IAEA / AL / 164

Report on the SECOND ALMERA NETWORK COORDINATION MEETING and the ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE IAEA/SIE/01

Trieste, Italy, November 2005,

Seibersdorf, Austria, May 2006

الوكالة الدولية للطاقة الذرية
国 际 原 子 能 机 构
INTERNATIONAL ATOMIC ENERGY AGENCY
AGENCE INTERNATIONALE DE L'ÉNERGIE ATOMIQUE
МЕЖДУНАРОДНОЕ АГЕНТСТВО ПО АТОМНОЙ ЭНЕРГИИ
ORGANISMO INTERNACIONAL DE ENERGÍA ATÓMICA

IAEA / AL / 164

Department of Nuclear Sciences and Applications

Physics, Chemistry and Instrumentation Laboratory

Chemistry Unit

SECOND ALMERA NETWORK COORDINATION MEETING

International Centre for Theoretical Physics
Trieste (Italy)
15 November 2005

ALMERA SOIL SAMPLING INTERCOMPARISON EXERCISE (IAEA/SIE/01)

International Centre for Theoretical Physics
Trieste (Italy)

Ente Regionale per lo Sviluppo Agricolo del Friuli Venezia Giulia
Pozzuolo del Friuli, Udine, (Italy)
14-18 November 2005

Seibersdorf, Austria, May 2006

Contact information

Umberto Sansone
International Atomic Energy Agency (IAEA)
Department of Nuclear Sciences and Applications
Physics, Chemistry and Instrumentation Laboratory
Chemistry Unit
Agency's Laboratories Seibersdorf
A-2444 Seibersdorf, Austria

Tel.	:	+431 2600 28234
Fax	:	+431 2600 28222
E-mail	:	u.sansone@iaea.org

Content

ALMERA (Analytical Laboratories for the Measurement of Environmental Radioactivity) network coordination meeting

The second ALMERA network coordination meeting took place in Trieste (Italy) on 15 November 2005 and was hosted by the International Centre for Theoretical Physics (ICTP).

The overall aim of the meeting was to evaluate the current status of the ALMERA network laboratories and to help to improve their technical competence through harmonization of sampling, monitoring and measurement protocols and staff training. The meeting was also addressed to defining the structure of the ALMERA network and future proficiency tests and intercomparison trials to be organized by the IAEA to help the laboratories to maintain and improve the quality of their analytical measurements. 45 participants from 29 different institutions attended the meeting.

The meeting was chaired by Mr. Umberto Sansone, Head of the the IAEA Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in Seibersdorf (Austria) and by Mr. Claudio Tuniz, Special Adviser to the Director of the Abdus Salam International Centre for Theoretical Physics.

List of participants to the ALMERA network coordination meeting

Benesch Thomas	:	IAEA, Seibersdorf (Austria)
Sansone Umberto	:	IAEA, Seibersdorf (Austria)
Shakhashiro Abdulghani	:	IAEA, Seibersdorf (Austria)
Trinkl Alexander	:	IAEA, Seibersdorf (Austria)
Will Karin	:	IAEA, Seibersdorf (Austria)
Mantoura R. Fauzi	:	IAEA, Monaco (Principality of Monaco)
Tuniz Claudio	:	ICTP, Trieste (Italy)
Belli Maria	:	APAT, Environmental Protection Agency (Italy)
de Zorzi Paolo	:	APAT, Environmental Protection Agency (Italy)
Rosamilia Silvia	:	APAT, Environmental Protection Agency (Italy)
Aguirre Jaime	:	Comision Nacional de Seguridad Nuclear y Salvaguardias (Mexico)
Al-Masri Mohammad S.	:	Atomic Energy Commission of Syria
Arnold Dirk	:	Physikalisch-Technische Bundesanstalt (Germany)
Avramov Valentin	:	Kozloduy NPP (Bulgaria)
Battisti Paolo	:	ENEA (Italy)
Bellintani Roberto	:	SOGIN (Italy)

Bucur Cristina	:	NPP Cernavoda (Romania)
Chang Byung-Uck	:	Korea Institute of Nuclear Safety
Christensen Gordon C.	:	Institute for Energy Technology (Norway)
Clouvas Alexandros	:	Aristotle University (Greece)
Derkach Grygoriy	:	Ukrainian Hydrometeorological Institute (Ukraine)
Ermakov Alexander I.	:	SIA “RADON” (Russia)
Fathivand Khalili Mir Ali Asghar	:	Iranian Nuclear Regulatory Authority
Grubel Stefan	:	NPP Mochovce (Slovak Republic)
Guilhot Joelle	:	Aristotle University (Greece)
Harms Arvic	:	National Physical Laboratory (United Kingdom)
Kanivets Volodymyr	:	Ukrainian Hydrometeorological Institute (Ukraine)
Korun Matjaz	:	Josef Stefan Institute (Slovenia)
Kozar-Logar Jasmina	:	Josef Stefan Institute (Slovenia)
Kwakman Pieter J.M.	:	National Institute of Public Health & Environment (The Netherlands)
Ladygiene Rima	:	Radiation Protection Centre of Lithuania
Lebacq Anne Laure	:	Studiecentrum Voor Kernenergie (Belgium)
Lee Dong-Myung	:	Korea Institute of Nuclear Safety
Madruga Maria José	:	Instituto Tecnologico e Nuclear (Portugal)
Ocone Rita	:	APAT, Environmental Protection Agency (Italy)
Romani Sabrina	:	SOGIN (Italy)
Romero Lourdes	:	CIEMAT (Spain)
Stralberg Elisabeth	:	Institute for Energy Technology (Norway)
Tarjan Sandor	:	National Food Investigation Institute (Hungary)
Tirollo Taddei Maria Helena	:	Brazilian Nuclear Energy Agency
Toma Alexandru	:	Institute for Nuclear Research (Romania)
Tzibransky Russian	:	Kozloduy NPP (Bulgaria)
Varga Beata	:	National Food Investigation Institute (Hungary)
Vasarab Julius	:	NPP Mochovce (Slovak Republic)
Voelkle Hansruedi	:	Swiss Federal Office of Public Health

Origin of ALMERA network

The participants to the second coordination meeting (CRM) of the IAEA Co-ordinated research programme (CRP) on *"Rapid instrumental & separation methods for monitoring radionuclides in food and environmental samples*", held in Vienna (Austria) from 12 to 16 August 1991, had a strong desire to maintain the network of monitoring laboratories, which had been assembled in response to the IAEA's request for assistance with the International Chernobyl Project [1]. The participants discussed how to maintain this network of laboratories that can be called upon to assess the extent of radioactive contamination in both the environment and food in case of a radiological emergency, and recommended that a new IAEA CRP be established:

- to maintain a network of analytical laboratories with special skills and experience to provide assessments of radionuclide contamination in the environment in case of a radiological emergency;
- to conduct intercomparison and testing of technologies, employing standard reference materials and procedures, and field measurements at significantly contaminated areas; and
- to conduct training of Member States laboratory personnel through fellowships, special courses and workshops.

Based on these recommendations, in 1994 a new IAEA CRP on "*Development and selection of analytical techniques and procedures for measuring accidentally released radionuclides in the environment*" was established. The objectives of this new CRP were to conduct research and development on applicable methodologies for responding to accidental releases, and to improve and maintain the capabilities of the network of laboratories and provide training of individuals within Member States. Thus, the CRP served as a vehicle to maintain contact within the network of laboratories, while developing and transferring analytical techniques and procedures for measuring accidentally released radioactivity. The participants at the first CRM, held in Abu Dhabi (U.A.E.), from 4 to 7 December 1994, made the following recommendations regarding the network of laboratories [2]:

- a network of monitoring laboratories should be established with a core set of laboratories. The laboratories represented at this CRM could form the required core. The core could be subsequently be expanded to cover gaps in the capabilities of the network;
- the network of laboratories should be coordinated through the IAEA.

Based on these recommendations, the IAEA established in 1995 a world-wide network of analytical laboratories, the ALMERA network (**A**nalytical **L**aboratories for the **M**easurement of **E**nvironmental **RA**dioactivity), that could provide radioanalytical data that would be accepted internationally in support of the Agency's activities in the field of radiation protection and radiological assessment of areas affected by accidental or intentional release of radioactivity. The objective of ALMERA is to maintain a group of laboratories capable:

- of providing a broader world-wide emergency assistance coverage to address situations needing a speedy response in order to mitigate the consequences of a radiological emergency;
- of providing emergency response to neighbouring regions or other Member States;
- to develop and establish rapid and reliable radiochemical methods,
- to prepare guidelines for sampling and methods of analysis for use by Member States laboratories;
- to achieve comparability of results which should be traceable to the SI system;
- to become a source of reliable and consistent information and advice for government bodies;
- to train fellows from Member States laboratories;
- to participate in the certification of IAEA reference materials and international projects.

Without standard procedures and a common understanding, responses to radiological emergencies may differ markedly from country to country, and it may be difficult for countries to interpret and use each other's data. The resulting confusion and public mistrust could hamper recovery operations and catalyse severe socio-economic and political consequences. It must also be kept in mind that radioactive releases can originate several sources, including:

- nuclear power plants;
- nuclear-powered naval vessels or submarines;
- nuclear-powered satellites;
- the production, transportation, use and disposal of radioactive materials;
- the threat of nuclear terrorism.

The nomination of laboratories for membership in ALMERA has to be made by their governments and the IAEA has to be informed about the nominations through the Permanent Missions of the Member States to the Agency. There is no deadline for such a nomination.

The Agency's Seibersdorf Laboratory in Austria and its Marine Environment Laboratory in Monaco are members of the network. The Chemistry Unit of the Physics, Chemistry and Instrumentation (PCI) Laboratory in Seibersdorf is the central coordinator of the ALMERA network's activities and provides technical assistance to help the laboratories to improve their readiness in case of need.

A primary requirement of the ALMERA members is the successful participation in IAEA proficiency tests or intercomparison trials, specifically organized for ALMERA on a regular basis, to monitor and demonstrate the performance and analytical capabilities of the network members [3]. The first ALMERA intercomparison exercise was performed in 1996 and involved the measurement of primordial and anthropogenic radionuclides in soil and sediment samples. A second exercise (proficiency test) was organized in 2001 with labelled soil samples. The proficiency test materials consisted of a soil from China labelled with gamma-emitting radionuclides, actinides and ^{90}Sr at different activity concentration levels. 53 laboratories from 37 countries and five continents agreed to participate and reported measurement results.

The ALMERA network has already contributed to the successful completion of the IAEA international project involving the assessment of the radiological situation of the atolls of Mururoa and Fangataufa [4] and for the assessment of the environmental consequences of the use of Depleted Uranium (DU) as a result of the Gulf war [5].

On 17 November 1997, the first ALMERA workshop took place in Vienna [6]. The workshop was attended by 38 participants, 14 from the IAEA and 24 external participants, representing 15 different countries. The purpose of the workshop was to provide the participants with an opportunity to review and discuss the results of the intercomparison exercise performed in 1996 and to discuss and decide the role and function of the network.

Present status of ALMERA network and recommendations of the second workshop

ALMERA currently consists of 104 laboratories drawn from 66 countries, whose goal is “to provide accurate radionuclide analysis in environmental samples for Member States in the event of an accidental or intentional release of radioactivity”. The IAEA's Seibersdorf Laboratory in Austria and its Marine Environment Laboratory in Monaco are also members of the network. The Chemistry Unit of the Physics, Chemistry and Instrumentation (PCI) Laboratory in Seibersdorf is the central coordinator of the ALMERA network's activities.

The ALMERA participants recommended that at least one inter-laboratory exercise should be organised per year as a Proficiency Test. In case of any special event it may be extended to an additional run. The following matrices were proposed: soil, water, vegetation and air filter. These matrices are of interest for routine monitoring and emergency situations. The following analytical techniques were recommended to be tested during the proficiency tests every two years.

Method	**Purpose**
gamma-spectrometry (HPGe)	environmental samples, emergency situation
gamma-spectrometry (NaI(Tl))	emergency situation
gross alpha/gross beta	emergency situation (liquid samples)
TRU by alpha spectrometry	environmental samples
^{90}Sr	environmental samples, emergency situation
^{3}H	environmental samples, emergency situation
natural radionuclides	environmental samples

It was recommended to prepare proficiency testing materials by characterization in expert laboratories and assigning the target values and associated total combined uncertainties according to ISO Guides 30-35 [7].

Considering that the ALMERA network is designed to support radionuclide measurement in issues of international concern or emergency situations, where the measurement results should be ready in a short time, the working group recommended that the laboratories participating in the ALMERA proficiency tests report the results to the organizer according to the following time schedule:

Analytical method	**Reporting time**
gamma-spectrometry (HPGe)	3 days
gamma-spectrometry (NaI(Tl))	3 days
gross alpha/gross beta	3 days
TRU by alpha spectrometry	no recommendations
^{90}Sr	1 week
^{3}H	3 days

In addition to the above reporting times, the laboratories participating in the ALMERA proficiency tests will be also asked to report results to the organizer at the reporting time indicated for the IAEA world-wide open proficiency test (i.e. normal or longer-term reporting). It should be noted that the approach for evaluation of the results for the rapid and normal (longer-term) reporting time may be carried out on a different basis. This is because it cannot be expected that the same trueness and precision would be reached for a rapid analysis as for a longer-term analysis.

In the future interlaboratory exercises performed in the frame of the ALMERA network, the performance evaluation results will not be anonymous for those laboratories nominating to participate as ALMERA members. If an ALMERA member wants the evaluation result of his/her participation to remain anonymous, then he/she will have the option to only take part in the IAEA world-wide open proficiency test. In this case, however, his/her results will not be included in the ALMERA report. In addition the statistical approach used in the evaluation of the analytical results of the proficiency test for the ALMERA network will be adapted in future to take into consideration also the reporting time above recommended.

It was also recommended that, for each proficiency test, the target values should be released soon after the deadline for data submission for the world-wide test. It was also requested that other field intercomparison exercises (gamma-spectrometry, surface contamination, gamma-dose rate measurement, sampling in aquatic systems, etc.) should be organized.

It was recommended that the ALMERA network be subdivided into a number of regional groups. The groups should be defined by the IAEA according to the geographical distribution of participating laboratories (e.g. Europe, America, Asia, Africa). If a laboratory wishes to join another group for any reason it should be allowed to do so. A regional focal point for each regional group should be selected. The focal point should have good knowledge, experience, equipment, time, staff and infrastructure to do this job and to assume this responsibility to maintain the regional group alive. The IAEA will propose a new structure of the ALMERA network at the 2006 ALMERA coordination meeting.

The annual meeting with the ALMERA members should take place on rotation between the different regional groups. The objectives of the meetings are to evaluate the status of the laboratories, to improve their technical competence through standardization of sampling, monitoring and measurement protocols, and staff training. To increase interaction between the laboratories so that, in an emergency, they will be ready and able to work together, the participants recommended that IAEA should partially cover the expenses for the participation to the meeting of some of the laboratories belonging to the same regional group.

The participants suggested the following criteria to be an active member of ALMERA: the laboratories should participate in the proficiency tests specifically organized by the IAEA for ALMERA, present acceptable results, and do analyses on a routine basis.

In order to improve communication between members, the ALMERA participants recommended development of an ALMERA website to report ALMERA news, the minutes of the ALMERA meetings, the capabilities of the laboratories of the ALMERA members, the number of analyses performed per year, the proficiency tests (passed/failed); a set of standard methods for radionuclide analysis in environmental samples, including rapid measurement techniques, and a set of standard methods for environmental sampling.

Questionnaires will be sent to ALMERA members concerning the laboratories capabilities; needs (reference materials, proficiency tests, etc.); research activities in which they are already involved, and interest in the NORM area and related expertise, experience & capabilities.

On the basis of the Korea Institute of Nuclear Safety proposal, the IAEA agreed to co-operate in the 2006 ALMERA network coordination meeting, to be held in Daejeon (Korea), in October-November 2006.

Statistical approach used in the evaluation of the analytical results of the first proficiency test for the ALMERA network

In the following is reported the statistical approach used in the evaluation of the analytical results of the first proficiency test for the ALMERA network [3]. In the future, if necessary, the statistical approach will be modified to take into consideration the recommendations made by the ALMERA participants, concerning the reporting time to the IAEA of the proficiency test results.

The proficiency testing evaluation procedures will evaluate the trueness and the precision of the reported data and it will include 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.

In the ‘z-score” system, the participant’s result $Value_{Lab}$ is converted into a ‘z-score’ given by the equation:

$$z = \frac{(Value_{Lab} - Value_{IAEA})}{s}$$

where:

- $Value_{Lab}$ is the participant's measurement result
- $Value_{IAEA}$ and S are the assigned value and the measure of its variability (assigned standard deviation), correspondingly.

The model is used both when $Value_{IAEA}$ and S are derived from all the participant's measurement results, or when $Value_{IAEA}$ and s are assigned by the PT provider. In an ideal PT scheme, the value given to $Value_{IAEA}$ and s is determined by fitness for purpose: it represents the amount of uncertainty in the result that is tolerable in relation to the purpose of the data. It should be clear that here $Value_{IAEA}$ and S describe the end-user's requirements, not the data. It is then up to the PT organizer to put arbitrary limits of acceptability on the value of z. If the participants as a whole complied with the criterion, but no better, we would expect z to be roughly like a random normal variable with a mean of zero and a standard deviation of unity [N(0,1)]. That is why the laboratory performance is evaluated as satisfactory for $|z| \leq 2$, questionable for $2< |z| <3$, and unsatisfactory for $|z| \geq 3$. Such critical values mean that the deviations $|Value_{Lab} - Value_{IAEA}| \leq 2s$ can be accepted (at the normal distribution of x, and the level of confidence 95%), while the deviations $|Value_{Lab}-Value_{IAEA}| \geq 3s$ cannot (at the level of confidence 99.7% [8]). The drawback of z-scores is that the uncertainty of the participant's measurement result is not taken into account for the evaluation of performance.

Additional statistical transformations are also used when evaluating participating laboratories results such as:

$$relative\ \ bias = \frac{Value_{Lab} - Value_{IAEA}}{Value_{IAEA}} x100\%$$

$$Ratio = \frac{Value_{Lab}}{Value_{IAEA}} x100\%$$

It should be noted that since the only variable in these transformations is the laboratory result, populations of all three derived performance parameters have the same distribution as the of original results.

One of the important quality criteria of the analytical data is the measurement uncertainty, a result bias might be relatively small but its uncertainty should also fit for the purpose of the application. Therefore, the shortcoming of z-scores is that the uncertainty of the participant's measurement result is not taken into account for the evaluation of performance. For many applications in trace elements and radionuclides analysis it is very important to evaluate the uncertainty of the analytical results.

The IAEA scoring system is based on the calculation and interpretation of u_{tes}. Three primarily assumptions must be met when applying the $u_{test:}$

1. the assigned target value ($Value_{IAEA}$) is independent from the laboratory results obtained;
2. the laboratory results ($Value_{Lab}$) and the target value follow normal distributions described by mean and variance N($Value_{Lab}$, $Value_{Lab2}$) and N($Value_{IAEA}$, $Value_{IAEA2}$), respectively;
3. $Value_{Lab}$ and $Value_{IAEA}$ are expressed as combined standard uncertainties.

The value of the u_{test} score is calculated according to the following equation:

$$u_{test} = \frac{|Value_{IAEA} - Value_{Lab}|}{\sqrt{unc^2{}_{IAEA} - unc^2{}_{Lab}}}$$

Condition	Probability	Status
u < 1.64	> 0.1	The reported result does not differ significantly from the expected value.
1.95 > u >1.64	0.1 ÷ 0.05	The reported result probably does not differ significantly from the expected value.
2.58 > u >1.95	0.05 ÷ 0.01	It is not clear whether the reported result differs significantly from the expected value.
3.29 > u > 2.58	0.01 ÷ 0.001	The reported result is probably significantly different from the expected value.
U > 3.29	< 0.001	The reported result is significantly different from the expected value.

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. The calculated u-test value is compared with the critical values listed in the u-statistic tables to determine if the reported result differs significantly from the expected value at a given level of probability.

The advantage of u_{test} that it takes into consideration the propagation of measurement uncertainties. 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 the ALMERA proficiency test it was set the limiting value for the u_{test} parameter to 2.58 to determine if a result passes the test ($u \leq 2.58$). The limitation of using u_{test} as acceptance/rejection parameter is its insensitivity for cases where reported results are associated with unrealistically large uncertainties.

Performance evaluation criteria

The proficiency test results are evaluated against the following acceptance criteria for trueness and precision and assigned the status “Acceptable” or “Not Acceptable” accordingly. Trueness criteria is assigned “Acceptable” status if:

$$A_1 \le A_2$$

where:

$$A_1 = \left| Value_{IAEA} - Value_{Lab} \right|$$

$$A_2 = 2.58 \times \sqrt{unc^2{}_{IAEA} - unc^2{}_{Lab}}$$

Precision criteria is assigned “Acceptable” status if:

$$RSD_{Max}(\%) \ge \sqrt{\left(\frac{unc_{IAEA}}{Value_{IAEA}}\right)^2 + \left(\frac{unc_{Lab}}{Value_{Lab}}\right)^2} \times 100\%$$

where RSD $_{\text{Max}}$(%) is the estimated maximum acceptable reproducibility standard deviation expressed in percentage of the measurement value and set for each analyte as a function of its concentration level and ease of determination based on fit for purpose principle.

A result must obtain “Acceptable” status in both criteria to be assigned the final status of “Acceptable”. If the result obtained a “Not Acceptable” status for trueness or precision, then the relative bias is compared to a predetermined limit (based on fit for purpose principle), and if the 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 the result obtained “Not Acceptable” status for both trueness and precision the final score will be assigned as “Not Acceptable”.

ALMERA Soil Sampling Intercomparison Exercise (IAEA/SIE/01)

The evaluation of the pattern of environmental contamination is vitally dependent on reliable data, which are derived from complex analytical processes. Each step of the process, i.e. sampling, sample transportation, treatment, analysis and evaluation and interpretation of the results can affect to some degree the accuracy of the final analytical data.

Advances in analytical techniques and improved laboratory practice have reduced many sources of uncertainties which can originate during the laboratory analytical procedures, but the assessment of uncertainties associated with sampling of environmental components has not been fully considered in the past, since collaborative field studies require considerable organisational efforts. Sampling and sample preparation/processing are known to carry large, but typically unknown, uncertainty contributions to the final analytical data and there is a lack of qualitative and quantitative data on the comparability of results achieved by the different sampling methods. ISO/IEC 17025 [9] reports that sampling is a factor to be considered as a contributor to the total uncertainty of measurement [10].

To this end, the International Atomic Energy Agency (IAEA) organized for selected laboratories of the IAEA ALMERA network a Soil Sampling Intercomparison Exercise (IAEA/SIE/01). The objective was to compare the soil sampling protocols used by the different participating laboratories, in the case they are asked to determine the mean value of several radionuclides in an agricultural area of about 10000 square meters. The radionuclides to be considered in planning the sampling exercise were those that require radiochemical separation (^{90}Sr, Pu, ^{241}Am, ^{238}U) and a Test portion (*quantity of* *material, of proper size for measurement of the concentration or other property of interest, removed from the test sample* [10]) ranging from 10 to 50 g, depending on the activity concentration of the radionuclide.

The intercomparison exercise took take place from 14 to 18 November 2005 in an agricultural area qualified as a “reference site” (*area, one or more of whose element concentrations are well characterised in terms of spatial and temporal variability* [11]), in the frame of the SOILSAMP international project, funded and coordinated by the Italian Environmental Protection Agency (APAT, Italy) and aimed at assessing the uncertainty associated with soil sampling in agricultural, semi-natural, urban and contaminated environments [10], [11]. The “reference site” is located in the north eastern part of Italy (Pozzuolo del Friuli, Udine), in the research centre belonging to the Ente Regionale per lo Sviluppo Agricolo del Friuli Venezia Giulia (ERSA). The “reference site” is characterised in terms of the spatial variability of trace elements, and it is suitable for performing intercomparison exercises. The trace elements present at the reference site are of a combination of natural and anthropogenic origins.

Soil is the final receptor of trace elements, including radionuclides and organic pollutants dispersed in the environment [12]. In the long term after deposition, the behaviour of long-lived radionuclides in soil can be expected to be similar to that of some stable trace elements and the distribution of these trace elements in soil can simulate the distribution of radionuclides. Trace elements in soil, including radionuclides, are mostly associated with finer particle-size fractions [12, 13, 14, 15, 16, 17]. For this reason, the “reference site” characterised in term of trace elements can be used to compare the soil sampling strategies developed for radionuclide investigations by the ALMERA laboratories. In addition, soil sampling procedures for radionuclides derive from techniques used in agriculture and engineering [18].

The "reference site" used for the soil sampling intercomparison exercise

The reference site (10,000 m^2) is flat and regular-shaped with three sub-areas of different gravel content. On average, the fraction above 2 mm represents only 13 % of the sampled soil. Crop production did not take place on the site over the last six years. The area is easily accessible by operators, and any accidental or unauthorised use has been prevented (i.e. spreading of unknown substances, transit of vehicles).

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%). The present and past land use of this site is well known. 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 $cmol_{(+)}/kg$) [19].

In order to be used as a reference sampling site, the area, subdivided into 100 sub-areas (10x10 m each) was characterised for long- and short-range spatial variation of trace element concentrations in soil (Figure 1). To assess the long-range spatial variation, samples were taken in each sub-area at a 2 m distances from each other, resulting in 25 soil samples per sub-area. An Edelman auger (20 cm length, 7 cm diameter), after removing any surface vegetation, was used. The sampling was performed up to 20 cm within the ploughed layer. These samples were pooled and processed to give one composite sample for each sub-area, resulting in 100 composite samples. The sampling device was cleaned after sampling of each sub-area. To verify that the short-range spatial variability of trace element is comparable among different sub-areas, 2 sub-areas were sampled again and the resulting 25 samples per sub-area were analysed separately. Figure 1 shows the sampling points selected for the reference sampling.

The sample preparation and analytical activity were performed by a single laboratory in order to rule out the variabilities due to different analytical laboratories.

The soil samples were used to characterise the “reference site” in terms of trace elements (As, Ba, Br, Ca, Ce, Co, Cr, Cs, Eu, Fe, Ga, Hf, K, La, Na, Nd, Rb, Sb, Sc, Sm, Sr, Ta, Tb, Th, U, Yb, Zn, Zr). The reference values for each trace element were determined by instrumental neutron activation analysis (INAA). This technique achieves high precision levels and requires little or no sample processing prior to analysis. This analytical technique also eliminates uncertainty associated with sample processing [10]. In addition the main soil pedo-chemical properties were determined.

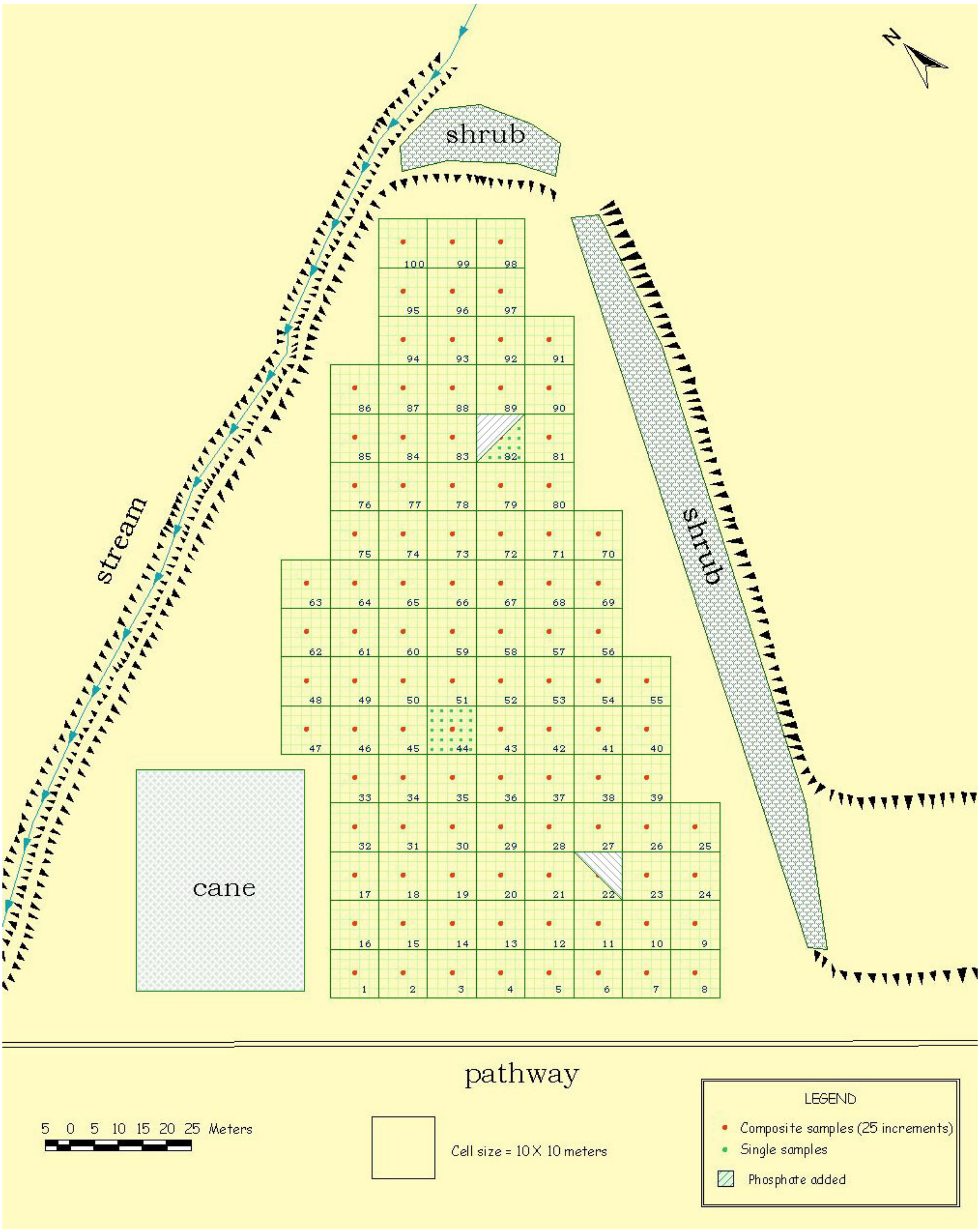

Figure 1 – The “reference site”

Participants to the soil sampling intercomparison exercise

Due to the limited extent of the reference sampling area and considering that collaborative field studies require considerable organisational efforts, for the current year exercise only 10 ALMERA Institutions were selected to participate in the sampling exercise. Experts from IAEA, APAT and ERSA provided the assistance during the sampling exercise.

Chang Byung-Uck	:	Korea Institute of Nuclear Safety	
Aguirre Jaime	:	Comision Nacional de Seguridad Nuclear y Salvaguardias (Mexico)	
Al-Masri Mohammad S.	:	Atomic Energy Commission of Syria	
Derkach Grygoriy	:	Ukrainian Hydrometeorological Institute (Ukraine)	
Fathivand Khalili Mir Ali Asghar	:	Iranian Nuclear Regulatory Authority.	
Grubel Stefan	:	NPP Mochovce (Slovak Republic)	
Kanivets Volodymyr	:	Ukrainian Hydrometeorological Institute (Ukraine)	
Korun Matjaz	:	Josef Stefan Institute (Slovenia)	
Kozar-Logar Jasmina	:	Josef Stefan Institute (Slovenia)	
Ladygiene Rima	:	Radiation Protection Centre of Lithuania	
Lee Dong-Myung	:	Korea Institute of Nuclear Safety	
Tarjan Sandor	:	National Food Investigation Centre (Hungary)	
Tirollo Taddei Maria Helena	:	Brazilian Nuclear Energy Agency	
Varga Beata	:	National Food Investigation Centre (Hungary)	
Vasarab Julius	:	NPP Mochovce (Slovak Republic)	
Benesch Thomas	:	IAEA, Seibersdorf (Austria)	(assistance)
Sansone Umberto	:	IAEA, Seibersdorf (Austria)	(assistance)
Shakhashiro Abdulghani	:	IAEA, Seibersdorf (Austria)	(assistance)
Trinkl Alexander	:	IAEA, Seibersdorf (Austria)	(assistance)
Belli Maria	:	APAT (Italy)	(assistance)
De Zorzi Paolo	:	APAT (Italy)	(assistance)
Rosamilia Silvia	:	APAT (Italy)	(assistance)
Menegon Sandro	:	ERSA, Udine (Italy)	(assistance)
Taccheo Barbina Maria	:	ERSA, Udine (Italy)	(assistance)

A meeting between groups, the IAEA team and the APAT experts took place in ICTP before the sampling exercise. During the meeting a visit to the sampling area took place.

During the sampling exercise, each participant laboratory was asked to collect soil samples on the “reference site”, in number enough to assess the mean value of the above considered radionuclides. Each participant laboratory was asked to apply its own soil sampling protocol (sampling strategy, sampling pattern, sampling design, sampling device, sampling depth, sampling techniques, etc.) and to collect not more than 15 soil samples.

For submission of the sampling protocols by the participants, a server has been prepared and established at the ICTP network, providing a web-based interface to the project database. Also all necessary information for the participants were offered via this system. The system was found to work in a satisfactory and robust manner, also as an example for possible arrangements for future exercises. In this frame:

- each participant laboratory provided a description of the sampling devices used and the sampling protocol applied during the intercomparison exercise (description of devices, number of samples, size of each sample, depth of sampling, etc.);
- each participant laboratory provided a description of its own methodology to prepare the Test Portion from each of the samples collected during the sampling exercise;
- each participant laboratory provided its own methodology to estimate the mean value of several analytes in a sampling exercise.

On Friday 18 November 2005 a meeting with the sampling groups, the IAEA team and the APAT experts took place at the ICTP for final comments and considerations on the Soil Sampling Intercomparison Exercise (IAEA/SIE/01). During the sampling exercise, the sampling of each participant team was documented photographically and a short video was recorded for each procedure. The videos were processed on the spot and presented to the participants for discussion during the meeting. Each participant presented and commented on his/her procedure briefly while showing the video.

To rule out variabilities eventually caused by different soil sample preparation techniques and by different analytical laboratories, a single laboratory (IAEA’s Chemistry Unit), following a predefined protocol developed by APAT, will perform all the sample preparation and a single laboratory (The Atomic Energy Commission of Syria), following an agreed analytical protocol, will carry out the determination of trace elements using a technique that can achieve high precision levels and requires little or no sample processing prior to analysis (INAA).

The participants’ data will be evaluated according to IS0 13528:2005 (E) [20].

To cover partially the expenses the participating laboratory incurred to participate in the soil sampling intercomparison exercise, the ICTP provided a lump sum to the following teams: Hungary, Iran, Lithuania, The Republic of Korea, Mexico, Slovakia, Slovenia and Syria,. The participation of the Ukrainian team was covered under the IAEA TC UKR 9023 project, while the cost connected with the Brazilian participant was covered by its own Institution.

It is planned to discuss the final results of the Sampling Intercomparison Exercise (IAEA/SIE/01) during the 2006 ALMERA network members meeting.

Acknowledgements

The assistance of the ICTP personnel, Ms. D. Giombi, Ms. D. Festa, Ms. D. Photiou and Mr. Grassberger is very much appreciated.

The Italian Environmental Protection Agency (APAT) provided the assistance of experts during the soil sampling exercise (Maria Belli, Paolo de Zorzi and Silvia Rosamilia), at no cost to the IAEA.

The Ente Regionale per lo Sviluppo Agricolo del Friuli Venezia Giulia (ERSA), located in Pozzuolo del Friuli, Udine (Italy) provided the assistance (Sandro Menegon and Maria Taccheo Barbina) and the facilities to conduct the sampling exercise at the "reference site", at no cost to the IAEA.

References

1. Report of the second Research Coordination Meeting on the Coordinated Research Programme "Rapid instrumental and separation methods for monitoring radionuclides in food and environmental samples", IAEA/AL/056, p. 38, August 2001.
2. Report of the first Research Coordination Meeting on the Coordinated Research Programme "Development and selection of analytical techniques for measuring accidentally released radionuclides in environment", IAEA/AL/082, p. 30, December 1994.
3. A.Shakhashiro, Z. Radecki, A. Trinkl, U. Sansone and T. Benesch, "Final Report on the Proficiency Test of the Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA) Network", IAEA/AL/152, p.242, August 2005.
4. IAEA Technical Report, "The Radiological Situation at the Atolls of Mururoa and Fangataufa", (six volumes), August 1998.
5. M. Astner, M. Burger, K. Burns, T Cabianca, M. Campbell, P.R. Danesi, J. Moreno, Z. Radecki, "Environmental Analyses for the Assessment of the Consequences of the use of DU Ammunition during the Gulf War in Kuwait, Radiological Conditions in Areas of Kuwait with Residues of Depleted Uranium", IAEA Radiological Assessment Reports Series, 2003.
6. Report of the First ALMERA Workshop, IAEA/AL/113, p.12, November 1997.
7. ISO GUIDE 35, "Certification of reference materials – general and statistical principles", International Organization for Standardization (ISO), Geneva, Switzerland, 1989.
8. ISO 5725-1994 (E), "Accuracy (trueness and precision) of measurement methods and results", International Organization for Standardization (ISO), Geneva, Switzerland, 1994.
9. ISO/IEC 17025:1999, General requirements for the competence of testing and calibration laboratories. International Organization for Standardization (ISO), Geneva, Switzerland, 1999.

10. de Zorzi, P., Belli, M., Barbizzi, S., Menegon, S. and Deluisa A., A practical approach to assessment of sampling uncertainty, Accreditation and Quality Assurance (2002), 7:182-188.
11. de Zorzi, P., Barbizzi, S., Belli, M., Ciceri, G., Fajgelj, A., Moore, D., Sansone, U. and Van der Perk, M., Terminology in soil sampling (IUPAC recommendations 2005), International Union of Pure and Applied Chemistry, Analytical Chemistry Division, 77 (5) 827-841.
12. Coughtrey, P.J. and Thorne, M.C., Radionuclide distribution and transport in terrestrial and aquatic ecosystems. A critical review of data. (1983), A.A, Balkema, Rotterdam.
13. Qian, J., Shan, X., Wang, Z. and Tu, Q., Distribution and plant availability of heavy metals in different particle-size fractions of soil, The Science of the Total Environment 187 (1996), 131-141
14. Haque, M.A., Subramanian, Cu, Pb and Zn, pollution of soil environment. CRC Crit. Rev. Environ. Contr., 12 (1982) 13-90
15. Foerstner, U. Trace metal analysis of polluted sediments, Part 1, Assessment of sources and intensities. Environ. Technol. Lett., 1. (1980) 494-505
16. Spezzano P., Distribution of pre- and post-Chernobyl radiocaesium with particle size fractions of soil Journal of Environmental Radioactivity 83 (2005) 117-127
17. Livens, F. R., Baxter, M. S., Particle size and radionuclide levels in some West Cumbrian soils. The Science of the Total Environment 70 (1988), 1-17.
18. HASL-300, 28th Edition, Section 2.4, Vol.1.
19. Barbizzi et al., Characterization of a reference site for quantifying uncertainties related to soil sampling. Environmental Pollution 127 (2004), 131-135
20. ISO 13528:2005 (E) Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization (ISO), Geneva, Switzerland, 2005.

The "reference site"

ALMERA network laboratories

Australia	Australian Radiation Protection & Nuclear Safety Agency (ARPANSA) Lower Plenty Road 619 Yallambie, Victoria 3085 Australia
	ANSTO – Environment New Illawarra Rd 2234, Lucas Heights Menai N.S.W. Australia
Austria	Atominstitut der Oesterreichischen Universitaeten Schuettelstrasse 115 A-1020, Vienna Austria
	Universitaet Wien Waehringerstrasse 42 A-1090, Vienna Austria
Belarus	Belarussian State Institute of Metrology Starovilenski Trakt 93 220053, Minsk Belarus
Belgium	Studiecentrum Voor Kernenergie Centre d'etude de l'Energie Nucleaire Boeretang 200 B-2400, Mol Belgium
Brazil	Brazilian National Commission for Nuclear Energy (CNEN) Caixa Postal 913 Br-37701-970, Pocos De Caldas Brazil
Bulgaria	Executive Environment Agency Ministry Of Environment & Water 136 Tzar Boris Iii, Blvd. P.O. Box 251 Bg-1618, Sofia Bulgaria

	Kozloduy Nuclear Power Plant Kozloduy, 3321 Bulgaria
Chile	Comision Chilena de Energia Nuclear Amunategui 95 Casilla 188-D Santiago De Chile Chile
China	China Institute for Radiation Protection 270 Xuefu Street P.O. Box 120 Taiyuan, 030 006 Shanxi Province China
	Chinese Academy of Agricultural Sciences P.O.Box 5109 100 094, Beijing China
	China Institute of Atomic Energy P.O.Box 275-24 Beijing, 102413 China
Costa Rica	Universidad de Costa Rica Ciudad Universitaria R. Facio San Jose Costa Rica
Cyprus	State General Laboratory Ministry of Health 44 Kimonos Street, Acropolis 1451, Nicosia Cyprus
Cuba	Centro de Proteccion e Higiene De Las Radiaciones Calle 20 Entre 41 Y 47 Apartado Postal 6791 Havana, 11300 Cuba

	Centro de Isotopos Carretera la Rada Km 3 Guanabacoa, Apto 22 Havana Cuba
Denmark	Risoe National Laboratory P.O. Box 49 Dk-4000, Roskilde Denmark
Egypt	National Center for Nuclear Safety & Radiation Control Atomic Energy Authority 3 Ahmed El-Zomor Street P.O. Box 7551, Nasr City Cairo, 11762 Egypt
Estonia	Estonian Radiation Protection Centre Kopli 76 Tallinn, Ee-10416 Estonia
Ethiopia	National Radiation Protection Authority P.O.Box 20486 Code 1000, Addis Ababa Ethiopia
Finland	Radiation & Nuclear Safety Authority (STUK) P.O. Box 14 Fin-00881, Helsinki Finland
France	IPSN/DPRE/SERNAT F-91191, Gif-Sur-Yvette France
Germany	Research Center Karlsruhe P.O. Box 3640 D-76021, Karlsruhe Germany
	Physikalisch-Technische Bundesanstalt P.O. Box 3345 D-38023, Braunschweig Germany

Greece	National Technical University of Athens 9 Iroon Polytechniou Street Polytechnioupoli Gr-157 73, Athens Greece
	Aristotelian University of Thessaloniki Gr-54124, Thessaloniki Greece
Hungary	Hungarian Academy of Sciences KFKI Atomic Energy Research Institute P.O. Box 49 H-1525, Budapest Hungary
	National Food Investigation Institute P. O. Box 1740 H-1465, Budapest, 94 Hungary
Iceland	Gelslavarnir Rikisins National Institute of Radiation Protection Laugavegur 118d Is-150, Reykjavik Iceland
India	Madras Atomic Power Station Kalpakkam, 603 102 Tamilnadu State India
	Tarapur Atomic Power Station Via Boisar Tarapur, 401 504 Maharashtra India
	Bhabha Atomic Research Centre Mumbai, 400 085 India
Indonesia	National Nuclear Energy Agency P.O. Box 7043 JKSKL Jakarta, 12070 Indonesia

Iran	Atomic Energy Organization of Iran (AEOI) North Karegar Street P.O. Box 14155-4494 14374, Tehran Iran
Iraq	Iraqi Atomic Energy Commission P.O. Box 765, Tuwaitha Baghdad Iraq
Israel	Nuclear Research Center Negev (NCRN) Negev Israel
Italy	Centrale Elettronucleare di Caorso So.G.I.N Laboratories 29012, Caorso, Piacenza Italy
	Centrale Elettronucleare di Trino So.G.I.N Laboratories Strade Statale 31 Bis 13039, Vercelli Italy
	ENEA Istituto di Radioprotezione Laboratorio Casaccia Via Anguillarese 301 I-00060, Santa Maria Di Galeria Roma Italy
	ENEA Istituto di Radioprotezione Laboratorio Trisaia Strada Statale Ionica 106, Km 419,5 I-75026, Rotondella Matera Italy
	ENEA Research Centre Strada per Crescentino I-13040, Saluggia (Vercelli) Italy

	APAT (Environmental Protection Agency) Via di Castel Romano 100 I-00128, Roma Italy
Jamaica	University of the West Indies Mona Campus Kingston, 7 Jamaica
Jordan	Jordan Atomic Energy Commission P.O. Box 70 Amman, 11934 Jordan
Kazakhstan	National Nuclear Center Ibragimov Str. 1 480082, Almaty Kazakhstan
Korea	Korea Institute of Nuclear Safety P.O. Box 114, Yusung Taejon, 305-353 The Republic of Korea
	Korea Atomic Energy Research Institute 150 Duckjin-Dong, Yusung-Ku Taejon, 305-353 The Republic of Korea
Kuwait	Kuwait Institute for Scientific Research P.O. Box 24885 Safat, 13109 Kuwait
Latvia	Radiation Safety Centre Maskavas 165 Riga, Lv-1019 Latvia
Lebanon	Lebanese Atomic Energy Commission P.O. Box 11-8281 Beirut Lebanon

Lithuania	Environment Protection Agency Ministry of Environment A. Juozapaviciaus 9 Lt-9311, Vilnius Lithuania
	National Veterinary Laboratory J. Kairiukscio 10 Lt-08409, Vilnius Lithuania
	Radiation Protection Centre Kalvariju 153 Lt-08221, Vilnius Lithuania
Luxembourg	Villa Louvigny Allee Marconi L-2120, Luxembourg Luxembourg
Malaysia	Malaysian Institute for Nuclear Technology Research (MINT) Bangi, 43000, Kajang, Selangor D.E. Malaysia
Mexico	Instituto Nacional de Investigaciones Nucleares (ININ) Km 36,5 Carretera Mexico Toluca Edo.De Mexico, C.P. 52045 Salazar Mexico
	Comision Federal de Electricidad Km 182 de la Carretera Federal 180, Tramo Poza Rica Puerto De Veracruz, C.P. 91490 Farallon, Veracruz Mexico
	Comision Nacional de Seguridad Nuclear y Salvaguardias (CNSNS) Dr. Jose Ma. Barragan # 779 Col. V. Narvarte, Del. B. Juarez Mex-03020, Mexico City Mexico

Myanmar	Myanmar Atomic Energy Committee No. 6 Kaba Aye Pagoda Road Yangon Myanmar
The Netherlands	National Institute of Public Health & Environment Antonie Van Leeuwenhoeklaan 9 NL-3720 BA, Bilthoven The Netherlands
	NRG Westerduinweg 3 P.O. Box 25 1755 ZG Petten The Netherlands
New Zealand	National Radiation Laboratory 108 Victoria St. P.O. Box 25099 Christchurch New Zealand
Norway	Norwegian Radiation Protection Authority P.O. Box 55 N-1345, Oesteraas Norway
	Institute for Energy Technology Instituttveien 18 P.O. Box 40 N-2027, Kjeller Norway
Pakistan	Pakistan Institute of Nuclear Science & Technology (PINSTECH) Mnsr Lab. Naa Acg P.O.Box 1482- Nilore 92/051, Islamabad Pakistan
Panama	Centro de Investigacion con Tecnicas Nucleares Fac. De Cienc. Natur. y Exactas 25 Universidad de Panama Ciudad Univ. O. Mendez Pereira Ciudad De Panama Panama

Philippines	University of the Philippines Commonwealth Avenue P.O. Box 213 Up, Diliman Quezon City, 3004 Philippines
Poland	Central Laboratory for Radiological Protection Ul. Konwaliowa 7 Pl-03-194, Warsaw Poland
	Institute of Oceanology Polish Academy of Sciences Powstancow Warszawy 55 P.O. Box 197 Pl-81-712, Sopot Poland
Portugal	Instituto Tecnologico e Nuclear Estrada Nacional 10 P-2686-953, Sacavem Portugal
Romania	Institute For Nuclear Research P.O. Box 78, Colibasi R-0300, Pitesti Romania
	Nuclear Power Plant Cernavoda Stada Medgidiei, 1 C.P. 42 8625, Cernavoda Jud. Constanta Romania
	Institute ff Physics & Nuclear Engineering P.O. Box Mg-6 R-76900, Bucharest Sector 5 Romania
Russian Federation	SIA "Radon' 7th Rostovsky Ln., 2/14 119121, Moscow Russian Federation

	Russian Institute of Agricultural Radiology & Agroecology 249020, Obninsk Kaluzhskaya Oblast Russian Federation
Saudi Arabia	Institute of Atomic Energy Research King Abdulaziz City for Science & Technology P.O. Box 6086 Riyadh, 11442 Saudi Arabia
Serbia and Montenegro	Center for Ecotoxicological Researches of Montenegro Put Radomira Ivanovica 2 Yu-81000, Podgorica Serbia and Montenegro
	Institute of Occupational & Radiological Health Deligradska 29 Yu-11000, Belgrade Serbia and Montenegro
Slovak Republic	Nuclear Power Plant Bohunice Sk-919 31, Jaslovske Bohunice Slovak Republic
	Nuclear Power Plant Mochovce Komenskeho 3 Sk-935 39, Levice Slovak Republic
	Institute of Preventive & Clinical Medicine Limbova 14 Sk-833 01, Bratislava Slovak Republic
Slovenia	Institute Jozef Stefan Jamova 39 P.P.3000 Si-1001, Ljubljana Slovenia
South Africa	South African Nuclear Energy Corporation P.O. Box 582 Pretoria, 0001 South Africa

Spain	C I E M A T Ed-7, Avda Complutense 22 E-28040, Madrid Spain
Sweden	Swedish Radiation Protection Institute Solna Strandväg 96 S-171 16, Stockholm Sweden
Switzerland	Federal Office of Public Health 3, Chemin Du Musee Ch-1700, Fribourg Switzerland
Syria	Atomic Energy Commission of Syria P.O. Box 6091 Damascus Syrian Arab Republic
Thailand	Office of Atomic Energy for Peace 16 Vibhavadi Rangsit Chatuchak Bangkok, 10900 Thailand
Tunisia	Centre National des Sciences et Technologies Nucleaires Pole Technologique Sidi Thabet, 2020 Tunisia
Ukraine	Ukrainian Hydrometeorological Institute Prospect Nauki 37 Kiev-28, 03028 Ukraine
United Arab Emirates	Central Food Control Laboratory Sharjah Municipality Sultan Ben Saqr El Qassimi P.O.Box 22 Sharjah United Arab Emirates
United Kingdom	Imperial College at Silwood Park Buckhurst Road Ascot, Sl5 7te Berkshire United Kingdom

Veterinary Laboratories Agency
Woodham Lane, New Haw
Addlestone, Surrey Kt15 3nb
United Kingdom

National Physical Laboratory
Fg-A1, Queens Road
Teddington, Tw11 0lw
Middlesex
United Kingdom

Centre for Environment, Fisheries & Aquaculture Science
Pakefield Road
Lowestoft, Nr33 0HT
Suffolk
United Kingdom

LGC Ltd
Queens Road
Teddington, TW11 0LY
Middlesex
United Kingdom

USA

Los Alamos National Laboratory
P.O. Box 1663
Los Alamos, NM 87545
United States of America

U.S. Department of Energy (DOE)
850 Energy Drive
Idaho Falls, ID 83402
United States of America

Purdue University
1396 Physics Building
West Lafayette, IN 47907-1396
United States of America

University of California
Lawrence Livermore National Laboratory
7000 East Avenue
P.O. Box 5001
Livermore, CA 94551
United States of America

	Savannah River Technology Center Westinghouse Savannah River Co. Building 773-A P.O. Box 616 Aiken, SC 29802 United States of America
Venezuela	Instituto Venezolano de Investigaciones Cientificas (IVIC) Apartado Postal 21827 Caracas, 1020-A Venezuela
Zambia	National Council For Scientific Research P.O. Box 310158 Lusaka, 15302 Zambia
IAEA	Department of Nuclear Sciences and Applications Physics, Chemistry and Instrumentation Laboratory Chemistry Unit Agency's Laboratories Seibersdorf A-2444 Seibersdorf, Austria
	Department of Nuclear Sciences and Applications Marine Environment Laboratory 4, Quai Antoine Premier MC980000 Monaco Principality of Monaco