

Review of the activities related to metrology at the IAEA's Seibersdorf Laboratories (2004-2005)

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Introduction

The International Atomic Energy Agency (IAEA) is the world's center of cooperation in the nuclear field. It was set up as the world's "Atoms for Peace" organization in 1957 within the United Nations family.



The Agency works with its Member States and multiple partners worldwide to promote the safe, secure and peaceful use of nuclear technologies.

The IAEA's mission is guided by the interests and needs of Member States, strategic plans and the vision embodied in the IAEA Statute. Three main pillars - or areas of work - underpin the IAEA's mission: Safety and Security; Science and Technology; and Safeguards and Verification.

The Agency's Laboratories are located at Seibersdorf (Lower Austria) about 35 km southeast of Vienna at the premises of the Austrian Research Center. One unit of the Agency's Laboratories, the Isotope Hydrology Unit, is located in Vienna, in the same building as the IAEA's Headquarters.



The Seibersdorf Laboratories were established in 1962 with the objective to contribute to the implementation of the Agency's programmes and projects within its department of Nuclear

Sciences and Applications, but also for Safeguards, Nuclear Safety and Security and Technical Co-operation. Therefore the main activities are the provision of scientific and analytical services, research and development and training of scientists from developing countries. Programmatic responsibilities lie in the fields of Assessment in Support of Sustainable Management of the Terrestrial Environment, and Supporting Quality in the Analysis of Terrestrial Environmental Samples.

The Chemistry Unit is a part of the Physics, Chemistry and Instrumentation (PCI) Laboratory. It supports Member State laboratories to enhance the quality of analytical measurement data both in trace element and radionuclide determination. This is accomplished through the provision of reference materials and validated procedures, training in the implementation of quality control, and through the evaluation of measurement performance by organisation of proficiency tests and intercomparison exercises.

Although the link between reference material production, proficiency tests, intercomparison exercises and environmental projects is not always easily visible, in principle all environmental studies, monitoring networks, environmental pollution databases, method developments related to environmental analysis, and remediation strategies rely on analytical data which are produced in analytical laboratories. Only accurate, reliable and representative analytical results should be the basis for decisions related to health care, environment and economics, and the tools provided by the reference material group are essential to achieve this.



The activities of the Chemistry Unit are also designed to support global radionuclide measurement systems in issues of international concern or emergency situations. To fulfil this obligation and ensure a reliable worldwide, rapid and consistent response, the Chemistry Unit coordinates the ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity). ALMERA is a network capable of providing a broader world-wide emergency assistance coverage to address situations needing a speedy response in order to mitigate the environmental consequences of a radiological emergency.



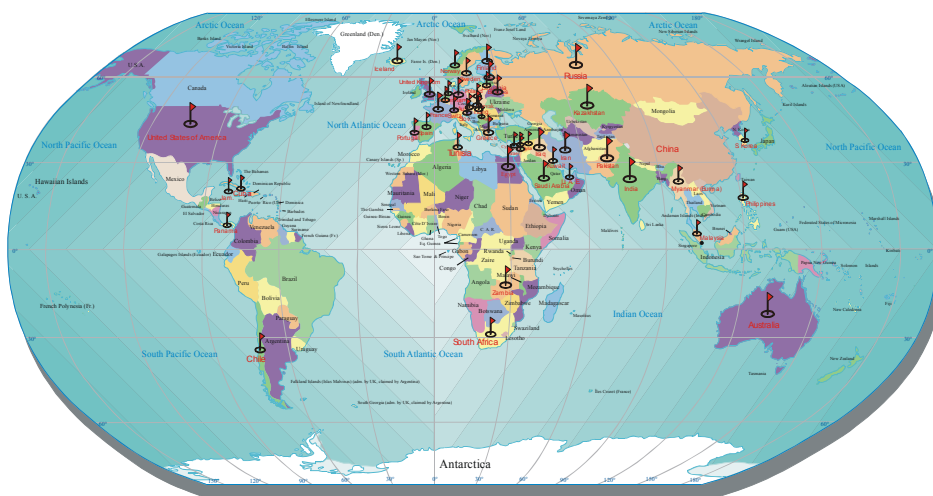
In order to remain as an active ALMERA member, a primary requirement is the successful participation in IAEA proficiency tests or intercomparison trials. These are specifically organized for ALMERA on a regular basis, to monitor and demonstrate the performance and analytical competence of network members. 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 and for the assessment of the environmental consequences of the use of Depleted Uranium (DU) as a result of the Gulf war.

Reference materials, interlaboratory comparisons and proficiency tests

The use of reference materials, interlaboratory comparisons and proficiency tests 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 proficiency tests or intercomparison trials, the IAEA provides direct consultation and guidance on appropriate measurement techniques to be applied, and identifies possible sources of bias in methods and procedures. This helps participating laboratories to improve both their measurement techniques and performance.



The IAEA is the major worldwide provider of matrix reference materials characterized for radionuclides. The IAEA database contains approximately 3500 institutes from almost all the IAEA Member States. Around 7000 individual scientists have contacted the agency for one or more reference materials. Every year approximately 1300 units of reference materials are distributed (around 800 units sold and 500 used for interlaboratory exercises). 60 % of all shipped matrix reference materials have been characterized for radionuclides, 28 % for trace elements, 7 % for methyl mercury and 5 % for organic contaminants.



The Agency holds currently a stock of approx. 25 000 units; 55 different products are currently available. The offer of reference materials is presented with a catalogue that is

shipped to 2500 to 3000 interested customers every second year; additionally the complete offer is presented on the IAEA web pages.

The new IAEA Reference Material Catalogue for 2004-2005 was issued in 2004 as hard copy and CD-Rom and distributed to about 3000 customers.

In 2003-2004, 501 orders for reference materials were received from 74 countries. The reference materials sold in 2003 and 2004 were valued at 61820 and 70370 US \$ respectively. Figure 1 presents a summary of the units sold, classified according to analyte group.

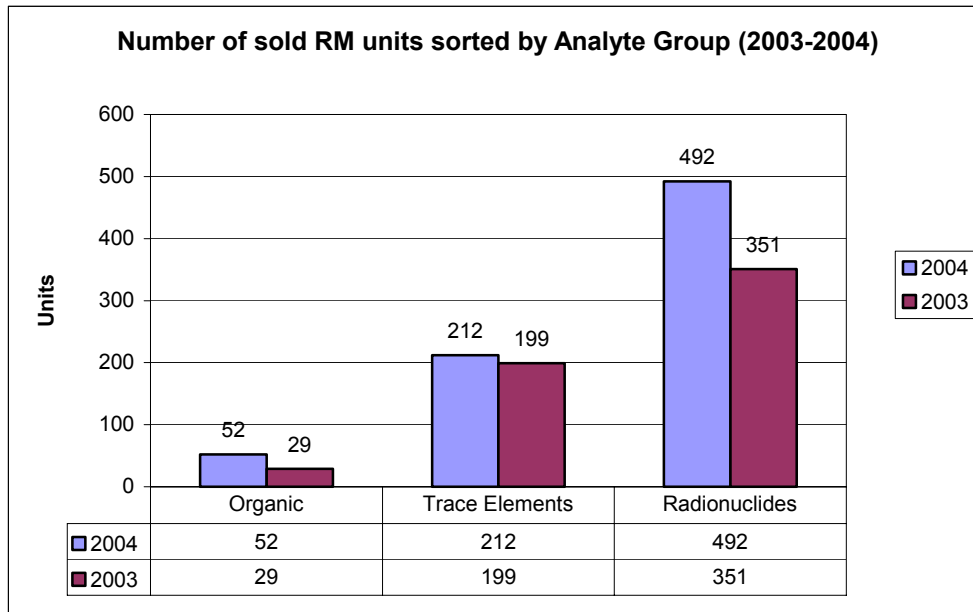


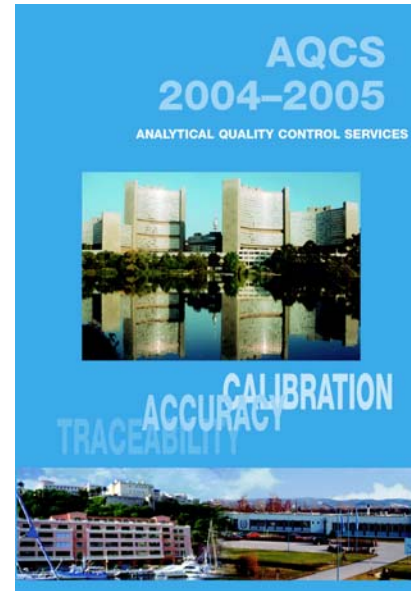
Figure 1. Reference materials units sold in 2003 and 2004, classified according to analyte group

Within the frame of the IAEA Technical Cooperation project INT/1/054 entitled “Preparation of reference materials for proficiency test rounds” the IAEA Chemistry Unit coordinated and assisted in implementation of the following exercises:

- preparation of mushroom reference material. The material was homogenized at the Institute for Reference Materials and Measurements (IRMM) in Geel, Belgium during the first workshop of the project (15-19 September 2003). Twelve participants contributed to the elemental characterization. Particle size and water content were analysed. The homogeneity test gave good results. Consensus values were determined after outlier rejection using the HISTO statistical software for the interlaboratory studies. A threshold value for acceptance for the material to be fit for the purpose was established.
- preparation of sediment reference material. The material was prepared within a previous European project, TRAP-LRM, led by the ISPRA laboratories. Cu, Cr, Ni, and Hg were used for homogeneity testing. Reference values were obtained by pooling results from INAA, ICP-AES and AAS. The comparison of reference values with the consensus values (mean of all results from the PT participants) showed large differences in several elements. Out of 53 requests for the material only 29 sets of results were obtained.

- preparation of mussel reference material. The material was homogenized, sterilized, and characterized for a large number of elements. Results were reported to the participating laboratories. The final report was distributed to the coordinators at the meeting.

Within the frame of the IAEA Technical Cooperation project BGD/8/018 the IAEA Chemistry Unit prepared a water reference material to be used in a proficiency test for the laboratories of Bangladesh involved in arsenic analysis in water. The water was collected from laboratory tap water (underground origin) at Seibersdorf. The samples were spiked with aqueous mono elemental arsenic standard 1000 mg As/L was used with different dilutions. The uncertainty associated with the dilution was estimated according to the guide for Quantifying Measurement Uncertainty issued by EURACHEM/CITAC (2000).



A proficiency test for the determination of trace elements in a soil and sediment matrix was organized for the IAEA Technical Cooperation project RAS/2/010 “Quality Assurance and Quality Control of Nuclear Analytical Techniques”. A total of 15 sets of samples were sent out to participants. The results have been discussed at a project meeting.

According to the programme of the IAEA Technical Cooperation project RAS/9/024 “Environmental Radiation Monitoring and Regional Data Base”, soil and milk reference materials were prepared for a proficiency test where 14 Asian countries participated in analysing alpha, beta and gamma emitters. Two sets of samples were prepared; each consisted of spiked matrix samples and the relevant standard solutions:

- one set contained spiked soil and spiked milk powder (250g each) and an ampoule with a standard solution of the relevant radionuclides; this set was designed for gamma-spectrometric measurements;
- the second set contained three equal samples of soil (5g each) spiked with a mixture of ^{238}Pu , $^{239+240}\text{Pu}$, ^{241}Am and ^{90}Sr radionuclides, one ampoule with a standard solution of Pu+Am radionuclides and one ampoule with ^{90}Sr standard solution.

An interlaboratory study for determination of radium and uranium isotopes (^{226}Ra , ^{228}Ra , ^{234}U , ^{235}U and ^{238}U) in water was organized. This exercise was a reaction to the increasing demand for support in analysing natural radioactivity in environmental samples.

A total of 13 different water samples (five natural waters and eight spiked samples) were prepared. 149 sets of sample were sent out to 147 participants from 58 countries. In total, 965 units with water samples and 316 ampoules with radioactive standards were distributed. Statistical evaluation of the reported data and individual reports were issued in 2004.

A proficiency test for the determination of a suite of ten trace elements in an IAEA-338 lichen material was performed. To better evaluate the laboratory performance a quality control

(lichen) sample and an ampoule of a trace element standard solution were supplied with the sample. This exercise attracted the interest of 169 laboratories from 66 Member State countries. The data have been collated and individual reports have been submitted to the participants.

For 2005 the following activities are expected:

- refinement of stock, widening the offer and replacing materials (matrices) that are running out of stock;
- improvement of processing and storage facilities;
- implementing an ISO-compliant quality system to ensure the quality of the materials available and the quality of the whole process of preparation of reference materials and organization of intercomparison exercises;
- preparation of a new phosphogypsum reference material (IAEA-434) for the determination of naturally occurring radionuclides and trace elements. The material, a waste product of the phosphate fertilizer production process, was collected from a fertilizer manufacturing plant in Poland. This material is relevant to environmental studies as it is often released to the environment in waste streams or finds use in the construction industry. The homogeneity test and the bottling of the material have been completed. Several expert laboratories have been invited to participate in a characterization study with the objective of assigning property values for some radionuclides and trace elements;
- preparation of a new soil reference material, with isotopic composition of depleted uranium. The material was collected at sites where depleted uranium ammunition had been used. Several expert laboratories will be invited to participate in a characterization study with the objective of assigning property values for some radionuclides and trace elements;
- preparation of a new grass reference material (IAEA-372) for the determination of artificial and naturally occurring radionuclides;
- preparation of a new soil reference material for the determination of artificial and naturally occurring radionuclides.



Assistance to ALMERA network laboratories to provide accurate radionuclide analysis in environmental samples

For 2005 the following activities are expected:

- a soil sampling intercomparison exercise in an agricultural area, qualified as a "reference site", to assess the uncertainties associated with soil sampling methodologies;
- the annual meeting with the ALMERA members to evaluate the current status of the laboratories, to improve their technical competence through standardization of sampling, monitoring and measurements protocols and staff training. During the meeting the final
- report of the first proficiency test organized with labelled soil samples will be presented and discussed. In this study, laboratory means from 53 laboratories were compared with target values. Outliers were identified according to the Grubbs test and eliminated. The trueness and the precision of the analytical data were evaluated. The participants' results

- were evaluated against acceptance criteria for accuracy and precision. Corrective actions for the laboratories were proposed where necessary.

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