Review of the activities of the Reference Material Group of the IAEA’s Seibersdorf Laboratories (2006-2007)

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Introduction

The Chemistry Unit of the Physics, Chemistry and Instrumentation Laboratory in the IAEA's Seibersdorf Laboratory in Austria, has the programmatic responsibility to provide assistance to Member State laboratories in maintaining and improving the reliability of analytical measurement results, both in trace element and radionuclide determinations. This is accomplished through the provision of reference materials of terrestrial origin, validated analytical procedures, training in the implementation of internal quality control, and through the evaluation of measurement performance by organization of worldwide and regional interlaboratory comparison exercises.

The use of reference materials and interlaboratory comparison exercises are among the most important tools for the production of reliable measurement results and for the achievement of a required quality level. Through the evaluation reports provided to the participating laboratories in the interlaboratory comparison exercises, the IAEA provides direct consultation and guidance on appropriate measurement techniques to be applied, and identifies gaps and problem areas where further development is needed. This helps participating laboratories to improve both their measurement techniques and performance. With the advent of “mutual recognition” on both a European and 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”.

The activities of the Chemistry Unit are also addressed to support global radionuclide measurement systems, in issues of international concern related to an accidental or intentional release of radioactivity in the environment. To fulfill this obligation and ensure a reliable worldwide, rapid and consistent response, the Chemistry Unit coordinates an international network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA). The network, established by the IAEA in 1995 [1] [2] [3], makes available to Member States a world-wide network of analytical laboratories capable of providing reliable and timely analysis of environmental samples in the event of an accidental or intentional release of radioactivity. The network is a technical collaboration of existing institutions. It provides an operational framework to link expertise and resources, in particular when a boundary-transgressing contamination is expected or when an event is of international significance.
ALMERA currently (May 2007) consists of 106 laboratories representing 67 countries. The Agency's Seibersdorf Laboratory in Austria and its Marine Environment Laboratory in Monaco are members of the network.

**ALMERA network activities**

**ALMERA second coordination meeting**

On 15 of November 2005 the second ALMERA network coordination meeting took place in Trieste (Italy) and was hosted by the International Centre for Theoretical Physics (ICTP) [4].

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.

**Soil Sampling Intercomparison Exercise (IAEA/SIE/01).**

Within the frame of ALMERA activities a Soil Sampling Intercomparison Exercise (IAEA/SIE/01) was organized for selected laboratories of the IAEA ALMERA network [4]. The objective of the study was to compare the soil sampling protocols used by the different participating laboratories, in the case they were asked to determine the mean value of several radionuclides in an agricultural area of about 10000 square meters. The radionuclides of interest in this exercise were those that require radiochemical separation ($^{90}$Sr, $^{241}$Am, $^{238}$U) and a test portion ranging from 10 to 50 g, depending on the activity concentration of the radionuclide.

The intercomparison exercise took 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, 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. 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.

The third ALMERA network coordination meeting took place in Daejeon (The Republic of Korea) from 13th to 16th November 2006 and was hosted by the Korea Institute of Nuclear Safety (KINS) [5].

The meeting was attended by 36 participants from 17 different institutions and by a representative of the Embassy of the United States of America in the Republic of Korea. The main aim of the meeting was to discuss the new structure and future development of the ALMERA network.

To facilitate interactions between the ALMERA laboratories, for the period 2007-2009 the network is subdivided into the following three regional groups:

- Africa-Europe-Middle East;
- North and Latin America;
- Asia-Pacific

A primary requirement of the ALMERA members is participation in the IAEA interlaboratory comparison exercises which are specifically organized for ALMERA on a regular basis. These exercises are designed to monitor and demonstrate the performance and analytical capabilities of the network members, and to identify gaps and problem areas where further development is needed. Continued membership has benefits in training and educational opportunities, enhanced mutual trust in results and methodology and objective evidence for accreditation purposes.

At least one interlaboratory comparison exercise should be organized per year by the IAEA for the ALMERA network. In case of any special event it may be extended to additional exercises. The following matrices are identified as of primarily interest due to their importance both for routine monitoring and issues where the measurement results should be ready in a short time: soil, water, vegetation and air filters. The performance evaluation results of the interlaboratory comparison exercises performed in the frame of the ALMERA network are not anonymous for those laboratories nominating to participate as ALMERA members.

Considering that the ALMERA network members may need to support radionuclide measurement in issues where the measurement results should be ready in a short time, the laboratories participating in the ALMERA interlaboratory comparison exercises should report the results to the organizer on a short reporting time (1-3 days). In addition to the above reporting times, the laboratories participating in the ALMERA interlaboratory comparison exercises will be also asked to report results to the organizer on a longer-term reporting timeframe (as usually indicated for the IAEA world-wide open proficiency test).

Reference materials

Reference materials selling and distribution

In 2005-2006 the IAEA Reference Materials Group sold around 1750 units of reference materials to laboratories from 95 countries around the world. In addition 2000 bottles of reference materials were prepared to be used in the IAEA proficiency testing.
55 % of all shipped matrix reference materials have been characterized for radionuclides, 38 % for trace elements, and 7 % for organic contaminants.

In 2007 a new central storage facility was put into operation in Seibersdorf for storing all matrix reference materials produced on the Agency’s Laboratories of Monaco and Seibersdorf.

A new IAEA Reference Material Catalogue for 2007 was issued on a CD-Rom and is distributed to customers.

Preparation and certification of matrix reference materials of terrestrial origin

In 2005 the certification report of the IAEA-392 reference material “Trace, minor and major elements in naturally grown algae” was issued [6]. More details can be found at: http://www.iaea.org/programmes/aqcs/database/ref_sheets/iaea_392.shtml

In 2006 the certified reference material IAEA-372 “Radionuclides in grass” was produced and certified.

In addition, feasibility studies of five candidate reference materials namely: three soils: IAEA-360. IAEA-378, IAEA-377, one lake sediment IAEA-344 and one vegetation IAEA-330 (spinach) were performed. Three of these materials are already bottled and ready for homogeneity testing and certification. These materials will be characterized for alpha, beta and gamma emitting radionuclides.

Expert laboratories (collaborators) meeting on production and characterization of the IAEA reference materials of terrestrial origin

The meeting was held at the Agency’s Seibersdorf Laboratory from 19-23 June 2006 to discuss with six expert laboratories participating in the characterization of the IAEA reference materials of terrestrial origin the quality assurance and metrological traceability aspects related to the certification of reference materials. During the meeting the approaches and methodologies for
reference materials characterisation were discussed. Special emphasis was put on the Agency’s quality requirements. Options for mutual cooperation on the selection and provision of raw materials for future candidate reference materials were discussed and agreed [7].

The meeting participants agreed that the Metrological traceability of the assigned property values of the IAEA reference materials should, whenever possible, be established to the SI Units and the metrological traceability chain described as clear as possible. Statement on metrological traceability should be an integral part of the certification report and certificates.

It was agreed during the meeting that it is becoming more and more important to apply a formal quality system according to ISO-Guides 34:2000 and ISO/IEC 17025:2005 in the production and certification of reference materials to meet the stakeholders requirements.

Future plans for the production of matrix reference materials of terrestrial origin

In 2007-2008 four matrix certified reference materials will be produced and certified for their content of radionuclides, namely:

- IAEA-330 Radionuclides in spinach;
- IAEA-434 TENORM in phosphogypsum;
- IAEA-444 Radionuclides in spiked soil;
- IAEA-445 Radionuclides in spiked water;
- TENORM in contaminated environmental samples from oil industry.

Interlaboratory comparisons exercises

In 2006 two CCRI supplementary comparisons were piloted:

- CCRI-BIPM supplementary comparison CCRI(II)-S4 on radionuclides in water, soil and grass. 5 National Metrological Institutes and 2 expert laboratories participated in this comparison [8]. Draft A of the final report was distributed to the participants for comments;
- CCRI-BIPM supplementary comparison CCRI(II)-S5 on the determination of TENORM in phosphogypsum. The samples were prepared and tested for homogeneity and distributed to 12 National Metrological Institutes.

In 2006 several proficiency tests were organized, where more than 500 laboratories from 90 Member states participated in these proficiency tests. The objectives of these proficiency tests were to (i) check the accuracy and precision of the analytical results produced by the participating laboratories from all over the world, (ii) test the international comparability of radiological measurements and (iv) encourage the participating laboratories in finding remedial actions where shortcoming in analytical performance are detected. The following proficiency tests were conducted:

- Determination of $^{210}$Po in water;
- Determination of Radionuclides and Trace elements in soil and compost [9];
- Determination of $^{210}$Pb and $^{137}$Cs in soil [10];
- Major, minor and trace elements in Chinese ceramics;
- Determination of Radionuclides in air filters in cooperation with US-DOE [11];
• ALMERA Network Proficiency Test for the determination of gamma emitting radionuclides in water, soil and grass [12];
• World wide open PT for the determination of gamma emitting radionuclides in water, soil and grass;
• Determination of Radionuclides in sea water [13].

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