3058
^{40}K	1136	1046-1226

* Confidence intervals are for significance level 0.05

List of six participating laboratories for Proficiency Test Exercise on Radionuclides in Mushroom Reference Material (arranged alphabetically by country)

Brazil

Ms. Vera Akiko MAIHARA

Instituto de Pesquisas Energéticas e Nucleares IPEN/CNEN-SP

Av. Prof. Lineu Prestes 2242

055008-000 São Paulo, Brazil

Tel.: 0055 1 138169182

Fax: 0055 1 138169188

Email: vmaihara@curiango.ipen.br

Hungary

Mr. Sandor TARJAN

Ministry of Agriculture and Regional Development

National Food Investigation Institute

Mester út. 81

P.O. Box 1740

H-1465 Budapest, Hungary

Tel.: 0036 1 2901532

Fax: 0036 1 2960281

Email: tsmg @oevi.axelero.net. or ts-t@vnet.hu

Korea

Mr. Kun Ho CHUNG

Korea Atomic Energy Research Institute (KAERI)

150 Deokjin-dong, P.O. Box 105

Taejon 305-600

Yuseong, Republic of Korea

Tel.: 0082 42 8682389

Fax: 0082 42 8631289

Email: chungkh@kaeri.re.kr

Pakistan

Ms. Shahida WAHEED
Pakistan Atomic Energy Commission
Pakistan Institute of Nuclear Science and Technology (PINSTECH)
Nuclear Chemistry Division.
P.O. Box 1482, Nilore
Islamabad, Pakistan
Tel.: 0092 51 2207261
Fax: 0092 51 9290275
Email: skhalid161@yahoo.com
swaheed161@hotmail.com

Poland

Ms. Halina POLKOWSKA-MOTRENKO
Institute of Nuclear Chemistry and Technology
Dept. of Analytical Chemistry
Ul. Dorodna 16
PL-03-195 Warsaw, Poland
Tel.: 0048 22 8112737
Fax: 0048 22 8111532
Email: hpolkows@ichtj.waw.pl

Syria

Mr. Mohammad Said AL-MASRI
Atomic Energy Commission of Syria (AECS)
P.O. Box 6091
Damascus
Syrian Arab Republic
Tel.: 00963 11 2132582
Fax: 00963 11 6112289
Email: atomic@aec.org.sy