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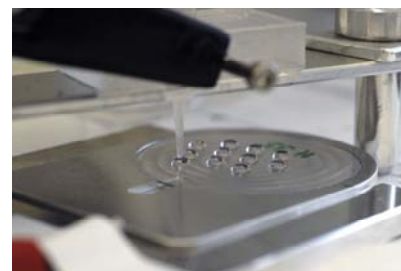
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ALMERA Activities

Procedures for determination of radionuclides in environmental samples

Reliable, comparable and 'fit for purpose' results are an essential requirement for any decision based on analytical measurements. For the analyst, the availability of tested and validated analytical procedures is an extremely important tool for production of such analytical measurements. For maximum utility, such procedures should be comprehensive, clearly formulated, and readily available to both the analyst and the customer for reference.

As reported in the second issue of the ALMERA newsletter (<http://www-pub.iaea.org/MTCDC/publications/PDF/Newsletters/ALM-NL-02.pdf>), one of the most frequent requests which Member State laboratories make to the IAEA Terrestrial Environmental Laboratory in Seibersdorf, Austria is that for recommended analytical procedures. The approach being taken for development of specific procedures and methods is to first review the literature on a given topic, and then based on this review develop a method written in accordance with ISO guidelines. The activities mentioned above are meant to be of general use to a wide range of laboratories. In parallel, a set of rapid procedures and methods needs to be developed for the ALMERA network of laboratories.



Preparation of the IAEA matrix reference materials

In the following are reported the most recent outcomes of the ALMERA activities aimed towards the development of a set of procedures for determination of radionuclides in environmental samples.

Review paper on ^{210}Po determination in environmental samples and recommended procedure for the determination of ^{210}Po in water samples by alpha-spectrometry

The activity for the development of specific procedures and methods started with a review of methods for determination of ^{210}Po in environmental samples. The determination of ^{210}Po is relatively straightforward, due to the ease of source preparation by spontaneous deposition onto metal surfaces and the uncomplicated alpha spectrum. Although several optimization studies have been carried out, published source preparation methods remain remarkably diverse. The review mainly focused on analytical methodology of ^{210}Po . About 130 papers were collected and reviewed. The literature surveyed included analysis of air, fresh water, rainwater, seawater, soil, sediment, coal, tobacco, phosphogypsum, foodstuffs, marine organisms, vegetation, human bone, and biota [1]. Based on the review paper a selected recommended procedure was validated in terms of repeatability, reproducibility and trueness [2]. Finally, IAEA published a procedure for the determination of ^{210}Po in water samples by alpha-spectrometry [3].

Development and application of on-line sequential injection system for the separation of Pu, ^{210}Po and ^{210}Pb from environmental samples

It is usual to preconcentrate target radionuclides from environmental samples before measurement, because of their low activities in the sample and the presence of interfering elements and radionuclides. Coprecipitation, solvent extraction, ion exchange and extraction chromatography are the commonly used methods for radionuclides. The chromatographic separation is a time consuming process, taking at least 5-6 hours to separate Pu. Sometimes, it may take much longer due to clogging caused by bulky matrix in the sample solution or by bubbling due to incomplete decomposition of NaNO_2 which is added to adjust the oxidation state of Pu. The IAEA developed an on-line sequential injection system, automatically operating, in order to shorten the separation time of radionuclides from the sample matrix. The on-line sequential injection system was applied to the separation of Pu from an IAEA reference material (IAEA Soil-6) and to the sequential separation of ^{210}Po and ^{210}Pb from phosphogypsum [4]. The calculation of uncertainty in the sequential determination of ^{210}Pb and ^{210}Po by liquid scintillation counting and alpha-spectrometry was published [5].

Review paper for the determination of Pu isotopes by alpha-spectrometry

The major disadvantage of alpha spectrometry originates from the necessity for the complete separation of Pu from the sample components in order to obtain 'infinitely' thin alpha sources. Radiochemical procedures, often fairly sophisticated, have to be applied to the samples to remove major and minor components. Nevertheless, alpha spectrometry is most commonly used for the determination of ^{238}Pu and $^{239+240}\text{Pu}$, due to the relatively low price of the equipment and the low background.

The IAEA has reviewed the radiochemical procedures that are currently the state-of-the-art for the determination of plutonium nuclides in various matrices, focusing attention on environmental samples and alpha spectrometry and comparing this technique with other techniques, like mass spectrometry. The review paper [6] was intended to give an overview about alpha spectrometric determination of Pu isotopes from the early nineties till the recent years. A brief description of the 'old' procedures is also included to show major trends. The literature survey is based on the publications of the last 20 years referred in the International Nuclear Information System (INIS) database.

Procedure for the rapid determination of Pu isotopes and ^{241}Am in soil and sediment samples by alpha-spectrometry

Americium (Am) and plutonium (Pu) nuclides are artificial radionuclides, produced in the nuclear fuel cycle as well as in atomic bombs. They have been released to the environment from various sources, mainly due to accidents in nuclear facilities (reactors, reprocessing plants) and to nuclear weapons tests. Detection of americium and plutonium nuclides in soil and sediment samples is of high interest during nuclear incidents for controlling and protecting the environment and furthermore to protect the population from the consequences of a possible contamination. In an emergency situation, only rapid analytical methods can provide adequate information for fast response.

Alpha spectrometric measurements have high sensitivity due to the low background and the high counting efficiency, and alpha spectrometric devices are available in many nuclear laboratories in contrast to other more sophisticated measuring equipment as mass spectrometers (ICP-MS, AMS). In this framework, the IAEA developed a rapid method for the determination of Pu isotopes and ^{241}Am by alpha spectrometry. The procedure includes sample destruction by fusion using lithium metaborate, separation by extraction chromatography using TRU® resin (Eichrom Industries

Inc.) and source preparation with neodymium fluoride micro-co-precipitation. The whole procedure can be performed within 6-7 hours followed by overnight counting of the alpha sources, thus providing results within 24 hours [7-9].

Review paper of determination of Pu concentration and its isotope ratio by ICP-MS

There are several different techniques used for the determination of Pu isotopes, including alpha-spectrometry, liquid scintillation counting (LSC), fission track, LX/-alpha ray measurement and mass spectrometry (MS). Alpha-spectrometry is the most widely used analytical technique due to its easy application and relatively low instrumentation cost. However, alpha-spectrometry is not a particularly sensitive technique for the determination of low levels of Pu, sometimes requiring from days to several weeks counting time for environmental samples. In addition, it is also difficult to distinguish ^{239}Pu and ^{240}Pu due to the small difference in their alpha particle energies, restricting the use of this technique in the determination of the Pu isotope ratio.

By contrast, mass spectrometry (MS) is an atom counting technique with several advantages over decay counting techniques for the determination of Pu isotopes. It enables the determination of low levels of Pu with a low detection limit, while requiring only a short detection time. In addition, it enables the accurate determination of ^{239}Pu and ^{240}Pu , and hence their isotope ratio. ICP-MS has strong advantages in both quantitative and isotope ratio measurements of Pu with respect to the relatively low analysis cost, easy sample loading and compatibility with diverse sample introduction systems by means of direct or indirect injection of aqueous or solid samples.

The IAEA has reviewed the sample pre-treatment and chemical separation of Pu for various kinds of samples, which depends on sample matrix, level of Pu concentration and sample amounts. The review included detection limit, sensitivity, background, hydride generation level, precision and accuracy according to the types of sample introduction system [10].

Rapid simultaneous determination of ^{89}Sr and ^{90}Sr in milk

In emergency case, the rapid analysis of ^{89}Sr and ^{90}Sr (radiostrontium) in milk is essential, since milk is a substantial pathway of radiostrontium to the human body, especially in infants. The analysis time is an important economic factor, rapidly identifying whether foodstuff is contaminated with radioactive materials

and whether the decision makers should take protection and intervention actions for protecting the public from radiation hazards. The analysis procedure of radiostrontium is always complicated because ^{89}Sr , ^{90}Sr and its daughter ^{90}Y are pure beta emitters, and prior to measurement they must be separated from the sample.

A rapid simultaneous determination procedure of ^{89}Sr and ^{90}Sr in milk using two energy windows was proposed [11], in which the separation method combining cation/anion exchange resin and Sr resin was used for the removal of Ca, Ba, ^{90}Y and other interfering elements, and applied to the spiked milk samples for the validation of the method. This work was implemented in the frame of the ALMERA Asia-Pacific regional group, with the aim of the development of recommended rapid method of ^{89}Sr and ^{90}Sr in milk. This method will be further developed and completed through inter-laboratory comparison in 2010.

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ALMERA MEETINGS

Minutes of the 6th Coordination meeting, Budapest (Hungary), 23-25 November 2009

The 6th ALMERA coordination meeting took place in Budapest, Hungary, from 23 to 25 November 2009 and was hosted by the Radio-analytical Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate.

The meeting was officially opened by Mr. Ákos Józwiak, the Deputy Director of the Central Agricultural Office, Food and Feed Safety Directorate. He presented the activities of the Directorate.

The meeting was attended by the following 51 participants from 22 countries, representing 32 different institutions. Representatives of the Korea Institute of Nuclear Safety (KINS), as coordinating centre of the ALMERA Asia-Pacific regional group, and representative of the Comissão Nacional de Energia Nuclear, Instituto de Radioproteção e Dosimetria (CNEN-IRD, Brazil) as coordinating centre of the

ALMERA North and Latin America regional group, attended the meeting:

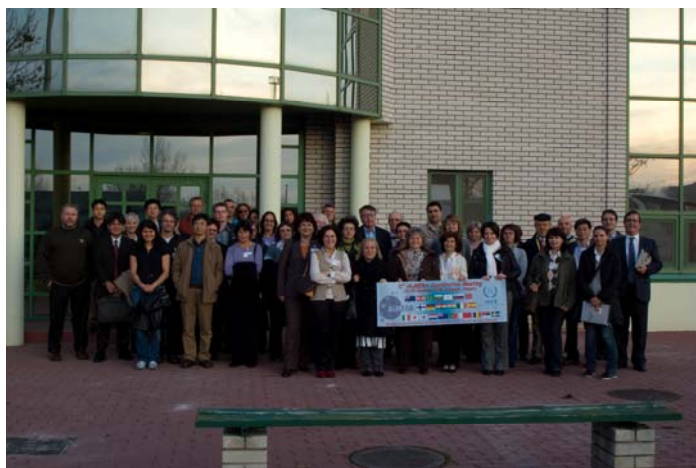


6th ALMERA Coordination meeting, Budapest (Hungary) 23-25 November 2009

IAEA	Chang Kyu Kim	IAEA, Seibersdorf Laboratories, Austria
	Umberto Sansone	IAEA, Seibersdorf Laboratories, Austria
	Abdulghani Shakhashiro	IAEA, Seibersdorf Laboratories, Austria
Australia	Daniela Fierro	Australian Nuclear Science & Technology Organization (ANSTO)
	Sandra Mary Sdraulig	Australian Radiation Protection & Nuclear Safety Agency (ARPANSA)
Austria	Franz Schoenhofer	BMLFUW i.R.
Brazil	Ana Cristina De Melo Ferreira	Brazilian National Commission for Nuclear Energy (CNEN), Instituto de Radioproteção e Dosimetria (IRD)
	Fabiana Ferrari Dias	Brazilian National Commission for Nuclear Energy (CNEN), Pocos De Caldas
Bulgaria	Valentin Georgiev Avramov	Kozloduy Nuclear Power Plant
	Lyubomir Aleksandrov Popov	Kozloduy Nuclear Power Plant
Cyprus	Anastasia Caballero	State General Laboratory, Ministry of Health
Germany	Zsolt Varga	European Commission, JRC, Institute for Transuranium Elements

Hungary	Tünde Ádámné Sió	Hungarian Agricultural Authority, Food & Feed Safety Directorate, Central Radio-analytical Laboratory
	Edit Bokori	Radanal Ltd
	Ferenc Deák, Éva Frindt	Department of Food Chain Control, Ministry of Agricultural and Rural Development Hungarian Agricultural Authority, Food & Feed Safety Directorate, Central Radio-analytical Laboratory
	Ákos Józwiak, Zsuzsa Molnár	Hungarian Agricultural Authority, Food & Feed Safety Directorate Radanal Ltd
	Tímea Sebestyén	Hungarian Agricultural Authority, Food & Feed Safety Directorate, Central Radio-analytical Laboratory
	Nóra Vajda	Radanal Ltd
	Beáta Varga	Hungarian Agricultural Authority, Food & Feed Safety Directorate, Central Radio-analytical Laboratory
	Sándor Tarján	Hungarian Agricultural Authority, Food & Feed Safety Directorate, Central Radio-analytical Laboratory
India	Sudheedran Vattaparambil	Bhabha Atomic Research Centre, Radiation Standards Section
Ireland	Alison Mary Mcintyre	Radiological Protection Institute of Ireland
Italy	Pierino De Felice	ENEA INMRI, Istituto di Radioprotezione, Laboratorio Casaccia
	Enzo Ferraris	Centrale Elettronucleare di Trino, So.G.I.N Laboratories
	Giorgia Iurlaro	ENEA, Istituto di Radioprotezione, Laboratorio Casaccia
	Daniela Lunescu	ARPA Lombardia, Agenzia Regionale per la Protezione dell'Ambiente della Lombardia
	Rosella Rusconi	ARPA Lombardia, Agenzia Regionale per la Protezione dell'Ambiente della Lombardia
Japan	Tetsuya Sanada	Japan Chemical Analysis Center
Korea (Republic of)	Jung Seok Chae	Korea Institute of Nuclear Safety
	Kun Ho Chung	Korea Atomic Energy Research Institute
	Yongjae Kim	Korea Institute of Nuclear Safety
Netherlands	Jan Marius Kok	NRG
	Frits Peter Moet	NRG
New Zealand	Nikolaus Helmut Hermanspahn	National Radiation Laboratory
Norway	G. Elis Holm	Norwegian Radiation Protection Authority
Poland	Maria Malgorzata Suplinska	Central Laboratory for Radiological Protection
Portugal	Maria Irene Pais Lopes	Instituto Tecnológico e Nuclear
	Maria José Bação Madruga	Instituto Tecnológico e Nuclear
Romania	Cristina Bucur	Nuclear Power Plant Cernavoda
	Cristian Nicolae Dulama	Institute for Nuclear Research
	Mirela Dulama	Institute for Nuclear Research
	Alexandru Toma	Institute for Nuclear Research
	Ruxandra Cristina Toma	Institute for Nuclear Research

Serbia	Gordana Pantelić	Institute of Occupational & Radiological Health
Slovenia	Jasmina Kozar Logar	Institute Jozef Stefan
Spain	Margarita Herranz Soler	Escuela Técnica Superior de Ingeniería, Departamento de Ingeniería Nuclear y Mecánica de Fluidos, Laboratorio de Medidas de Baja Actividad
	Fernando Legarda	Escuela Técnica Superior de Ingeniería, Departamento de Ingeniería Nuclear y Mecánica de Fluidos, Laboratorio de Medidas de Baja Actividad
Sweden	Lilian Del Risco Norrlid	Swedish Radiation Safety Authority



Participants of the 6th ALMERA Coordination meeting, Budapest (Hungary), 23-25 November 2009

Objectives of the meeting

The overall aim of the meeting was:

- to discuss the implementation of the current activities of the ALMERA network;
- and to define the future activities of the ALMERA network.

Current activities of the ALMERA Network

Mr. Chang Kyu Kim from the IAEA was the scientific secretary of the meeting and he presented the current status of the network. The IAEA's programme related to ALMERA has included activities aimed towards the development of a set of procedures for determination of radionuclides in environmental samples.

The activity started with the publication of review papers on the development and validation of an analytical method for ^{210}Po in water, and a rapid method for Pu isotopes and ^{241}Am in soil and sediment. In addition to these activities, a rapid method for $^{89}\text{Sr}/^{90}\text{Sr}$ in milk was proposed in 2007 by the Asia-Pacific ALMERA Regional Group and it is being developed. In addition review papers will be published in 2010 related to analytical procedures for ^{228}Ra and ^{226}Ra determination in water, and the methodology for indoor radon surveys.

Mr. Abdulghani Shakhashiro from the IAEA presented and discussed with the participants the results of the IAEA-CU-2008-04 ALMERA proficiency test on the determination of naturally occurring radionuclides in phosphogypsum and spiked water and the results of the IAEA-CU-2009-04 ALMERA proficiency test on the determination of gamma emitting radionuclides in simulated air filters. In addition, Mr. Abdulghani Shakhashiro presented the 2010 and 2011 plan of IAEA proficiency tests for the ALMERA network. The participants expressed their need for organizing a proficiency test on ^{226}Ra -in soil and ^{226}Ra , ^{228}Ra , ^{234}U , ^{238}U , gross alpha/beta in water. The proficiency test samples are planned to be distributed in June, 2010.

Mr. Pierino De Felice, from the Italian National Ionizing Radiation Metrology Institute (ENEA INMRI) presented the preparation of simulated air filters for the IAEA-CU-2009-04 ALMERA proficiency test on the determination of gamma emitting radionuclides. The filters were prepared in the IAEA Seibersdorf laboratories (Austria), with the scientific support of the Italian National Ionizing Radiation Metrology Institute. The presentation of Mr. De Felice included also a technical evaluation, from the analytical point of view, of the results of the IAEA-CU-2009-04 ALMERA proficiency test gamma emitting radionuclides in simulated air filters.

Mr. Sandor Tarjan, Head of the Radio-analytical Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate, presented the results of the homogeneity test of simulated air filters for the IAEA-CU-2009-04 ALMERA proficiency test on the determination of gamma emitting radionuclides.

On 24 November 2009, all participants moved to the Paks nuclear power plant, located 5 km from Paks in central Hungary, for a workshop on rapid method for radionuclides determination in environmental samples. In the workshop, five lectures were presented by invited experts.



Visit to the Paks nuclear power plant

Ms. Nóra Vajda, from Radanal Ltd, Hungary, gave a lecture on ‘Rapid method of Pu isotopes and ^{241}Am in soil and sediment samples using alpha-spectrometry’.

Mr. Zsolt Varga from the European Commission Joint Research Centre (Institute for Transuranium Elements), presented a lecture on ‘Measurement of long-lived radionuclides by ICP-MS’.

Mr. Franz Schönhofer, from BMLFUW, i.R, Austria, discussed the use of liquid scintillation spectrometry for rapid determination of radionuclides in the environment.

Mr. Chang Kyu Kim from the IAEA gave a lecture on ‘application of sequential injection system to radiometric and non-radiometric measurement techniques of radionuclides’.

Ms. Beáta Varga, from the Radio-analytical Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate gave a lecture on ‘Radio analytical monitoring for ecological vulnerability and sustainability’.

After the workshop, all participants visited the maintenance centre of the Paks nuclear power plant where the structure of the reactor vessel and steam generator of the pressurized water reactor are installed for practice and educational purpose of the reactor maintenance staff.

TABLE 1

Work plan for validation of rapid method of $^{89+90}\text{Sr}$ in milk using a combined method of Cherenkov and scintillation counting

Time	Work plan	Institute
January–April 2010	Spiked milk powder preparation <ul style="list-style-type: none"> ▪ blank ▪ milk powder spiked with ^{89}Sr & ^{90}Sr ($^{89}\text{Sr}/^{90}\text{Sr}$ ratio: 1~ 5) ▪ milk powder spiked with ^{89}Sr and ^{90}Sr ($^{89}\text{Sr}/^{90}\text{Sr}$ ratio: 10~15) 	IAEA Seibersdorf & Central Agricultural Office, Hungary
May 2010	Distribution of the milk powder and blank samples to participants	IAEA Seibersdorf
June 2010	Determination of radionuclides in the samples (5 replicates for each sample) and report to the IAEA	All participants
Sept. 2010	Preparation of the IAEA report	All participants

On 25 November 2009, the ALMERA participants discussed the validation result of the rapid method of $^{89}\text{Sr}/^{90}\text{Sr}$ in milk developed by the ALMERA Asia-Pacific group. Mr. Chang Kyu Kim from the IAEA presented the validation result of the method and application result of the measurement technique combining Cherenkov and scintillation counting for simultaneous determination of $^{89}\text{Sr}/^{90}\text{Sr}$ in milk. All laboratories which had participated in the validation of rapid method of $^{89}\text{Sr}/^{90}\text{Sr}$ in milk also presented their results and discussed the validation results and the future plan.

Mr. Chang Kyu Kim from the IAEA introduced the future plan for the validation of a candidate of recommended procedure of $^{226}\text{Ra}/^{228}\text{Ra}$ in water using Liquid Scintillation Counting.

Conclusions

The ALMERA participants proposed and agreed that for the period 2010-2014, the ALMERA coordinating centre for the Europe regional group will be the Radio-analytical Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate, Budapest, Hungary.

The ALMERA participants were informed about the request made to the IAEA by the Syrian Arab Republic Atomic Energy Commission (AECS) to coordinate the ALMERA regional group for Middle East. The ALMERA participants considered this request acceptable, in order also to involve more laboratories from Middle East into the network.

The ALMERA participants agreed that the validation study for the rapid method of $^{89}\text{Sr}/^{90}\text{Sr}$ in milk was not concluded and agreed to extend this study during 2010, involving where possible additional other ALMERA laboratories. The work plan proposed is provided in Tables 1 and 2.

TABLE 2

Work plan for preparation of IAEA's report on rapid method of $^{89+90}\text{Sr}$ in milk using a combined method of Cherenkov and scintillation counting

Time	Work plan	Institute
January 2010	Foreword and Introduction of the report	IAEA Seibersdorf
February 2010	Preparation of spiked milk powder Homogeneity test of spiked milk powder	Central Agricultural Office, Hungary
April 2010	Preliminary test results from each participating laboratory	All participants
June 2010	Analysis results of spiked milk for the validation of rapid method of $^{89/90}\text{Sr}$ in milk using a combined method of Cherenkov and scintillation counting	All participants
Sept. 2010	Preparation of IAEA procedure of rapid method of $^{89/90}\text{Sr}$ in milk	IAEA Seibersdorf

The ALMERA participants proposed to start in 2010 the activities related to the validation of recommended procedure for the determination of $^{226/228}\text{Ra}$ in water using Liquid Scintillation Counting (LSC). The proposed work plan is described in Table 3.

TABLE 3

Work plan for validation of recommended procedure of ^{228}Ra and ^{226}Ra in water by LSC

Time	Work plan	Institute
April 2010	Preparation of spiked water samples – 3 different level	IAEA Seibersdorf
June 2010	Characterization of spiked water samples Distribution spiked samples to participating laboratories	IAEA Seibersdorf
Sept. 2010	Analyze spiked samples	Participating labs
November 2010	Data evaluation for the validation of a candidate recommended procedure: <ul style="list-style-type: none"> ▪ Repeatability ▪ Reproducibility ▪ Trueness ▪ Interference effect ▪ Measurement range ▪ Detection limit ▪ Uncertainty evaluation Preparation of a recommended procedure	IAEA Seibersdorf & participating labs

The ALMERA participants proposed and agreed that the Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate, Budapest (Hungary) will coordinate both projects on the validation of rapid method of $^{89}\text{Sr}/^{90}\text{Sr}$ in milk and the validation of a candidate of recommended procedure of $^{226}\text{Ra}/^{228}\text{Ra}$ in water using Liquid Scintillation Counting.

The ALMERA participants requested the IAEA to organize during 2010 the following proficiency tests:

- $^{226,228}\text{Ra}$, uranium, gross alpha/beta in three water samples;
- ^{226}Ra in soil.

The ALMERA participants requested the IAEA to organize a workshop in the IAEA, Agency's Laboratories, Seibersdorf, in the 3rd quarter 2010, to discuss coincidence summing and geometry correction in gamma ray spectrometry.

The ALMERA participants requested the IAEA to organize during 2011 a workshop on liquid scintillation

technique. The feasibility of the workshop, possible date and location could be defined during the 2010 ALMERA coordination meeting.

The Comissao Nacional de Energia Nuclear, Instituto de Radioprotecao e Dosimetria (CNEN-IRD, Brazil) as coordinating centre of the ALMERA North and Latin America region regional group, proposed to organize a workshop in Brazil in 2010 for the Latin America ALMERA members. The main aim of the workshop will be to harmonize the radio-analytical techniques, gamma spectrometry in particular, between the ALMERA laboratories of the area. On this basis, CNEN-IRD will formally request the IAEA to assist them in the implementation of this workshop.

The ALMERA participants agreed to have the 7th ALMERA coordination meeting in Addis-Ababa (Ethiopia), in the 4th quarter 2010, hosted by the Ethiopia Radiation Protection Authority. A preliminary meeting with the Ethiopian counterparts took place in Addis-Ababa from 16 to 18 December 2009, in preparation for the proposed meeting in Ethiopia,

It was announced that the IAEA will start during 2010 the characterization of the Korean soil, which is one of candidate IAEA reference materials, to be used for proficiency test and requested all ALMERA members the analysis contribution for the characterization of the samples.

Minutes of the ALMERA meeting at the Ethiopian Radiation Protection Authority, Addis-Ababa (Ethiopia), 16-18 December 2009

The meeting in Addis-Ababa (Ethiopia), from 16 to 18 December 2009, took place at the Ethiopian Radiation Protection Authority headquarters and had as its objective preparation for the 7th ALMERA network coordination meeting, which will take place in Addis-Ababa in October 2010. The Ethiopian Radiation Protection Authority is an ALMERA member since 2005. It is organized under the Ethiopian Science and Technology Agency and led by Ethiopian Radiation Protection Board (ERPBB), which consist of a group of state ministers and director generals of different ministries. In this respect, the ERPA has also the legal task to ensure an adequate system in monitoring any increase in the level of radiation in the environment, and to provide for proper management of radioactive wastes on the alert for interventions during radiological and nuclear emergency situations.

The meeting was attended by Mr. Yohannes Balcha Tedla, acting Director General of the Ethiopian Radiation Protection Authority, Mr. Atnatiwos Zeleke Mashasha and Mr. Markos Fikreab.

The ALMERA network laboratories agreed to contribute to the characterization and homogeneity studies of the IAEA candidate reference materials by providing analytical services. The contribution will be acknowledged in the IAEA official publications. Laboratories interested to participate in the characterization and homogeneity studies have to submit their availability at the following email address: ALMERA@iaea.org

The JCAC (Japan Chemical Analysis Centre) offered a reference materials (Japanese cedar leaves) already prepared in Japan to be used as an IAEA reference material for proficiency test. It was agreed that the JCAC will provide the IAEA about all the information related to this material in order to verify the feasibility of the JCAC request.

The ALMERA participants requested the IAEA to organize a meeting in Vienna, to define programme and actions in the field of environmental data evaluation (identification of the relevant pathways and driving parameters; organization of training for young researchers in sampling, data evaluation, transfer parameters evaluation, development of site specific prediction models and tools applications).



Visit at the gamma spectrometry laboratory

An overview, structure and current status of the ALMERA network was presented to the staff of the Ethiopian Radiation Protection Authority followed by a very active discussion. The discussion pointed out the need to take more active actions to involve into the ALMERA network, institutions from Africa. It was also pointed out to involve into the ALMERA network institutions linked with radioecological studies (modelling, environmental parameters definition, etc.).

The facilities and laboratories of the Ethiopian Radiation Protection Authority were visited.

It was agreed that the 7th coordination meeting will take place in Addis-Ababa (Ethiopia) from 11 to 13 October 2010, and will be hosted by the Ethiopian Radiation Protection Authority. The Ethiopian Radiation Protection Authority has officially requested the IAEA to support the organization of the meeting covering, as was in the past for the ALMERA meetings, the attendance costs for some of the ALMERA members.

It was agreed that the main aim of the meeting will be the establishment of an ALMERA regional group for Africa and to discuss and harmonize the analytical counting and sampling techniques between the radio-analytical laboratories of Africa. The meeting will be also addressed to verify the feasibility to link the ALMERA network with the MARLIN network in marine radioecology.



During the meeting it was pointed out that several Technical Cooperation Projects are strongly linked with the activities of the ALMERA meeting and it was recommended to contact the respective technical officers to verify the interest of the IAEA Technical Cooperation Department to be involved in the next ALMERA coordination meeting, mainly addressed to radio-analytical laboratories interested on harmonization of analytical counting and sampling techniques.

ALMERA INTERLABORATORY COMPARISON EXERCISES

The IAEA-CU-2009-04 proficiency test on the determination of gamma emitting radionuclides in simulated air filters

In the frame of the IAEA efforts to monitor and demonstrate the performance and analytical capabilities of the ALMERA network members, and to identify gaps and problem areas where further development is needed a proficiency test was organized by the Terrestrial Environment Laboratory in Seibersdorf, to assess the technical capacity of ALMERA Members in analysing radionuclides in simulated air filters.

The proficiency test set consisted of four filters. The participating laboratories were requested to analyze ^{54}Mn , ^{57}Co , ^{59}Fe , ^{60}Co , ^{65}Zn , ^{109}Cd , ^{133}Ba , ^{134}Cs , ^{137}Cs , ^{152}Eu and ^{241}Am in filters 01, 02 and 03. The participants were informed that only some of the listed radionuclides were present in the filters. The participating laboratories were provided with a control filter 04 containing only ^{60}Co and ^{133}Ba with known activities to the participants to be used as a control for the efficiency calibration.



Simulated air filters

Test items (simulated air filters) were prepared and distributed to 69 participants from 46 countries on 15 September 2009. The deadline for reporting of results was set to three working days from the date of package delivery, confirmed by the forwarder tracking system. However, the on-line results' reporting system was available for each participant during one week from the date of package delivery. The whole proficiency test was concluded in one month by 16 October 2009.

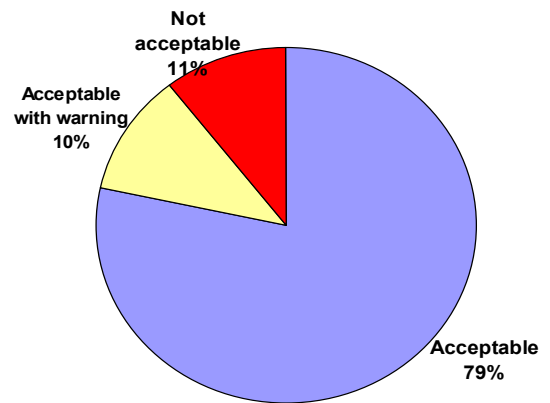


Proficiency test materials set

In this proficiency test, 877 measurement results were reported to the IAEA from 53 laboratories in 36 IAEA Member States. The performance evaluation results demonstrated the competence of ALMERA network laboratories in determination of gamma emitting radionuclides in air filters, 79% of all reported results obtained acceptable score and only 11% of all reported results failed to meet the proficiency test criteria. The following figure shows the summary evaluation of the reported results.

The IAEA wishes to thank the participant laboratories to this proficiency test. In particular the IAEA is grateful to Mr. Pierino De Felice, from the Italian

National Metrology Institute for Ionizing Radiation (ENEA-INMRI, Rome, Italy), who provided the technique for the preparation of the simulated air filters and to Mr. Sandor Tarjan, from the Radio-analytical Reference Laboratory of the Central Agricultural Office, Food and Feed Safety Directorate of Hungary, who carried out the homogeneity test on all the simulated air filters.



Summary evaluation of the reported results

OTHER NEWS

The new IAEA-413 reference material: Major, Minor and Trace Elements in Algae

http://www-pub.iaea.org/MTCD/publications/PDF/IAEA-AQ-14_web.pdf

Within the frame of the IAEA activities in production and certification of matrix reference materials a new reference material, the IAEA-413: Major, minor and trace elements in algae was produced and certified. Reference values for the mass fractions and associated standard uncertainties have been established for: As, Ca, Cd, Co, Cr, Fe, K, Mg, Mn, Na, Ni, Pb and Zn. Information values were reported for Cu and Hg.

The property values assigned to the algae IAEA-413 reference material are element mass fractions, expressed in the derived SI unit mg/kg. The utmost care was taken regarding the metrological traceability of the property values assigned to this reference material already at the planning phase and during the entire characterization process. Laboratories participating in the characterization campaign were requested to carefully choose the calibrants and to provide the IAEA with all related information, including certificates.

During sample production and certification, the requirements for reference material production and certification as stated in ISO guides 34 [1] and 35 [2] were taken into account. This reference material is intended to be used for quality assurance purposes, basically as a quality control material for the measurement of the elemental composition of biological materials especially of biomonitors, for the assessment of a laboratory's analytical work and for the validation of analytical methods.

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ICTP/IAEA Advanced School on in-situ X ray Fluorescence and Gamma Ray Spectrometry

An advanced school on in-situ X ray fluorescence and gamma ray spectrometry was conducted from 26 to 30 October 2009, in Trieste (Italy), (http://cdsagenda5.ictp.trieste.it/full_display.php?smr=0&ida=a08186). This school was jointly organized by the IAEA Terrestrial Environment Laboratory and the IAEA Nuclear Spectrometry and Applications Laboratory, together with the Abdus Salam International Centre for Theoretical Physics (ICTP).

X ray fluorescence (XRF) and gamma ray spectrometry techniques have successfully been applied in the field and in industrial environments for the in-situ analysis which cover the analysis of artefacts and materials that have not been moved from their original place of deposition/storage, soil screening for metals, indoor and outdoor air pollution monitoring, screening of contaminated areas in emergency situations, mapping of large seabed area to estimate the levels and distribution of natural and/or anthropogenic radionuclides, radioactive mapping of terrestrial environment, monitoring of airborne materials and building materials, investigation of the radiation field in the vicinity of sunken objects, decontamination assessment etc.



Practical experience in 'in-situ' measurements by gamma ray spectrometry techniques

A modern portable analyzer based on XRF and gamma ray spectrometry brings to the field site unsurpassed savings in time and labour as well as an excellent

performance often matching that of the laboratory instrument. Major advantages of portable (or transportable/movable) analyzers include simplicity, speed of operation and flexible requirements for sample preparation. XRF and gamma ray spectrometry methods applied for in-situ analysis provide immediate analytical results in a truly non-destructive way which is of prime importance in the fields of environmental, archaeological and industrial applications.



Practical experience in 'in-situ' measurements by gamma ray spectrometry techniques

The school presented the recent advances in this area as well as the benefits to apply these techniques. It also created an opportunity for scientists from developing countries to initiate collaboration with the more advanced laboratories on using portable XRF and gamma ray spectrometry and associated in-situ analytical methodologies. The school represented a possibility for scientists and students of UN, UNESCO and IAEA Member States, and in particular members of the IAEA ALMERA network, to work on practical exercises, and to refresh and up-date their knowledge and skills in X ray fluorescence (XRF) and gamma ray spectrometry techniques. In this framework a field exercise was organized in collaboration of the 'Sezione di Fisica Ambientale' of ARPA Friuli-Venezia Giulia, Provincial Department of Udine (Italy).

Staff member farewell



In April, our colleague Ms. Adelaide Maria Gondin Da Fonseca Azeredo returned to work in her native country, at the Brazilian National Commission for Nuclear Energy (CNEN), Instituto de Radioprotecao and Dosimetry (IRD), the Coordinating Centre of the ALMERA North and Latin America regional group. Adelaide spent four years with the Terrestrial Environment Laboratory in Seibersdorf, and provided strong support to all environmental activities of the laboratory. Highlights included coordination of the consultancy groups on radioactive tracers in the environment and the ALMERA soil sampling exercise, support to the air filter proficiency test, and preparation of numerous technical reports including IAEA-TECDOC-1616 and the report of the technical meeting on radon as an atmospheric tracer. The staff of the Terrestrial Environment Laboratory wishes her all success in her future career.

New ALMERA members

- The Radiological Protection Institute of Ireland in Dublin joined the ALMERA network in October 2009.
- The National Council for Science and Technology of the Republic of Kenya joined the ALMERA network in February 2010.
- The Institut National des Sciences et Techniques Nucléaires (Madagascar-INSTN) of the Republic of Madagascar joined the ALMERA network in February 2010.

ALMERA currently (May 2010) consists of 122 laboratories representing 77 countries.

ALMERA IN MEMBER STATES

The Radio-analytical Reference Laboratory of the Central Agricultural Office – Food and Feed Safety Directorate, Hungary

The Food and Feed Safety Directorate of the Central Agricultural Office was established in 2007 continuing the activities of the former National Food Investigation Institute (NFII). The mission of the Directorate is to provide safe food and feed to the consumers and to ensure confidence in food-chain safety, through a complex network providing control, legislation support, risk assessment and analysis. Currently the Directorate consists of two Sections, the Authority and the Laboratory.

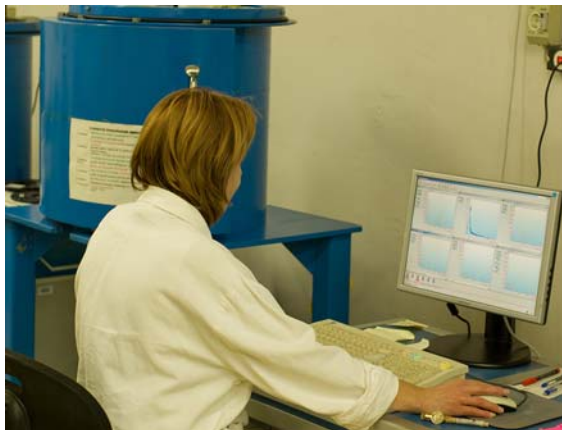
The Radio-analytical Reference Laboratory is part of the Laboratory Section. The laboratory was established in 1959 and later it became the coordinator of a network of analytical laboratories, namely Radio-analytical Control Network of Hungarian Ministry of Agriculture and Rural Development. The radio-analytical monitoring activities were strengthened after the Chernobyl accident of 1986 and at the beginning of 90's the network consisted of 19 county laboratories and 6 laboratories involved in food industries. The network has been reduced during the last years and currently it consists of nine laboratories including the Radio-analytical Reference Laboratory of Food and Feed Safety Directorate of the Central Agricultural Office. The laboratories of the network are accredited according to MSZ EN ISO/IEC 17025:2005.

The Radio-analytical Reference Laboratory is an active member of the IAEA ALMERA network since 1995 and the ALMERA Coordinating Centre for the European region for the period of 2010-2014.



Staff of the Radio-analytical Reference Laboratory

In addition, since 2005 the Radio-analytical Reference Laboratory works as an IAEA Collaborating Centre for the production and characterization of matrix reference materials of terrestrial origin. In this field, the primary objective is the production and characterization of reference materials, the organization of interlaboratory comparison and proficiency test exercises, and the development and validation of radio-analytical procedures. These activities are closely linked with the IAEA's Subprogrammes to enhance the reliability and comparability of measurement results obtained by nuclear and nuclear related analytical techniques in Member State laboratories through the provision of reference materials, organization of proficiency tests and intercomparison exercises, and training.



The Radio-analytical Reference Laboratory is also an active member of the IAEA Advisory Group for the production and characterization of reference materials of terrestrial origin.

The Radio-analytical Reference Laboratory has facilities for alpha, beta and gamma spectrometry, and radiochemical separation for radiostrontium, ^3H , ^{14}C and transuranic isotopes. The government supports the execution of the monitoring programme

Low background gamma ray spectrometry system

The Radio-analytical Reference Laboratory and the environmental radioactivity monitoring

The monitoring programme includes the control of the radioactivity in food and feed produced in Hungary, the import of these products, the agricultural area and the production in the vicinity of the Hungarian nuclear facilities. The Radio-analytical Reference Laboratory acts as coordinating centre of the Radio-analytical Control Network of Hungarian Ministry of Agriculture and Rural Development. The objective of the Radio-analytical Control Network is to maintain a group of laboratories capable of the following:

- providing systematic regular radiological monitoring survey programme implemented throughout the entire territory of Hungary;
- providing reliable and timely analysis of environmental samples in the event of an accidental or intentional release of radioactivity in the environment and;
- being a source of reliable and consistent information and advice for government bodies.



System for homogenization of solid materials

The role of the Radio-analytical Reference Laboratory, as central coordinator of the Radio-analytical Control Network is:

- to coordinate the network and develop medium and long-term targets to help the laboratories to improve their capabilities in routine and emergency situations;
- to evaluate the analytical performance of the network laboratories by organization of interlaboratory comparison exercises and support the laboratories to enhance the quality of their analytical measurement in radionuclide determination;
- to ensure that the results produced by the laboratories are traceable to SI whenever possible thus enabling their comparability;
- to study the scientific literature, collect and disseminate various information regarding the radio-analytical work, quality assurance, risk assessments, radioecology;
- to develop guidelines for sampling, sample collection and treatment, analytical methods, etc.;
- to convene and chair the annual network coordination meeting;
- overview the monitoring program on yearly basis and make the necessary changes according to the need of the society (changes of consumption, food and feed production, changes in the field of export and import strategies, etc.)
- to train the technical staff belonging to the network.



Carbolite: automatic combustion system for ^3H and ^{14}C sample preparation

Currently the Network is able to provide the following:

- determination of radiostrontium (2000 samples/year);
- determination of gamma emitting radionuclides (3500 samples/year);
- gross alpha measurement (2000 samples/year);
- alpha-spectrometry (U-isotopes, Pu-isotopes, ^{241}Am) (100 samples/year);

- tritium (50 plant samples/year and 300 water samples/year);

The Radio-analytical Control Network of Hungarian Ministry of Agriculture and Rural Development has to support radionuclide measurement also in emergency situation, when the measurement results should be ready in a short time. The laboratories of the network are trained to rapid response, including the delivering of their analytical results with sensitivity of the measurement and accuracy optimized for this purpose. In the case of an accidental release of radioactivity in the environment the laboratories belonging to the network are able to provide the following:

- determination of radiostrontium (100 samples/week);
- determination of gamma emitting radionuclides (1500 samples/day);
- gross alpha measurement (80 sample/day);
- tritium in water (50 sample/day)
- and data presentation on digital map.

The countrywide monitoring program is performed by nine laboratories and in order to achieve the total spatial coverage, each of them is responsible for two or three counties of Hungary, as reported in the following table:

Counties	Gamma spectrometry	Low background beta counter	Low background alpha counter	Alpha spectrometry	Liquid scintillation
Bács-Kiskun	+	+			+
Borsod-Abaúj-Zemplén	+	+			
Budapest (RRL)	+	+	+	+	+
Fejér	+	+			
Hajdú-Bihar	+	+	+		
Somogy	+	+			
Tolna	+	+	+	+	
Vas	+	+	+	+	
Veszprém	+	+			

Analytical capabilities available in the Radio-analytical Control Network of Hungarian Ministry of Agriculture and Rural Development

A central data collection system is implemented for the management of the sample data and measurement results and connected to a Geographical Information System for data evaluation and mapping. Risk assessment and vulnerability studies, including the

determination of spatial differences regarding the radionuclide transport, is also important part of the work of the laboratory.

Hungary, as European Union country, has harmonized the relevant measurement techniques in compliance

with EU Directives and Recommendations, such as the Council Directive 96/29 Euratom of 13 May 1996 laying down basic safety standards for the protection of the health of workers and the general public against the dangers arising from ionizing radiation, the Council Directive 98/83/EC of 3 November 1998 on the quality of water intended for human consumption, and the Commission Recommendation 2000/473/Euratom of 8 June 2000 on the application of Article 36 of the Euratom Treaty concerning the monitoring of the levels of radioactivity in the environment for the purpose of assessing the exposure of the population as a whole.

The training is also an integral part of the activities of the Radio-analytical Reference Laboratory. IAEA fellows are regularly hosted in the laboratory and trainings are organized at national and international level. Close collaborations with the Hungarian Universities offer the opportunity for students to do their experimental activities and to complete the diploma under the supervision of experts.

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The Italian Institute for Environmental Protection and Research (ISPRA)

The Italian Institute for Environmental Protection and Research (Istituto Superiore per la Protezione e la Ricerca Ambientale - ISPRA) is a national public body, established by the law 133/2008. ISPRA is subject to the supervision of the Ministry of Environment, Territory and Sea and to the control of the Italian Supreme Court of Accounts.

ISPRA deals with environmental issues of national relevance which include the promotion, diffusion and enhancement of environmental protection and related research activities. It represents the fulcrum of a strategic system based on cooperation and mutual referrals of a network of Italian Regional and Provincial Environmental Protection Agencies (ARPA/APPA). This environmental network system is able to guarantee a truly efficient exchange of information and skills

across the entire national territory for the environmental improvement.



The ISPRA Laboratories in Rome, Italy

The Italian Institute for Environmental Protection and Research represents the focal point for the European Environmental Agency and in this capacity ISPRA collects and elaborates the environmental data produced in Italy at regional/provincial level by the Regional and Provincial Environmental Protection Agencies.

The Institute results from the merging of three former institutions, the Agency for Environmental Protection and Technical Services (APAT), the Central Institute for Scientific and Technological Research Applied to the Sea (ICRAM) and the National Institute for Wildlife (INFS).

ISPRA provides technical and scientific support to environmental decision makers, particularly to the Ministry of Environment, Territory and Sea, by offering the necessary tools and know-how to address economic and social changes, while safeguarding the environment and following the sustainable development paths agreed within the European Union.

The technical and scientific assets of the Institute contribute to support better the environmental governance, providing a wide range of expertise in several key areas such collection, processing, management and diffusion of environmental data and information; surveying and evaluation of environmental and wildlife heritage and of its evolution and interaction with other environmental components; project development for the recovery and improvement of wildlife communities and natural environments; protection of water resources and of marine and coastal areas; monitoring of marine environmental quality; prevention and mitigation of impacts of polluted marine and coastal sites; soil protection; climate change; sustainable use of natural resources; habitats and biodiversity protection; study and evaluation of physical and human factors influencing environmental conditions.



ICP-MS measurements

In addition to these activities, ISPRA plays an important role in assisting laboratories of the Italian Regional and Provincial Environmental Protection Agencies laboratories to enhance the quality of their analytical measurement data. This is accomplished through the provision of matrix reference materials and

validated procedures, training in the implementation of quality control, and through the evaluation of measurement performance by organization of proficiency tests and intercomparison exercises.

Collaboration of ISPRA with the IAEA

ISPRA is member of the IAEA Advisory Group for the production and characterization of reference materials of terrestrial origin and since 2005, it is an official member of the IAEA ALMERA network. ISPRA has collaborated with the IAEA Chemistry Unit of the Terrestrial Environment Laboratory in Seibersdorf, in Austria, in the production of reference materials and in the organization of ALMERA proficiency testing for soil sampling. In this framework ISPRA has collaborated in the preparation of the IAEA-450 algae material containing trace elements. A key comparison K75 for Pt and Ni and a parallel pilot study P118 for As, Cd, Cr, Hg, Ni, Pb and Pt using this algae material were organized by the Inorganic Analysis Working Group (IAWG) of the CCQM in 2009.



GC-MS measurements

In addition, ISPRA collaborated with the IAEA in a soil sampling intercomparison exercise for the ALMERA members [1]. The soil sampling intercomparison exercise took place in November 2005 in an agricultural area qualified as a 'reference site' in the frame of the SOILSAMP international project, funded and coordinated by ISPRA and aimed at assessing the uncertainty associated with soil sampling in agricultural, semi-natural, urban and contaminated environments [2]. The 'reference site' is located in the North Eastern part of Italy (Pozzuolo del Friuli, Udine), in the research centre belonging to the Agenzia Regionale per lo Sviluppo Rurale del Friuli Venezia Giulia (ERSA). The 'reference site' is characterized in terms of spatial/temporal variability of trace elements. The trace elements present at the reference site are of natural and anthropogenic origins [3]. The objective of the exercise was to compare soil sampling strategy/pattern and sampling techniques utilized in the ALMERA network in the case of the determination of

several radionuclides mean values in an agricultural area of about 10,000 m².

ISPRA reference materials and inter laboratory comparison exercises

Reference materials are one of the tools used to obtain comparability of analytical results. In recent times, there has been increasing interest worldwide in the accuracy, traceability and comparability of analytical measurements and the role that matrix reference materials play in the process. The ultimate goal of any measurement process is to ensure accuracy and to establish traceability to common universal reference points (preferably the SI) through an unbroken chain of comparisons.

The reference materials produced by ISPRA are matrix reference materials, widely used for method and measurement validation. Matrix reference materials incorporate the analytes of interest in a natural matrix, which is identical or similar to the test sample being measured. The advantage of matrix reference materials is that they help identify matrix effects in the measurement process.



Particle size measurement by laser diffraction

The ISPRA laboratory for the production and characterization of reference materials was established in 1999 and is part of the Environmental Metrology Service. Since January 2009 the Environmental Metrology Service has been formally accredited by the Italian Accreditation Body for Calibration (SIT) according to ISO Guide 34 'General requirements for the competence of reference material producers' and ISO/IEC 17025 'General requirements for the competence of testing and calibration laboratories' for production and characterization of reference materials. The accreditation includes the methods utilized for homogeneity and stability studies (Dispersive X ray fluorescence -ED-XRF, Direct Mercury Analyser -DMA), the methods for the quality control of the materials as CHN and particle size analyser. For the characterization of the reference materials the methods accredited are the EN ISO 13656:2002 and ICP-MS.



ED-XRF measurements: sample holder

Reference materials are used for the organization of intercomparison exercises (proficiency testing and collaborative studies). Proficiency testing is a method for regularly assessing the accuracy of the analytical data produced by laboratories. In analytical chemistry, proficiency testing usually comprises the distribution of effectively homogenous portions of the test material to each participant for analysis as an unknown. The laboratories conduct the test under routine conditions, and report the result to the organizer by a deadline. The results generated in proficiency testing should be used for the purpose of a continuing assessment of the technical competence of the participating laboratories. 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'. Collaborative studies are generally organized to determine precision parameters (repeatability and reproducibility) of standardized methods or to assign value to reference materials (characterization). Laboratories participating in collaborative studies are generally 'expert' laboratories. Laboratories involved in proficiency testing use their routine methods, while those involved in collaborative studies apply well defined or standardized methods.

In Italy, the environmental monitoring activities, involving physical, chemical and biological measurements, are performed by more than one hundred Italian Regional and Provincial Environmental Protection Agency laboratories. To support these laboratories, since 2003, when the ISPRA laboratory for reference material production and characterization was completed, about twenty proficiency tests and collaborative studies were organized. Measurements and matrices covered by these exercises are reported in following table.

TABLE 1.
Proficiency testing and collaborative studies organized by ISPRA

Year	Measurement type	Matrices
2003	Trace elements	Sediment
2004	Trace elements	Compost
2004	Sampling – trace elements	Soil
2005	Trace elements	Water, soil
2005	Anions - cations	Water
2005	<i>Daphnia magna</i> EC ₅₀	Water
2006	Anions - cations	Water
2006	Trace elements	Soil
2006	Pesticides	Water
2007	Pesticides	Water
2007	Trace elements	Soil, Extract, Standard solution
2007	Micro-invertebrates	Water
2007	PAHs	Soil, Extract, Standard solution
2008	Trace elements – <i>Daphnia magna</i> EC ₅₀	Leachate, Standard solution
2008	Asbestos	Airborne fibers
2008	PCBs	Soil
2009	PCB - dioxins	Soil, Standard solution
2009	Electromagnetic fields	-
2009	Hydrocarbons	Soil
2009	Anions - cations	Water
2010	PM ₁₀	Air
2010	COD	Water

Concerning the characterization of reference materials, following the recommendation of the IAEA Advisory Group for the production and characterization of reference materials of terrestrial, depending of the type and intended use of the reference material the property values of the reference materials produced in the ISPRA laboratories are assigned through one of the approaches listed below,

1. From measurement results produced by a group of expert laboratories using the same or different well established, stable and validated measurement procedures, different methods and different calibrants. The property value derived according to this approach will be based on a consensus value;

2. From measurement results produced by one expert laboratory using well established, stable and validated measurement procedures, confirmed by results from two or more expert laboratories using the same or

different validated methods/procedures. In this case one expert laboratory performs characterization of the material in reproducibility conditions and establishes the property value and its uncertainty. Results from other expert laboratories, obtained by the same or different validated methods/procedures using the same or different calibrant, are used for confirmation purpose only;

3. Formulation. In this approach the material is spiked with standard solution of the analyte(s) of interest, blended or diluted. The property value is calculated from the amounts used, and the associated uncertainty is derived from data given in certificate of relevant analyte (measurand) and uncertainty components arising from operations, such as dilution and weighing.

4. Interlaboratory comparison study (retroactively). The property value is assigned as a consensus value

derived from results reported by participants using established statistical approaches.

Organizational structure of ISPRA laboratory for the production and characterization of reference materials

The organizational structure of the laboratory, the available equipment and the production quality control system were designed to separate the different production steps, employing different rooms equipped with specially designed de-dusting systems. All rooms are connected to a specially device and centrally installed dust extraction system. For reference materials production the laboratory is equipped with equipment for all drying steps at all combinations of temperature regulation and natural and artificial (inert gas) application, a glove-box system, equipped with a number air, gas and water delivery devices and connected to bottom water outlet. All machines for breaking, grinding and sieving may be rolled in and out according to the occurring needs. In this way operators may handle all critical materials in a dust-free fashion.



Microscope observation of fish cells

The production of matrix reference materials in the ISPRA laboratories involves the following six working steps:

- feasibility studies;
- preparation of raw materials;
- homogenization and bottling of raw materials;
- characterization of the reference materials;
- stability test of the reference materials;
- storage of final products.

The feasibility studies start with the identification of the matrix to be considered, the parameters to be analyzed and the variables which can influence the final results. The preparation of raw material is performed in an area fully equipped to carry out pre-treatment processes with large volumes of samples, including drying systems, a freeze-drying system, and grinding and sieving devices.



Filter weighing to determine the net mass gain on airborne particulate matter <math><10\ \mu\text{m}</math>

The area next to the preparation area is equipped with mechanical systems for homogenization of solid and liquid materials. Analyte homogeneity testing is of prime importance for the production of useable reference materials. It should demonstrate the validity of the assigned values and their uncertainties in the analysis of individual units (bottles). Homogeneity is comprised of two components, between-bottle and within-bottle homogeneity. The between-bottle homogeneity is verified from a number of bottles by the determination of the analytes on appropriate sample intake, ranging from a few mg for analytical methods consuming small amounts to hundreds of grams. The within-bottle homogeneity is assessed by replicate determinations on subsamples taken from individual bottles. The homogeneity determinations are performed to confirm that the variations between bottles are not statistically significant compared to the certified uncertainty. The commonly used general approach to assuring technically valid reference material analyte homogeneity testing is the use, for each analyte of interest, of a single method of the highest accuracy.

The analyses required in the homogeneity and stability studies have the support and use of the analytical facilities of the ISPRA laboratory. The Environmental Metrology Service uses a number of analytical techniques to carry out quantitative analysis in a wide range of environmental matrices in support of reference material production activities (e.g. bulk characterization, homogeneity testing, certification of reference materials, characterization of samples for proficiency tests, etc.).

The characterization of the reference materials, defined as the assignment of concentration data to the interested analytes which approaches as closely as possible the 'true value', together with uncertainty limits, is another step of prime importance for the use of reference materials. The key characteristic of a reference material is that the properties of interest are measured and assigned on the basis of the accuracy. The goal is the arrival at the best possible estimate of the 'true value'. It implies the reliable assignment of a value to a

property of a material. It encompasses selection of measurands, appropriate analytical methodologies, adequately calibrated and properly used.

To carry out homogeneity, stability and characterization studies, ISPRA has been equipped with an Energy Dispersive X Ray Fluorescence (ED-XRF), a Direct MERCURY Analyzer (DMA), an Atomic Fluorescence Spectroscopy (AFS) for determination of mercury in water samples, an Ionic Chromatography (IC), an Inductively Coupled Plasma Mass Spectrometry (ICP-MS), a High Performance Liquid Chromatography (HPLC), a gas chromatography mass spectroscopy (GC-MS) and a high resolution gas chromatography high resolution mass spectroscopy (HRGC/HRMS). For the quality control of the candidate reference materials the laboratory is equipped with Karl Fisher method designed to determine the water content in the materials, CHN elemental analyzer to determine the homogeneity of the candidate reference material through the measurement of the

content of elemental C and N and a Laser Diffraction particle size analyzer

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Publications of potential interest to ALMERA members

Quantification of Radionuclide Transfer in Terrestrial and Freshwater Environments for Radiological Assessments (IAEA-TECDOC-1616)

http://www-pub.iaea.org/MTCD/publications/PDF/te_1616_web.pdf

For many years the IAEA has published a set of documents aimed at limiting the radiation exposure of the population from various nuclear activities. The IAEA-TECDOC-1616 Quantification of Radionuclide Transfer in Terrestrial and Freshwater Environments for Radiological Assessments is intended to support IAEA Technical Reports Series No. 364 (TRS 364). It provides radioecological concepts, models, parameters and data for assessing site-specific past, present and potential future radiation exposures of humans and other biota in terrestrial and freshwater environments in different climate conditions.

It can be used for radioecological assessment of both routine discharges of radionuclides to the environment and accidental releases. In addition, it will serve as useful background documentation for other relevant activities, such as training in radioecology and radiation protection.

Handbook of Parameter Values for the Prediction of Radionuclide Transfer in Terrestrial and Freshwater Environments (IAEA Technical Reports Series No. 472)

http://www-pub.iaea.org/MTCD/publications/PDF/trs472_web.pdf

This publication is a revision of the IAEA Technical Reports Series No. 364. The revision takes account of progress in radioecology since the original publishing of Technical Reports Series No. 364 in 1994. The revised handbook provides transfer parameter values most commonly used in radiological assessment models. It covers radionuclide transfer in the terrestrial and freshwater environments, through food chains to humans, and provides the assessor with data for use in the radiological assessment of routine discharges of radionuclides to the environment. Some parts of the dataset may also be used for assessing the impact of accidental releases and potential future releases. Although transfer to non-human species is not specifically addressed in the publication, the parameters may also be useful for this purpose. Based on the extensive input and the expertise of an international expert group, this revised handbook serves as a useful tool for both regulators and assessors for assessing site-specific past, present and future radiation exposure to humans and biota in terrestrial and freshwater environments.

Soil Sampling Intercomparison Exercise by Selected Laboratories of the ALMERA Network

IAEA Analytical Quality in Nuclear Applications Series No. 1

http://www-pub.iaea.org/MTCD/publications/PDF/IAEA-AQ-1_web.pdf

ALMERA Proficiency Test on the Determination of Radionuclides in Spinach, Soil and Water

IAEA-CU-2007-04

IAEA Analytical Quality in Nuclear Applications Series No. 3

http://www-pub.iaea.org/MTCD/publications/PDF/IAEA-AQ-3_web.pdf

ALMERA Proficiency Test on the Determination of Po-210 in Water

IAEA-CU-2007-09

IAEA Analytical Quality in Nuclear Applications Series No. 4

http://www-pub.iaea.org/MTCD/publications/PDF/IAEA-AQ-4_web.pdf

ALMERA Newsletter

<http://www-pub.iaea.org/MTCD/publications/newsletter.asp?id=146>

Rapid method for the determination of actinides in soil and sediment samples by alpha spectrometry

N. Vajda, A. Toerveyi, G. Kis-Benedek, C. K. Kim, B. Bene and Zs. Macsik

Radiochimica Acta 97, 395-401 (2009)

Determination of Pu isotopes by alpha spectrometry: a review of analytical methodology

N. Vajda and C.K. Kim

Journal of Radioanalytical and Nuclear Chemistry, DOI 10.1007/s10967-009-0342-x (2009)

Development of extraction chromatographic separation procedures for the simultaneous determination of actinides

N. Vajda, A. Toerveyi, G. Kis-Benedek, C. K. Kim,

Radiochimica Acta 97, 9-16 (2009)

Method validation of a procedure for determination of ²¹⁰Po in water using DDTC solvent extraction and Sr. Resin

C.K. Kim, M. Ho Lee, P. Martin

Journal of Radioanalytical and Nuclear Chemistry, 279, 2, 639-646 (2009)

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