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XRF Newsletter

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X ray Fluorescence in the IAEA and its Member States

In This Issue

Activities in the IAEA XRF Laboratory

A few selected examples of recent activities and results in the field of XRF are presented.

In vivo investigation of the physiology of larvae, pupa, and adult species of Tsetse flies by high speed X ray phase-contrast imaging

The X ray phase-contrast radiography is a technique which enables imaging internal structures of soft tissue sections, whole small animals, especially insects, and other kinds of low atomic number samples where there is not enough material to produce satisfactory contrast by using X ray absorption radiography [1]. The advantage of the technique over the X ray absorption radiography is minimization of the radiation dose to the sample. The technique was used to investigate dynamic process in living Tsetse flies. In case of the Tsetse flies there is an interest in the in vivo investigation of the dynamics of mating, feeding, emergence of adult species from pupa, as well as in learning more about the morphology of specific organs in various stages of development.

The experiments were performed at the Tomo-Topo beamline, ANKA, Institute for Synchrotron Radiation (ISS), Forschungszentrum Karlsruhe GmbH, D-76344 Eggenstein-Leopoldshafen, Germany.

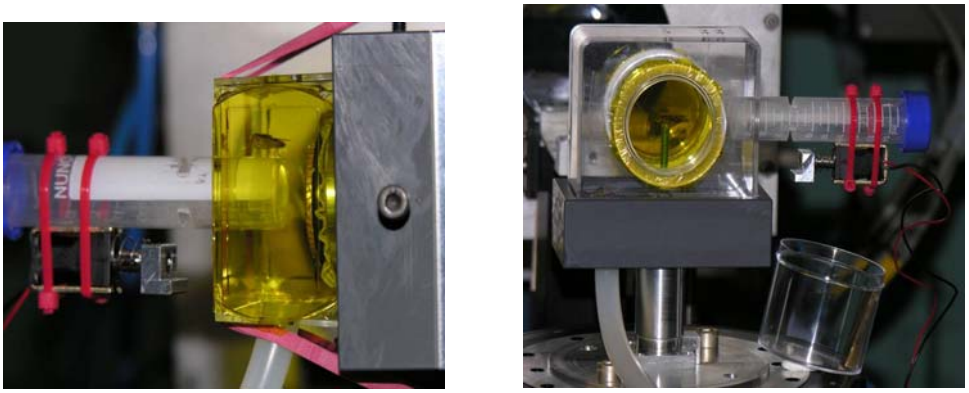

Fig. 1. Containers, supplied with humid air, used during the measurements of living Tsetse flies.

Two measurement set ups were utilized:

- Set up #1 was equipped with a high magnification X ray microscope consisting of a single crystal scintillator, magnifying optics coupled to an ultrafast, cooled CCD camera. The image resolution was about 2.5 micrometers and the filed of view was about 2 mm x 2mm.
- Set up #2 consisted of a medium magnification X ray microscope with high brilliance optics/single crystal scintillator coupled to an ultrafast, cooled CCD camera. The image resolution was about 20 micrometers and the field of view was equal to about 10 mm x 10 mm.

A 3 mm thick Si filter was used to harden the white synchrotron beam and reduce the total absorbed X ray dose by the specimen. The number of photons arriving at the sample after passing the Be exit window and the Si filter per pixel and second was approximately $8.5 \cdot 10^6$ (effective pixel size of 5.5 μm). Provisions were taken to sustain the life of the animals during the whole week. Particularly, food and feeding utensils were deployed to guarantee the fitting of the specimens.

Special measurement chambers were designed and manufactured to permit both the work with living, free animals in the synchrotron hutch and to reduce the stress under the measurement environment, see Fig. 1.

A total of 6 species of flies were taken for this study, males and females of *Glossina pallidipes*, *Glossina morsitans centralis*, *Glossina morsitans morsitans*, *Glossina swynnertoni*, *Glossina brevipalpis*, *Glossina fuscipes fuscipes.* A group of pregnant females (*Pallidipes)* was also taken as well as a group of larvae at different stages of development.

The imaging of living species was performed inside specially designed chambers. In the chamber a single female was glued to a pole and the male was inserted (flying free) by means of a remote device, see Fig. 1. The X ray synchrotron beam passed through two windows (entrance and exit) made of 7.5 micrometers thick KAPTON foil. During the mating a sequence of phase-contrast images was taken with a speed up to 250 frames per second and duration of a few minutes. Other processes recorded included fly emergence from pupa, larvae and pupa development. Several attempts were made to record the fly feeding, unfortunately without success. A few frames from the collected sequences are shown in Fig. 2.

During the experiment around 1,517 TB of data, in the form of X ray phase contrast enhanced images, were collected. That means that about 800,000 images were collected during 5 days measurement. These data are being processed and transformed by specially developed computer code. It is to remove image defects and artefacts and to increase the signal to noise ratio. The data are being evaluated in collaboration with the staff of the Entomology Unit, IAEA Laboratories at Seibersdorf.

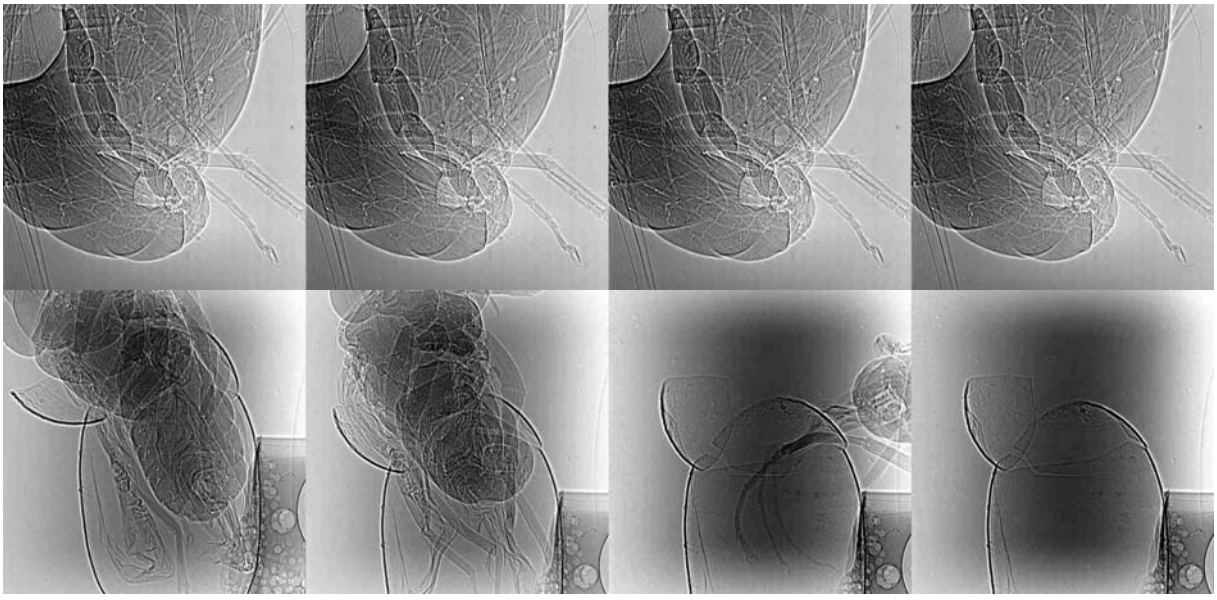

Fig. 2. Sequences of X ray phase-contrast enhanced images taken during mating (top) and emergence of Tsetse flies (bottom).

Conclusions

For the first time live, high-speed X ray phase-contrast image sequences of mating Tsetse flies were recorded with a spatial resolution in the single micrometer range. Other processes, including fly emergence from pupa, and various stages of larvae development, were also recorded in vivo. The recorded sequences of images provided sufficient information about the details of dynamic processes under study. The collected data will allow for better understanding of Tsetse flies physiology and characterization of the selected organs.

A large-scale, synchrotron facility appeared to be a useful tool to support the Agency's research programme on eradication of Tsetse flies. The unique data were collected with the advanced instrumentation at a minimum cost to the Agency.

References

[1] P. Cloetens, W. Ludwig, J. Baruchel, D. Van Dyck, J. Van Landuyt, J. P. Guigay, M. Schlenker (1999) Applied Physics Letters vol. 75 no 19, 2912- 2914.

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Proficiency test for XRF laboratories (PTXRFIAEA05)

The proficiency test (code PTXRFIAEA05) was the fifth worldwide exercise organized by the IAEA Laboratories at Seibersdorf in order to assist X ray fluorescence laboratories in assessment and improvement of their analytical performance. The test involved distribution to the participating laboratories a sample with established homogeneity and known target values of the mass fractions for a number of chemical elements. The test sample was marine sediment prepared and characterized by an independent external laboratory. The powdered, homogenized, and dried material was distributed to 49 laboratories in sealed plastic bottles, each bottle containing 100 g of the material.

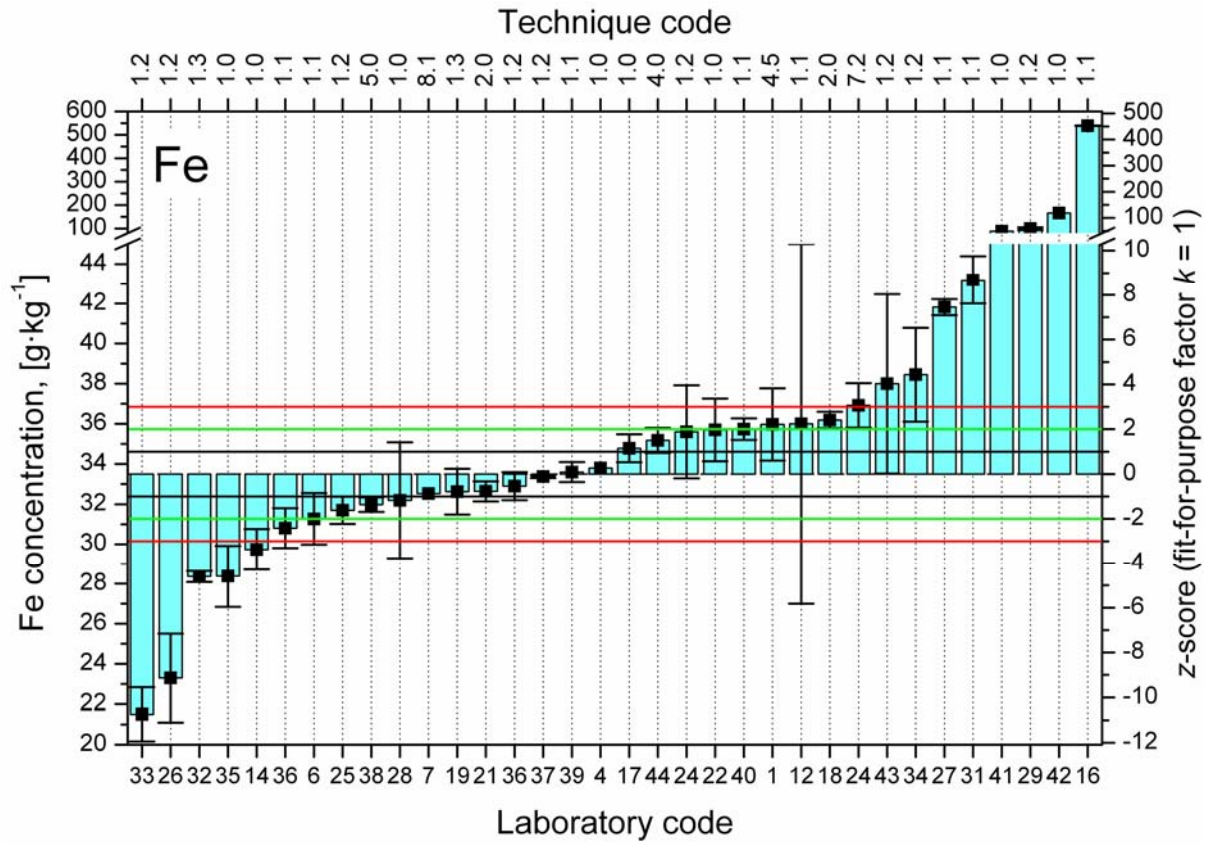

Fig. 1. Distributions of the z-scores for iron reported by at least 6 laboratories participating in thePTXRFIAEA05 proficiency test. The bar charts show the distance between the reported and the assigned mass fraction (concentration) of iron. The submitted results and their uncertainties, as provided by the participants, are marked with filled squares accompanied by uncertainty bars. The horizontal lines show the admissible levels of z-score for three different fit-for-purpose ranges defined by factor k in Eqn. (2): k = 0.5 - solid black lines, k = 1.0 - solid green lines, and k = 1.5 - solid red lines.

The participants were requested to determine the mass fractions of chemical elements according to their routine analytical procedures. They were also advised to determine the moisture content of the material by using a separate sample and to report the results on a dry-weight basis together with uncertainty. As usual, the laboratories were requested to analyze the sample using established techniques following their analytical procedures. The submitted results were processed, grouped versus analytes/laboratories and compared with the assigned values for the analytes. For the elements with known assigned values a set of *z*-scores and *u*-scores was calculated for each submitted result (see Fig. 1 as an example for iron). A *z*-score was calculated by comparing the assigned and the reported values according to the formula:

$$z = \frac{x - X_A}{\sigma_A} \qquad (1)$$

Where the term x denotes the reported mass fraction of elements, X_A, its assigned (known) value, and σ_A is the target value of the standard deviation calculated according to a modified Horowitz function [1]:

$$\sigma_A = kH_A, \quad k = 0.5, 1.0, 1.5 \qquad (2)$$

$$H_A = \begin{cases} 0.22 X_A & X_A < 1.2 \cdot 10^{-7} \\ 0.02 \left(X_A\right)^{0.8495} & 1.2 \cdot 10^{-7} \le X_A \le 0.138 \\ 0.01 \sqrt{X_A} & X_A > 0.138 \end{cases} \qquad (3)$$

The factor k defines the desired 'fit-for-purpose' uncertainty level: k = 0.5 - appropriate for high precision analysis; k = 1.0 - appropriate for well established routine analysis; k = 1.5 - satisfactory for common analytical tasks. When examining their results the participating laboratories select the k factor appropriate for their type of work. The result can be considered satisfactory if the absolute value of the *z*-score is less than or equal 2.

A *u*-score takes into account the laboratory estimate of the uncertainty, σ_x:

$$u = \frac{\left|x - X_A\right|}{\sqrt{\left(\sigma_A\right)^2 + \left(\sigma_x\right)^2}} \qquad (4)$$

The *u*-score is helpful in deciding whether the laboratory selected "fit-for-purpose" criteria were fulfilled.

Altogether 33 laboratories participated in the proficiency test submitting 556 individual results for 49 different chemical elements. The analytical techniques utilized included energy dispersive X ray fluorescence (EDXRF) with radioisotope (EDXRFISO) and X ray tube based (EDXRFTUBE) excitation, total reflection X ray fluorescence (TXRF), wavelength dispersive X ray fluorescence (WDXRF), proton induced X ray emission (PIXE), PIXE combined with proton induced gamma emission (PIXE-PIGE), neutron activation analysis (NAA), flame atomic absorption spectroscopy (FAAS), and inductively coupled plasma atomic emission spectroscopy (ICP-AES). The frequency of utilization of the techniques is shown in Fig. 2. The obtained results and the detailed description of the evaluation procedures were presented in the final report. Each laboratory was assigned a code to make sure that anonymity of the presented results is fully guaranteed. Based on the results of the proficiency test each participating laboratory should be able to assess a quality of the analytical results by using standard performance criteria and, if appropriate, to identify discrepancies, and finally to correct the analytical procedures. The next proficiency test exercise will be carried out in 2009. Further information on the proficiency test and the results obtained is available from Roman Padilla-Alvarez (A.Padilla-Alvarez@iaea.org).

References

[1] THOMPSON, M., *Analyst* **125**, 385 (2000).

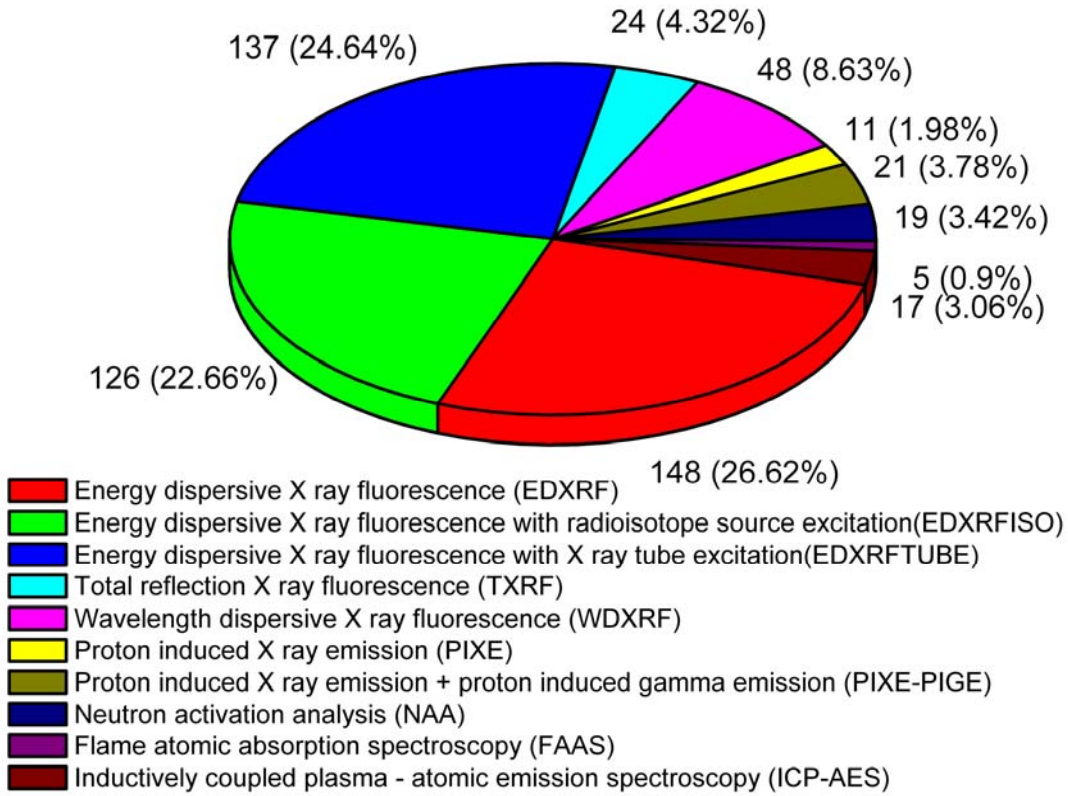

Fig. 2. Utilization of the analytical techniques by the participants of the PTXRFIAEA05 proficiency test. For each analytical technique the number of submitted results is shown. The per cent values relate to the total number of 556 submitted results.

Quality Management - related activities in the IAEA X ray fluorescence laboratory

One of the main missions of any analytical chemistry laboratory is provision of reliable results. Many X ray fluorescence laboratories in the IAEA Member States carry out research aimed at improving the performance and extending the applicability of various EDXRF techniques. The increased demand from society for reliable/certified analytical results impose an urgent need for implementation of quality management system (QMS) that often complies with the general requirements for the competence of testing and calibration laboratories of the latest approved ISO standard (ISO 17 025 : 2005).

The implementation of QMS in a chemistry laboratory requires the validation/verification of the used analytical methods. Although EDXRF is a well established and routinely applied for analysis, there are not many methods approved by international standardization bodies, and therefore a rigorous method validation becomes unavoidable. Some specific features of EDXRF techniques require a careful interpretation of the concepts related to quality control and method validation.

The IAEA X ray Fluorescence Laboratory, fully committed to endorsing the IAEA quality policy, has continuously been working to assist the laboratories in the Member States in their efforts to implement QMS. Since 2002, a series of contributions have been published in the IAEA XRF Newsletter related to different aspects of quality assurance. In 2005 a complete set of recommendations and associated documentation related to a general organizational structure of QMS was developed to serve as general guidelines to X ray fluorescence laboratories in support of their efforts to adopt quality assurance principles and policies. This documentation is ready for distribution upon request.

According to the recommendations for the organization of the work and effective functioning of the quality system minimum 3 staff members should be involved. Set of templates in documentation complies with the requirements of the ISO 17025 (2005) standard, which includes a quality manual and an organizational system for keeping the documented and validated analytical instructions. In the outlined operational procedures, an effort has been made to provide a comprehensive interpretation of the concepts of traceability, uncertainty estimation and internal quality control, as applied to the specifics of X ray fluorescence analytical

practice. In particular, recommendations are provided to assess the traceability of the XRF results by using suitable reference materials for both the calibration of the spectrometers and assessment of analytical performance of the applied methods. A general plan of actions to organize the implementation of internal quality control practice is suggested, which can be used to assess the precision and trueness of the obtained results in a systematic way. Detailed information can be found in a paper published elsewhere [*X ray Spectrom. 2007; **36**: 27–34*]

Since 2001, the IAEA X ray Fluorescence Laboratory has organized five proficiency test exercises (PTXRF), aimed at supporting the efforts of X ray fluorescence laboratories to assess and improve the quality and traceability of the results. The participation of the X ray fluorescence laboratories from Member States in these PT has continuously increased (see Figure 1), and the quality of the reported results has improved.

In total, 63 laboratories from 49 countries have taken part in the PTXRF exercises. Out of this amount, 28 laboratories have participated with certain continuity (two to four times), thus proving that such PT exercises are very relevant for the Laboratories QM programmes. PT exercises are extremely cost-effective, and any suggestions on the nature of the materials to be selected for the future PTs are welcome.

In 2009, the IAEA XRF Laboratory intends to develop an ICT module on implementation of Quality Management Systems for X ray Fluorescence analytical practice. This module shall provide training sessions that build up step by step an understanding of practical aspects of quality assurance (QA) for X ray fluorescence analytical practice including structure of quality management (QM) according the ISO/IEC 17025 standard.

Further information on Quality Management in the IAEA XRF Laboratory is available from Roman Padilla-Alvarez (R.Padilla-Alvarez@iaea.org).

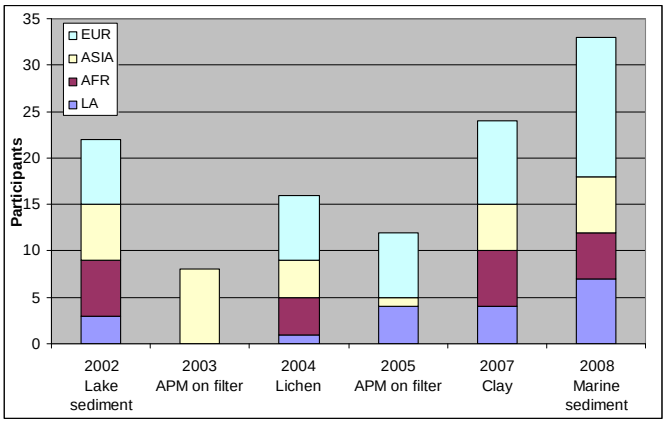

Fig.1 Increase in participation in the PTXRF exercises

Meetings and conferences

Technical Meeting on special configurations and new applications of micro-analytical techniques based on nuclear spectrometry, Vienna, Austria, 20 – 24 October 2008

Over the last years considerable progress has been observed in development and applications of micro-analytical techniques based on nuclear spectrometry. One of the major reasons for the developments was the possibility to use new excitation sources such as synchrotron radiation facilities and low-power X ray tubes combined with availability of X ray optics and thermoelectrically cooled semiconductor detectors. Several groups involved in the new applications of nuclear spectrometry are developing integrated instruments and analytical methodologies based on nuclear spectrometry. As a result the quality of characterization of various materials improved considerably and new applications became possible in support of applied research, teaching and education in nuclear science and technology, industry, environmental pollution monitoring, food and agriculture, human health, study of cultural heritage etc. The following nuclear spectrometry techniques can be used for microanalysis: (i) X ray fluorescence (XRF), (ii) total reflection X ray fluorescence (TXRF), (iii) ion beam analysis based on applications of low-energy particle accelerators (including particle X ray emission – PIXE, particle induced gamma-ray emission – PIGE, Rutherford backscattering spectrometry – RBS), (iv) extended X ray absorption fine structure spectroscopy (EXAFS) and X ray absorption near-edge spectroscopy (XANES), (v) fluorescent X ray micro-tomography, etc. The techniques are usually used for elemental analysis, 2D and 3D imaging and chemical speciation.

The range of possible applications of micro-analytical techniques is very wide and covers the following fields:

- Industrial applications including microelectronics, mineralogy, study of corrosion processes, measurement of coating thickness, study of catalytic materials, waste characterization
- Environmental and biomedical applications including individual particles analysis, biomonitoring, trace element mapping of tumour

tissues and living plants, study of waste disposal sites etc.

- Archaeological applications and study of cultural heritage objects including analysis and identification of artefacts in museums for authentication and provenance studies, analysis of artefacts for purposes of their preservation and restoration
- Forensic applications
- Food and agriculture

The Technical Meeting on special configurations and new applications of micro-analytical techniques based on nuclear spectrometry was organised by the IAEA Laboratories at Seibersdorf (Scientific Secretary: Andrzej Markowicz) under the IAEA project 1.4.3.4. ***Nuclear Spectrometry for Analytical Applications*** which a major objective is *to enhance capability of interested Member States to effectively utilize nuclear spectrometry and analytical services in industry, human health, agriculture and environmental pollution monitoring*.

A major objective of the Technical Meeting (TM) was to review the current status, developments, trends and applications of micro-analytical techniques based on nuclear spectrometry, and to produce a relevant report (proceedings). In particular, the participants assessed the capabilities, advantages and limitations of the techniques which should be of benefit for the developing Member States and contribute to the effective applications in support of scientific and technological development.

The following specific topics were discussed:

- State-of-the-art instrumentation for micro-analytical techniques
- Modern analytical methodologies
- Integration and complementary use of nuclear spectrometry techniques and benefit for characterization of materials
- Role of synchrotron radiation sources
- Progress in X ray optics
- New applications in support of innovation, research and technological development
- Further improvements in analytical performance of micro-analytical techniques including instrumentation and analytical methodologies
- Role of the IAEA in the promotion and improvement of access to advanced facilities for microanalysis and micro-tomography for the benefit of the developing Member States.

The TM was attended by participants from Austria, Croatia, Germany, Greece, Italy, Netherlands, Poland, Republic of Moldova, South Africa and Spain as well as by staff members from the IAEA Department of Nuclear Sciences and Applications (see photos 1 and 2).

Photo 1. Participants of the Technical Meeting (credit: A. Markowicz)

Photo 2. Visit to the XRF Group of the IAEA Laboratories at Seibersdorf (credit: A. Markowicz)

Major nuclear spectrometry-based micro-analytical techniques presented during the Technical Meeting are summarised below with emphasis on their advantages, limitations, new applications and current trends.

1. Ion beam analysis – methodology and applications

Brief overview of the techniques

Ion beam analysis (IBA) corresponds to a group of analytical techniques based on detection of interaction products leaving a sample exposed to an ion beam from an accelerator. IBA techniques provide information about the elemental concentration and are typically performed with protons or He ions accelerated to MeV energy range. Very strong advantage of IBA techniques is a possibility to obtain 2D and 3D distributions of

sample constituents (elements or isotopes). Perhaps the most known among the IBA techniques are RBS (Rutherford backscattering spectrometry) and PIXE (Particle Induced X ray Emission). RBS is based on elastic scattering of beam particles and nowadays is a routine technique for determination of composition and depth profiles of thin films. PIXE is based on the excitation of characteristic X rays and is currently widely accepted as one of the best techniques for routine and sensitive elemental analysis of air particular matter. Other IBA techniques are: nuclear reaction analysis (NRA), particle-induced gamma emission (PIGE), elastic recoil detection analysis (ERDA), scanning transmission ion microsocopy (STIM), ion beam induced charge (IBIC), ionoluminescence (IL) and secondary electron imaging (SEI). A concept of 3D imaging by using confocal arrangement has recently been proposed.

Applications

The current main application fields of microbeam IBA techniques are certainly biology and medicine, materials science and cultural heritage. Other fields of applications include earth sciences, monitoring of environmental pollution, agriculture, ecotoxicology, study of cultural heritage, determination of hydrogen in materials etc.

Advantages/benefits

A most important advantage of IBA techniques is simultaneous multielemental analysis and determination of the elements over practically entire periodic table. The analysis might be nondestructive and requires very small amount of material. For most of the elements the detection limits are in a ppm weight fraction concentration range for bulk samples (down to pg range in absolute units). In some cases spatial resolution can be below 1μm. An important feature is also a possibility to perform quantitative elemental microscopy. Some IBA techniques can be used simultaneously to provide complementary information about the materials under investigation.

Limitations

In some materials excessive irradiation currents may induce changes or losses of elements. In order to prevent (reduce) the effects, limited currents of the excitation beam are required that may, however, lead to the low counting statistics and decreased sensitivity. Another limitation is that IBA microanalysis requires dedicated beam lines at the particle accelerator which hampers access for a wider research/user community.

Current developments and trends

1) Confocal arrangement for 3D PIXE defined by the intersection between a focused ion microbeam, formed by a microprobe, and the sensitive detection field of an X ray polycapillary lens, placed in front of an X ray detector. When the position of a sample is sequentially changing with respect to this common volume, both lateral and depth-resolved (3D) information can be extracted with a spatial resolution of some micrometers depending on the performance of the nuclear microprobe and the X ray lens.

2) In order to fully utilize all possible IBA techniques at the microbeam line, a construction of universal systems with many IBA techniques integrated is required.

3) A need for microbeam analysis of unique objects (such as cultural heritage) requires development of new external microbeam systems. The use of thin beam exit foils (Si_3N_4), and performing PIXE/RBS measurement in He atmosphere improved capabilities and expanded applications of such facilities

4) New quadrupole focusing systems developed in different microbeam laboratories resulted in reduced beam spot sizes (down to 100 nm) which opened new application areas for IBA techniques.

5) Some laboratories developed cryo-systems for the analysis of samples at low temperature. This creates new capabilities for the analysis of biological specimens as well as in materials science.

Currently about 50 ion microbeam installations are available worldwide. Some other are under construction, and the laboratories that in the past applied broad beam IBA techniques, are now moving towards a more useful and powerful microbeam operation mode.

2. Synchrotron radiation-based micro-analytical techniques – methodology and applications

Brief overview of the methodologies and techniques

Synchrotron radiation (SR) is electromagnetic radiation generated by the acceleration of ultrarelativistic charged particles in magnetic fields. The advantages of SR over conventional sources are: high brightness and intensity, high collimation, wide energy/wavelength tunability (from infrared to hard X rays), specific polarization and spatial coherence, pulsed time

structure. SR facilities offer a new tool for researchers involved in many fields of science and technology.

Methodologies:

- X ray fluorescence (XRF) gives elemental information, qualitative and quantitative, even at trace element level. Can be performed at large or grazing angles (total reflection X ray fluorescence – TXRF).
- X ray absorption near-edge spectroscopy (XANES) and Extended X ray absorption fine structure (EXAFS) give detailed information about local chemical and oxidation state. Can be performed in transmission and fluorescence mode.
- InfraRed (IR) spectroscopies provide chemically specific information through molecule conformation and aggregation analysis, especially about biomolecules and secondary structure of proteins. Many approaches are used, e.g., Raman spectroscopy, Fourier Transform IR, etc.
- X ray photoelectron spectroscopy (XPS) gives information about chemical species and chemical environment of elements.
- X ray Diffraction (XRD) gives structural and chemical information of crystalline samples through analysis of X ray diffraction patterns. Can be performed on single crystals and on powder samples.
- X ray radiology gives morphological information through analysis of X ray attenuation.

Techniques:

- Full Field (or Imaging) techniques: a condenser lens illuminates the specimen, and a second objective lens behind the specimen generates a magnified image onto a spatially resolving detector.
- Scanning techniques: photon optics demagnify the incident photon beam to a small spot, as in the scanning electron microscope, and an image is acquired by detecting the photon or electron signal while rastering the sample.
- Phase-sensitive techniques: spatial coherence of the synchrotron radiation makes it possible to visualise and reconstruct the phase variations of the X rays. This significantly enhances the contrast in samples with light element constituents and in general materials with weak variation of absorption from point to point.
- Tomographic techniques: three-dimensional (3D) version of different techniques (radiology, XRF, XRD), based on the analysis of the sample from different orientations. The 3D image is reconstructed with dedicated algorithms.
- Confocal techniques: 3D version of different techniques (XRF, IR, XANES) obtained restricting the analysed volume by scanning of the sample in different directions. The data analysis is straightforward and can be applied to bulky samples.
- Diffraction enhanced techniques: make use of the scattered light from the specimen in addition to the direct absorption and phase information in order to increase resolution and/or to obtain additional morphological information.

Applications

Nanotechnology, neurochemical research, medical diagnosis and therapy, pathological biochemistry, toxicology, parasitology, cellular pharmacology, virology, cultural heritage, plant science, environmental science, earth science, agrifood technology, magnetic structures, forensic science, treatment of nuclear and non-nuclear waste, global warming, sterile insect techniques to reduce disease transmitting insects.

Advantages/benefits

Use of SR brings a strong improvement in analytical performances and capability to select optimum experimental parameters (photon energy, polarization, time structure, etc.). This implies very low detection limits (down 10^{-17} g of element from boron to uranium) with spatial resolution down to 40 nm, time-resolved experiments down to femtoseconds, improved chemical speciation down to picogram level. Photon-in/photon-out imaging and spectroscopies require simple specimen preparation and the sample itself can be studied in different environments or during changes of external conditions. In general, it is possible a complementary and versatile approach, which is strongly requested by multidisciplinary, research programs.

Limitations

The limitation of the single techniques (e.g., need of vacuum environment) is the same that is experienced with conventional laboratory sources. Sometimes versatility reduces performance as compared to single-technique methodologies. In general, access to the few existing SR facilities can be non trivial to some users.

Strict rules of SR use require particular attention in the experiment preparation.

Current developments and trends

- Development and improvement of focusing optics (polycapillaries, zone plates, multilayers, etc.), electron and X ray detectors
- Development of novel applications of coherence and diffraction imaging
- Development of new experimental setups for functional specimen environment.

The participants concluded that state-of-the-art IBA and synchrotron based micro-analytical techniques could play even more important and competitive role in the current and future research in various fields. These techniques provide unique possibilities to obtain chemical and structural information in micro- and nano-scale levels with a high sensitivity which is increasingly important in development of advanced technology and new materials, study and protection of cultural heritage, life and environmental sciences etc.

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Workshop on X ray techniques in the investigations of the objects of cultural heritage, Cracow, Poland, 18-19 September 2008

The Workshop was a satellite event of the 11th European Powder Diffraction Conference (EPDIC-11) which took place in Warsaw, Poland from 19-22 September 2008. The aim of the Workshop was to promote the current developments and use of X ray techniques for characterization of cultural heritage objects and to present the advanced techniques to analysts, scientists, curators and conservators. The participants from Austria, Czech Republic, France, Germany, Poland, Spain, and United Kingdom attended the event.

The program of the Workshop included poster and oral presentations and a visit to the National Museum in Cracow. The presentations were related to utilization of X ray radiography, X ray powder diffraction (XRD), X ray fluorescence (XRF), and X ray absorption near edge spectroscopy (XANES). The applications and examples of instrumentation used at synchrotron radiation facilities and portable spectrometers suitable for *in situ* characterization of objects were presented. Moreover, the methods of passive preventive conservation of valuable objects, particularly paintings, with the use of the so-called microclimate frames were discussed. During a visit to the National Museum in Cracow the conservation and preventive methods for unique and valuable objects were demonstrated. The participants supported the idea to continue such meetings associated to the future EPDIC.

Preservation of cultural heritage is relevant to the developed and developing IAEA Member States. In case of the developing countries a proper care about their cultural heritage is of particular importance in view of the expected revenue from tourism and associated commercial activities.

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Support to Technical Cooperation projects

The XRF Group at Seibersdorf provides assistance to a number of IAEA regional and national Technical Cooperation (TC) projects where XRF is used as one of the analytical techniques. The following events related to TC projects were supported in 2008:

Final Project Coordinators Meeting of the regional project RLA/7/011 (ARCAL LXXX) on 'Assessment of Atmospheric Pollution by Particles', Santo Domingo, Dominican Republic, 18 – 22 February 2008

The Meeting was attended by the following representatives from the countries participating in the project RLA/7/011: Rita Plá (Argentina), Eduardo Cortés Toro (Chile), Alfonso Salazar (Costa Rica), Juana Grizel Pérez Zayas (Cuba), José Bernardino Contreras Pérez (Dominican Republic), Francisca Aldape de Flores (Mexico), Javier Flores Maldonado (expert, Mexico) and Raiza del Valle Fernández Malave representing the Venezuelan Project Coordinator (see photos 1 and 2). The major objectives

of the Final Project Coordinators Meeting were: (i) to report and summarize the results of the project, (ii) to discuss the follow-up actions to ensure sustainability of the results of the project and (iii) to assess the achievements of the project against the performance indicators established at the beginning of the project implementation.

It was concluded that the agreed regional work plan was completed and the major objectives of the project were met.

Specific conclusions

- All countries have improved their knowledge in the implementation of procedures and techniques related to characterization of airborne particulate matter
- Regional expertise was effectively utilized as confirmed by recruitment of experts and lecturers available in the region
- Advanced nuclear analytical facilities available in the region were utilized in support of countries participating in the project
- Some laboratories initiated activities in air pollution monitoring by using nuclear analytical techniques, and regional cooperation was strengthened
- In order to observe trends and positive impact of new legislation a long term study (including sampling and analysis) of air pollution is required.

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Photo 1. Participants of the meeting (credit: A.Markowicz)

Photo 2. Visit to the analytical laboratory in ore mine and processing factory (credit: A.Markowicz)

Workshop on applications of nuclear analytical techniques for characterization and authentication of art objects, Daejeon, Republic of Korea, 11-13 June 2008

The Workshop was organised by the Korea Nuclear International Cooperation Foundation (KONICOF) in cooperation of the Daejeon Metropolitan Art Museum and the IAEA. More than 50 participants from Korea Nuclear Energy Foundation, Korea Atomic Energy Research Institute, Korea Institute of Nuclear Safety, Korea Research Institute of Bioscience and Biotechnology, Leeum – Samsung Museum of Art, National Institute of Scientific Investigation, Korean Art Appraisal Association, Korea Institute of Geoscience and Mineral Resources, National Cultural Properties Research Institute, National Science Museum, various universities and private enterprises attended the event (see photo 1). Recent progress and applications of synchrotron radiation sources, portable XRF spectrometers, ion beam analysis, and dating techniques for scientific investigation, restoration, conservation and authentication of cultural heritage objects were presented by the lecturers from Italy, Korea and IAEA. The Workshop was hosted by the Daejeon Metropolitan Art Museum (11-12 June 2008). The last day (13 June) a visit was organised to the Conservation Department of the National Museum of Korea, Conservation Institute of the Leeum – Samsung Museum of Art, and the Electrostatic Accelerator Research Centre of the Seoul National University (all in Seoul).

Photo 1. Participants of the Workshop attending a lecture (credit: A. Markowicz)

The Workshop confirmed a growing interest of the cultural heritage community in nuclear analytical techniques (NATs) as well as recognition of the benefits, advantages and uniqueness of these analytical techniques. The programme of the Workshop covered a wide spectrum of the topics including applications of laboratory and portable instruments, synchrotron radiation sources, low-energy charged particle accelerators and methodology of in situ measurements. The nuclear analytical facilities available in the Republic of Korea are fully adequate to provide meaningful support to the cultural heritage community. The organizers of the Workshop expressed interest in a follow-up meeting in order to assess the impact of the event and to introduce new techniques for a wider audience.

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Progress Assessment Meeting under the IAEA/RCA Technical Cooperation Project on 'Characterization and Source Identification of Particulate Air Pollution in the Asian Region' (RAS/7/015), Wellampitiya (Colombo), Sri Lanka, 25 – 29 August 2008

The meeting was organised by the Atomic Energy Authority of Sri Lanka in cooperation with the IAEA. The National Project Counterparts (NPCs) from Australia, Bangladesh, China, India, Malaysia, Mongolia, Myanmar, New Zealand, Pakistan, Philippines, Republic of Korea, Singapore, Sri Lanka, Thailand, Vietnam attended the meeting (see photos 1 and 2)

Photo 1. Participants of the meeting (credit: A. Markowicz)

Photo 2. Visit to the Atomic Energy Authority of Sri Lanka (credit: A. Markowicz).

Major objectives of the meeting were:

- To assess the current status and progress achieved under RAS/7/015 with an emphasis on the linkages of air particulate matter pollution to visibility and air quality management
- To review the current status of sampling and analysis, status and capabilities of nuclear analytical techniques, data interpretation, regional database and actions to ensure sustainability

- To identify gaps in the current set of data and the improvements required
- To assess the outcomes of the project against performance indicators
- To review and update the work plan for 2009 and 2010.

The country presentations showed a high level of end-user involvement in the project. The end-users are contributing to the project either in-kind or cash. The presentations demonstrated that most participants have several years of data that is used by end-users to assess the level of air pollution and in support of air quality management. The participating Member States are using the long-term database for fine and coarse air particulate matter for the identification of anthropogenic and natural pollution sources.

Following the workshop on degradation of visibility by fine particulate matter held in Kuala Lumpur, Malaysia in June 2008, most participating countries attempted to apply the techniques presented at the workshop to visibility data obtained locally, e.g., from nearby airports.

The reports presented by the national project coordinators indicate that the majority of the Member States have used HYSPLIT back trajectory methods for the identification of transboundary pollution events that occurred between 2003 and 2007. Natural and anthropogenic source emissions from wind blown soil, biomass burning and industrial events have been identified and quantified.

The currently available database is extensive and contains data from 14 of the 16 Member States covering time period from January 2001 to May 2008. The database contains three subsets of the masses and elemental concentrations for fine and coarse particulate matter, uncertainties and limits of detection.

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Workshop on harmonization procedures related to nuclear analytical techniques for characterization and preservation of cultural heritage artefacts (RER/1/006), Athens, Greece, 13 – 17 October 2008

The Workshop was attended by the specialists in nuclear and complementary analytical techniques applied for characterization and preservation of cultural heritage objects, and conservators/restorers from the following countries involved in the regional IAEA Technical Cooperation project RER/1/006 on 'Nuclear techniques for the protection of cultural heritage artefacts in the Mediterranean Region': Albania, Bosnia and Herzegovina, Croatia, Greece, The Former Yugoslav Republic of Macedonia, Malta, Romania, Slovenia, Serbia, and Turkey as well as by the lecturers from Germany, Greece and IAEA. The participants gave individual reports about the relevant activities in their countries and some of them presented the available procedures for NATs applied in support of CH studies. Only a few countries (Romania, Malta, and Turkey) have written NAT procedures which are currently applied only by their laboratories and further harmonization is required before they can be used by other countries in the region. Moreover, the laboratories realised an urgent need to establish quality assurance/quality control system in order to improve both the quality of analytical results and competitiveness of NATs. In order to help in these efforts the main elements of QA/QC were presented with a focus on technical requirements as specified in the ISO/IEC 17025 standard for the competence of testing and calibration laboratories related to in situ analysis.

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Announcements

International Atomic Energy Agency Technical Meeting on Quality Assurance for Nuclear Spectrometry Techniques, 12 – 16 October 2009, IAEA Headquarters, Vienna and IAEA Laboratories, Seibersdorf, Austria

Background

Chemical measurements are increasingly applied as the basis of important decisions which are required for setting up national and international regulations in such fields as trade, law, medicine, environmental pollution monitoring etc. The commercial value of traded products depends critically on the measurements which

determine the degree of quality. This requires trust in the measurements on the international level that can be achieved through introduction of uniform criteria. An area where regulation is mandatory is environmental pollution monitoring. Release of chemical compounds into the soil, the water and the air, and consequently into the human body, requires setting limits and verification of compliance with these limits through chemical measurements. Again, since pollution is often a transboundary issue (chemical pathways in the environment cross the man-made borders), decisions related to environmental pollution must be acceptable internationally.

Some laboratories established quality management system following relevant international quality standards (e.g., ISO 17025) and received accreditation from the relevant national authorities. Most efforts in this field were focused on implementation of proper Quality Assurance/ Quality Control practices in the analytical laboratories. For sake of clarity the following definition of the terms is applied:

Quality assurance includes planned activities designed to ensure that the Quality Control activities are being properly implemented,

Quality control includes the operational techniques and activities that are used to fulfil the requirements for quality.

The following nuclear spectrometry techniques are of particular interest:

- X ray spectrometry
- Gamma spectrometry
- Mössbauer spectrometry
- Raman spectroscopy
- Alpha spectrometry

A Technical Meeting is organised under the IAEA project on Nuclear Spectrometry for Analytical Applications which major objective is to enhance capability of interested Member States to effectively utilize nuclear spectrometry techniques and to provide analytical services in industry, human health, agriculture and environmental pollution monitoring.

The Technical Meeting is planned to highlight, review and discuss issues related to the current status of quality assurance (QA) in the nuclear spectrometry laboratories. It is considered beneficial to Member States to define the requirements and resources to establish QA, to demonstrate and assess the advantages of QA in promotion and improvement of competitiveness of nuclear spectrometry techniques for a wide range of applications. Proposed subjects of discussion include:

- Ingredients of quality assurance
- International quality standards
- Implementation of quality assurance
- Evaluating measurement uncertainty
- Role and availability of reference materials
- Proficiency testing and interlaboratory studies
- Validation of analytical methods
- Metrological traceability of measurement results
- Quality audits
- Significance of certification and accreditation
- Benefits/impacts of quality assurance for nuclear spectrometry laboratories
- Harmonization of approaches
- Role of the IAEA in promotion and establishing QA in nuclear spectrometry laboratories

Objectives of the Technical Meeting

To review the current status in implementation and use of quality assurance in the nuclear spectrometry techniques; to assess/demonstrate the benefits and impacts of QA on improvement of reliability of chemical measurement results in order to further extend the applicability range of the analytical techniques, and to produce a meeting report (proceedings).

Participation

A person will be eligible to participate only if nominated by the Government of an IAEA Member State (Ministry of Foreign Affairs or National Atomic Energy Authority) or by an Organisation invited to participate. The participant should be an analytical chemist (or physicist) familiar with methodology and applications of nuclear spectrometry techniques including quality assurance aspects.

Deadlines

- 31 May 2009: Submission of requests to the IAEA for participation and financial support
- 30 June 2009: Participants informed of their acceptance of participation and request for financial support.

IAEA Scientific Secretary

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Advanced School on in-situ X ray Fluorescence and Gamma ray Spectrometry, Trieste, Italy, 26 - 30 October 2009

The Abdus Salam International Centre for Theoretical Physics (ICTP) together with the International Atomic Energy Agency (IAEA) will organize an Advanced School on in-situ X ray Fluorescence and Gamma Ray Spectrometry from 26 to 30 October 2009. School directors: Drs. A. Markowicz, P. Martin, and U. Sansone (all IAEA, Vienna, Austria); Local Organiser: Dr. C. Tuniz (ICTP, Trieste)

X ray fluorescence (XRF) and gamma ray spectrometry techniques have successfully been applied in the field and in industrial environments for in-situ analysis, which covers the analysis of artefacts and materials that have not been moved from their original place of deposition/storage. Examples of applications include soil screening for metals, indoor and outdoor air pollution monitoring, screening of contaminated areas in emergency situations, investigation of cultural heritage objects (paintings, sculptures, etc), radioactive mapping of the terrestrial environment, monitoring of building materials, investigation of the radiation field in the vicinity of sunken objects, and decontamination assessment etc. A modern portable analyser based on XRF or 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.

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.

Purpose

The School will present recent advances in this area as well as the benefits of applying these techniques. It will also create an opportunity for scientists from developing countries to initiate collaboration with more advanced laboratories on use of portable XRF and gamma ray spectrometry and associated in-situ analytical methodologies.

Topics

- Current status of portable instruments based on XRF and gamma ray spectrometry for in-situ measurements
- Analytical methodologies for in-situ analysis
- Advantages and limitations of XRF and gamma ray spectrometry techniques for in-situ measurements
- Selected in-situ applications
- Practical experience in in-situ measurements by using X ray fluorescence (XRF) and gamma ray spectrometry techniques
- Role of the IAEA in promotion and effective use of XRF and gamma ray spectrometry instrumentation and analytical methodologies for in-situ applications in developing Member States

Participation

The school represents a possibility for scientists and students of UN, UNESCO and IAEA Member States to refresh and up-date their knowledge and skills in X ray fluorescence (XRF) and gamma ray spectrometry techniques. Although the main purpose of the ICTP is to help researchers from developing countries, through a programme of training activities within a framework of international co-operation, scientists from developed countries are also welcome to apply. As the activity will be conducted in English, participants should have an adequate working knowledge of this language.

As a rule, travel and subsistence expenses of the participants should be borne by the home institution. Every effort should be made by candidates to secure support for their fare (or at least half-fare). However, limited funds are available for some participants, who are nationals of, and working in, a developing country, and who are not more than 45 years old. Such support is available only for those who attend the entire activity. There is no registration fee.

How to apply for participation

The application form can be accessed at the activity website http://agenda.ictp.it/smr.php?2064

Once in the website, comprehensive instructions will guide you step-by-step, on how to fill out and submit the application form.

Deadline for receiving applications: 1 July 2009

SECRETARIAT of SCHOOL

Telephone: +39-040-2240576
Telefax: +39-040-2240585
E-mail: smr2064@ictp.it ICTP
Home Page: http://www.ictp.it/

Joint ICTP/IAEA School on Novel Synchrotron Radiation Applications, ICTP, Miramare - Trieste, Italy, 16 – 20 March 2009

The ICTP/IAEA School (Director: Dr. F. Mülhauser, IAEA, Vienna; Local Organizer: Dr. N. Binggeli, ICTP, Trieste) will cover the following topics:

- Fundamentals of synchrotron radiation based techniques
- Food quality and food processing technology using small and wide angle x ray scattering, infrared and fluorescence techniques
- X ray absorption spectroscopy, high resolution inelastic x ray scattering, resonant magnetic soft x ray scattering, surface diffraction, and micro-focused fluorescence analysis for speciation in environmental and soil sciences
- Analysis of airborne particulates with SR techniques.

For more information please visit:
http://cdsagenda5.ictp.trieste.it/full_display.php?smr=0&ida=a08154

6th European Winter School (NESY 2009) on Research with Neutron and Synchrotron Radiation, Sportheim Planneralm (Styria), Austria, 9 – 13 March, 2009

Neutron and synchrotron radiation have become essential tools in many fields of research, including materials science, biological and biomedical sciences etc. The Winter School will offer a possibility for students and young scientists from other European countries to participate and get in contact with the Austrian students and researchers. The objective is to establish the communication and links with the international scientists working at the facilities and relevant research fields. Students and scientists from the disciplines of physics, earth sciences, chemistry, and engineering should become familiar with the fascinating possibilities of the various techniques offered by world-class facilities in Europe.

For further details please visit web site
http://www.ibn.oeaw.ac.at/nesy2009/.

X ray fluorescence in Member States

During the last months we have received contributions from Argentina, Greece, Philippines and Spain on the current XRF activities. Below there are communications based in the original submissions (with only minor editorial changes).

Argentina

X ray Fluorescence Group at Comisión Nacional de Energía Atómica, Gerencia Química
Contributor: Cristina Vázquez (vazquez@cnea.gov.ar)

Synchrotron Radiation Total Reflection X Ray Fluorescence Study of Gums

Susana Boeykens [1], Néstor Caracciolo[1], Graciela Custo [3], Carlos Pérez [2], Cristina Vázquez [1,3]

(1) University of Buenos Aires. Paseo Colón 850. Buenos Aires, Argentina
(2) X ray Fluorescence and Absorption Group. Brazilian Synchrotron Light Source. Brazil
(3) Atomic Energy Commission. Av. Gral. Paz 1499. B1650KNA. San Martín. Argentina.

Over the years, the term gums has been used for a wide range of compounds including polysaccharides, terpenes, proteins, and synthetic polymers. In the 1990s, the term more specifically denotes a group of industrially useful polysaccharides or their derivatives that hydrate in hot or cold water to form viscous solutions, dispersions, or gels. Gums are used in industry because their aqueous solutions or dispersions have suspending and stabilizing properties. In addition, gums may produce gels or act as emulsifiers, adhesives, flocculants, binders, film formers, lubricants, or friction reducers, depending on the shape and chemical nature of the particular gum. They have increasingly been used in recent years by industry due to their controlled, reproducible and economical biosynthesis, and their biodegradability.

Gums are classified as natural or modified. Natural gums include seaweed extracts, plant exudates, gums

from seed or root, and gums obtained by microbial fermentation. Modified (semi-synthetic) gums include cellulose and starch derivatives and certain synthetic gums such as low methoxyl pectin, propylene glycol alginate, and carboxymethyl and hydroxypropyl guar gum. Selected polymers from the different groups were characterised in this work. Specifications of these polymers have to be controlled by European Community, Mercosur, etc. especially for toxic metals in food and pharmaceutical products.

Synchrotron Radiation (SR) induced Total Reflection X Ray Fluorescence (SRTXRF) analysis expands the possibilities of conventional TXRF based on x ray tube excitation. In this study the SRTXRF technique was successfully applied for the quantification of F, Na, Mg, S, K, Ca, V, Cr, Mn, Fe, Ni, Cu, Zn, As and Pb in high-viscosity gum aqueous solutions. The results were analysed from both toxic and alimentary point of view.

Characterization of Black Vulcanites coming from Rincón Chico 2 Site, Neuquén, Argentina, by using X Ray Fluorescence Spectrometry

Oscar Palacios[1], René Van Grieken [2], Katleen Van Meel[2], Cristina Vázquez[3,4]

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[3] *University of Buenos Aires. Paseo Colón 850. Buenos Aires, Argentina*
[4] *Atomic Energy Commission. Av. Gral. Paz 1499. B1650KNA. San Martín. Argentina*

The investigation of prehistoric hunter-gatherers archaeological sites in the Limay river basin (provinces of Neuquén and Río Negro) raised different questions concerning the lithic technology. In Rincón Chico 2, a site located on the margin of the Limay river in the Neuquén province and nowadays submerged by the Piedra del Águila reservoir (see Fig.1), many tools and debitage made in black volcanic rock has been found. The identification of this raw material can give information about possible sources of provenance. The rocks were classified macroscopically either as basalt (with coarse grains) or dacite (with fine grains) (see Fig.2).

Fig. 1. Map of the site (credit: C. Vazquez)

However, in several cases, a dual behaviour was observed in the fracture zone: the texture was coarse on the surface and very fine, sometimes almost vitreous, in the interior. Following this observation, it was decided to introduce a new more general category called ‘black vulcanites’. Looking for the origin and more precise identification of this raw material, artifactual samples and a set of black vulcanite fragments coming from

Paso Limay, a dacite source located 50 km from the archaeological site, were characterized by x ray fluorescence spectrometry using a Philips Minipal energy dispersive system. Samples were analysed as loose powder. Statistical analysis reveals greater variability between artefact samples than between the samples from the source itself. This discrepancy is more evident if Fe, Ti and Zn are used as markers. These results suggests that the quarry at Paso Limay was not the main source of provenance of the black vulcanites utilized by the hunter-gatherers occupying Rincón Chico.

Fig. 2. Black vulcanite samples from Rincón Chico archaeological sit. (credit: C. Vazquez).

Identification of inorganic components and lipids in a rock art sample from Alero Hornillos 2 (Jujuy, Argentina) by XRF, FT-IR and GC-MS

Cristina Vázquez[1,2], Marta Maier[3], Sara D. Parera[3], Hugo Yacobaccio[4], Patricia Solá[4]

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[4]University of Buenos Aires, Institute of Archaeology, Buenos Aires, Argentina

Archaeological samples are very complex in terms of composition since they generally consist of a mixture of materials exposed to deterioration factors that are largely dependent on the environmental conditions. Therefore, the integration of analytical tools such as TXRF, FT-IR and GC-MS can maximize the amount of information provided by the sample.

Recently, two rock art samples of black camel figures at a Hornillos 2 cave, an archaeological site located 20 km from the town of Susques, Jujuy Province, Argentina at 4020 m above sea level were investigated. The purpose of the present work is to analyze the inorganic and organic components of the samples in order to improve understanding of the artistic practices and technological knowledge of the early inhabitants of this region.

Considering the small amount of sample available for the study, TXRF technique was selected for characterization of inorganic components. Analysis by TXRF showed the presence of manganese among others elements, consistent with manganese dioxide for the black pigment. Aiming at the detection of any residual organic compounds, the sample was extracted with a chloroform/methanol (2:1) mixture and the extract was analysed by FT-IR, showing the presence of bands at 2970-2870, 1729 and 1450-1380 cm^{-1}, attributable to lipids. Analysis by GC and GC-MS of the carboxylic acid methyl esters prepared from the sample extract, indicated that the main organic constituents were saturated ($C_{15:0}$, $C_{16:0}$, $C_{17:0}$ and $C_{18:0}$) and unsaturated ($C_{16:1}$ and $C_{18:1}$) fatty acids, together with hydrocarbons of 22, 23, 24 and 25 carbons and one sterol. This composition suggests the use of lipids of vegetable origin as binders. This study shows the advantage of the combination of analytical tools to generate full information on the cultural heritage objects.

Getting an insight into soft polymeric systems properties by X Ray techniques

Susana Boeykens[1] , Cristina Vázquez[1,2]

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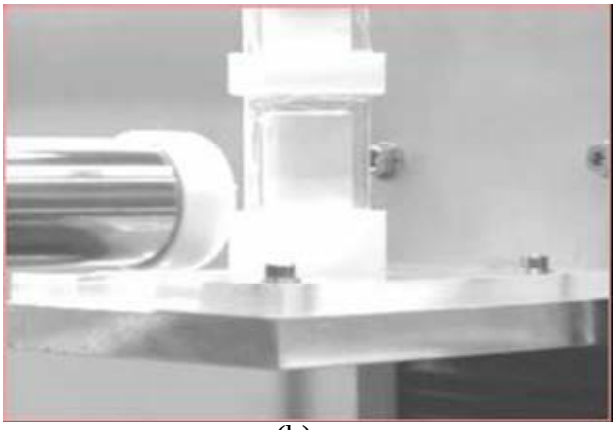
[2] Atomic Energy Comisión, Av. Gral. Paz 1499, B1650KNA, San Martín, Argentina

Soft polymeric systems cover the range from quasi-homogenous dispersions, in which the number, the position and the size of pores are fluctuating, to porous heterogeneous rigid structures. Theoretical and experimental investigation of transport phenomena of dispersed systems of different nature is recently of particular importance. This is because this knowledge is essential for a variety of technologies applied in diverse areas such as preparation of emulsions, gel permeation chromatography, separation and filtration operations, enhanced oil recovery, medicinal, food and cosmetic production, decontamination procedures, etc. Concerning decontamination properties, soft polymeric appeared to be efficient systems for trapping metals from contaminated media and therefore are good candidates for metal cleaning applications. In order to design technological devices for such purposes it is necessary to have a good understanding of the transport properties of the diffusing species in a given matrix (polymer-solvent) which are influenced by changes in structural characteristics (see experimental set-up on Fig. 3).

(a)



(b)

Fig. 3 Acrylic cell arrangement in the SRμXRF beam line (a) and details of the setup (b) (credit: C. Vazquez)

Greece

Ion micro-beam analysis and XRF related activities at the Institute of Nuclear Physics, NCSR 'Demokritos', Greece (2007-2008)

Institute of Nuclear Physics, NCSR 'Demokritos', 153 10 Aghia Paraskevi, Athens, Greece

Contributors: A.G. Karydas (Karydas@inp.demokritos.gr), D. Sokaras and V. Kantarelou

The activities of the Institute of Nuclear Physics in the field of Ion-Beam and XRF analytical techniques during 2007-2008 included research and development projects together with training and dissemination activities, in particular concerning the implementation of nuclear analytical techniques in the field of cultural heritage.

a) Development of the 3D-MicroPIXE technique

The use of a polycapillary half lens in a typical Micro-PIXE set-up renders the conventional Micro-PIXE technique into a new tool for depth resolved and in general 3D elemental analysis. The information depth can range even up to 40-50 µm, overcoming in some cases the analytical capabilities of standard ion beam techniques for elemental depth profiling. Two confocal micro-PIXE set-ups have been developed and evaluated up to now with the contribution of the INP-NCSR "Demokritos", Technical University of Berlin, Jožef Stefan Institute and AGLAE accelerator groups [1, 2]. The first in vacuum confocal micro-PIXE set-up was applied for depth resolved analysis of layered materials (Fig. 1) at the Micro-analytical Center of the Jožef Stefan Institute, Ljubljana, Slovenia [1] and for the determination of element specific local concentrations and of their respective positions with a micron spatial resolution in a PM10 aerosol filter [3]. A confocal Micro-PIXE set-up under atmospheric pressure was developed and applied at the external micro-proton beam facility of the AGLAE particle accelerator at the Centre de Recherche et de Restoration de Museés de France, Paris, France [2]. This project was co-financed by the EU-ARTECH program.

A simulation algorithm for PIXE intensities in confocal geometry was recently developed for a quantitative description of 3D MicroPIXE intensities acquired in both the beam and sample scanning modes [4]. This model has been applied to provide concentration profiles in stratified materials such as a glazed ancient ceramic sample [1] and an artificially corroded copper alloy [2].

The implementation of the confocal geometry at an ion-microprobe beam line has certain advantages with respect to 3D Micro-XRF set-ups. For 3D Micro-PIXE only one X ray lens in front of the detector is required taking advantage of the excellent intrinsic spatial resolution of the ion microprobe. Also, the beam scanning possibilities play an important role not only for the relative ease and fast alignment of the confocal set-up, but also for deducing faster and more precise depth intensity profiles. A decrease of the proton ionization cross sections with increasing depth, in contrast to the fluorescence cross sections which are depth independent, is certainly a disadvantage and a limiting factor as far as the range of depth analysis is concerned, however, it may improve the spatial resolution of the 3D MicroPIXE method. Higher proton energies (>=3 MeV) and relative low atomic number matrices (organic, aluminum-silicate) increase the penetration depth of protons to more than 100 µm, thus, improving the information depth for elements emitting characteristic X rays with energies more than about 5 keV.

In general, 3D Micro-PIXE may be used for the investigation and determination of the structure and composition of layer systems and of elemental depth concentration gradients. Furthermore, it is suitable to select individual micrometer size objects which are within a probing depth beneath the surface and provide specific elemental compositional analysis. With its non-destructive and depth-resolving properties, 3D Micro-PIXE seems especially suited for investigations in the field of environmental science, biology and cultural heritage.

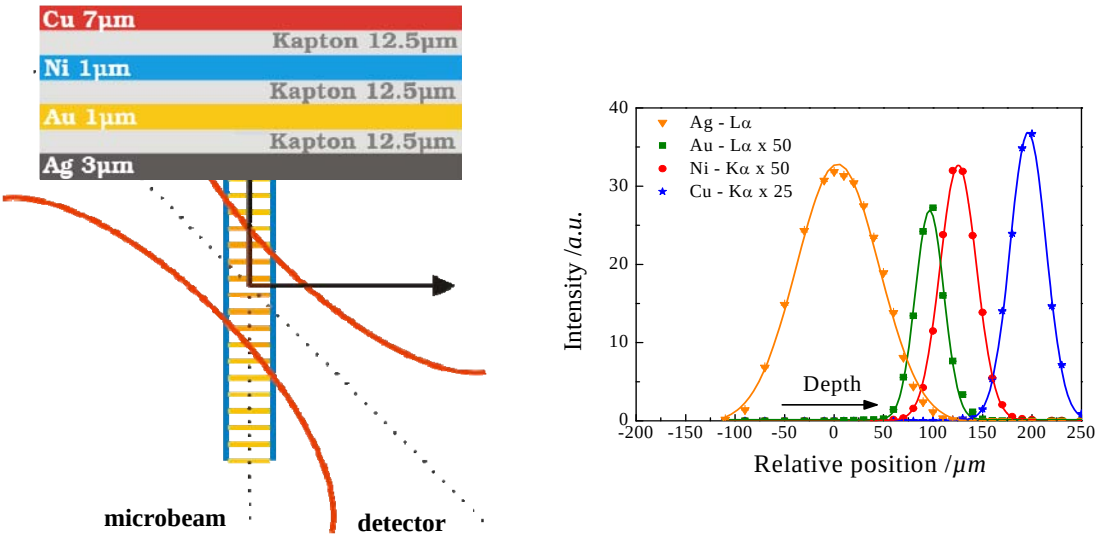


Fig. 1. In-depth intensity profiles of a multilayer sample (left figure) with the following structure obtained by means of the 3D Micro-PIXE analysis. The solid lines represent corresponding Gaussian fits [1]

b) Development of an external Ion-Beam set-up at the Tandem accelerator of the Institute of Nuclear Physics

At the Tandem *VdG* 5.5MV TN accelerator of the Institute of Nuclear Physics of NCSR 'Demokritos', Athens, an external ion-beam set-up has recently been installed. The aim of this development was to establish a complete experimental set-up integrating the analytical capabilities of the PIXE, RBS and PIGE techniques, so that a complete elemental and near surface structural characterization of samples/artifacts to be attained in an almost non-destructive way and without any limitation concerning their size or conductive state. A careful 3D mechanical drawing optimized the experimental parameters of the set-up so that the special requirements imposed for optimum performance of the aforementioned techniques are fulfilled. The first applications were focused in the quality control of tagged materials (technologically authentic replicas of attic ceramics and in coatings used by conservators for paintings).

The external ion-beam setup utilizes simultaneously five detectors, two for the implementation of PIXE, one for RBS, one for PIGE, and one X ray detector for the dose monitoring (Fig. 2).

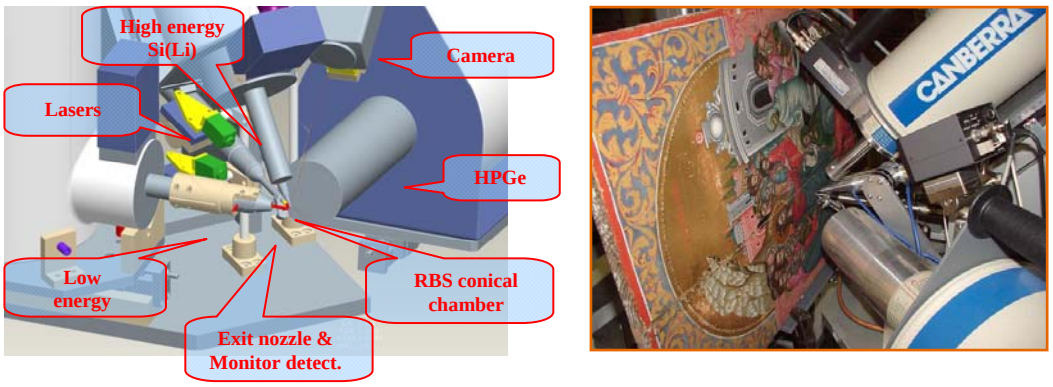


Fig. 2. Schematic drawing of the external ion-beam set-up (left). The photo at right shows the analysis of a painting from the Valadoros Collection, 18th cent., belonging to the Benaki Museum, Athens, Greece.

The exit ion-beam nozzle provides collimation and minimizes the beam dimensions to a diameter less than about 1 mm. The beam exit window was selected to be a 100 nm Si_3N_4 window that produces minimum energy loss and straggling of the beam, whereas the small air path between the window and sample position also improves beam energy resolution. The geometrical arrangement of the detectors was optimally designed and constructed in order to allow: 1) A simultaneous utilization of two X ray detectors for PIXE measurements, the one ('low-energy') being in He atmosphere and the other one ('high energy') with 'hard' filtering in order to achieve combined detection of major, minor and trace elements emitting X rays with energy from 1 to 30 keV. 2) A surface barrier (SB) detector placed in vacuum inside a special constructed conical vacuum chamber for the RBS mode that enables near surface characterization of layered structured materials or of the surface depletion or enrichment of particular elements. The silicon SBD detector has a thickness of 300 μm and it is placed in at an angle of about 160^o with respect to the beam axis, whereas it is possible to approach a few mm away from the analysis point. In order to minimize the energy loss of the detected backscattered particles, 100 nm of Si_3N_4 was used as chamber's entrance window aiming to offer the best possible resolution for the RBS spectra. 3) Detection of prompt γ rays induced by nuclear reaction on the isotopes of the low atomic number elements that compose the sample (Li, B, F, Na, Mg, Al, Si), 4) Precise dose normalization by means of the Si-K signal emanating from the exit Si_3N_4 window, and 5) Visual inspection of the analysis area and tools for easy and reproducible alignment of the sample/artifact with respect to the reference position at the particle beam direction. Visualization is achieved by means of a CCD camera and two lasers whereas for proper handling the sample/artifact is mounted on an x-y translation stage.

For the data acquisition, novel hardware and software tools were used to perform digital pulse processing. In particular, two Xilinx XtremeDSP Development kits for Virtex-4 SX cards were used as a complete platform for development of an on-chip digital pulse processor for X ray, γ ray and particle detectors. Each card accepts two analog signals from prefilter and performs digitization, shaping and recording using custom FPGA configuration.

A typical example indicating the analytical performance of the PIXE analytical mode is indicated in the analysis of a glass certified reference material (BAM) by 3 MeV protons (Fig. 3). The blue line indicates the PIXE spectra taken from the low energy PIXE detector, while the red curve is the PIXE spectra from the high energy PIXE detector. The first runs were very promising towards the synergistic use of PIXE, RBS and PIGE techniques, confirming a rather good precision of the measurements and integrated analytical capabilities.

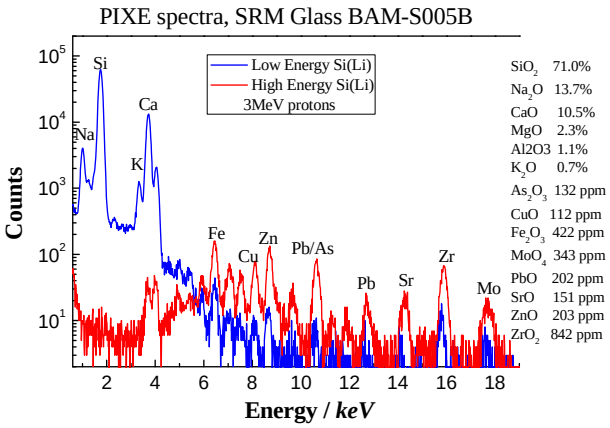


Fig. 3. Typical PIXE spectra of a SRM Glass acquired at the external ion-beam set-up using the 'low-energy' and 'high energy' X ray detectors, respectively.

c) Development of an integrated PIXE-XRF technique

A multi-purpose scattering chamber was developed and constructed at the Demokritos Tandem *VdG* 5.5MV TN accelerator aiming to address fundamental and applied research topics in the filed of X ray spectrometry. The novel set-up combines effectively particle and photon interactions with matter targeting to: Sub-keV X ray fluorescence spectrometry, measurement of X ray Resonant Raman Scattering (RRS) cross sections, study of the PIXE continuum polarization properties and to the successful treatment of interference problems that usually appear in conventional XRF and PIXE techniques. The functionality of the PIXE induced XRF chamber is based on the effective ionization of a primary target by a heavy ion beam (1-3 MeV/amu) and thus the production of an X ray source with good specifications (intensity, monochromaticity) for further X ray fluorescence studies.

The experimental setup (Fig. 4) includes the following components: a) A two level chamber (low level: primary targets, upper level: samples) b) Two rotatable feedthroughs for positioning six primary targets and six samples. The primary target feedthrough is electrical isolated and liquid cooled. c) A filter wheel with 8 positions for absorbing completely the backscattered ions and enabling appropriate filtering of the exciting X ray beam. All excitation/detection angles are set at 45 degrees. Additional instrumentation components include an oil-free vacuum pump (upper level) and an X ray UTW Si(Li) detector (Gresham/e2v Sirius®).

The dose monitor is achieved through charge integration or/and by means of an X ray detector that records the primary particle induced X ray radiation.

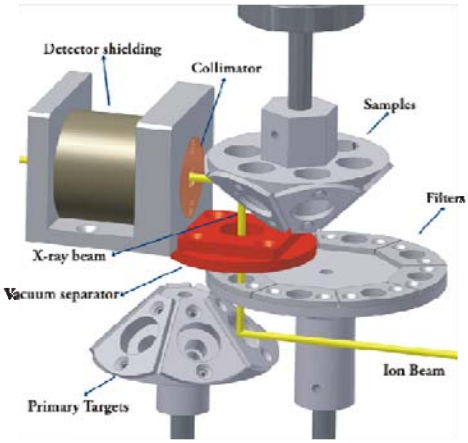


Fig. 4. Schematic drawing of the PIXE-XRF scattering chamber

A sequence of spectra from the reference material SL-1 are presented (Fig. 5) as they measured with the PIXE induced XRF chamber using various experimental conditions (primary target, proton beam energy and filter). Suitable selection of multiple energy exciting X ray beams provide obvious advances towards accuracy in the quantification and improved minimum detection limits (for example, Na: 150ppm in the case of SL-1 for a measuring time of about 10 minutes with 1 μA beam current).

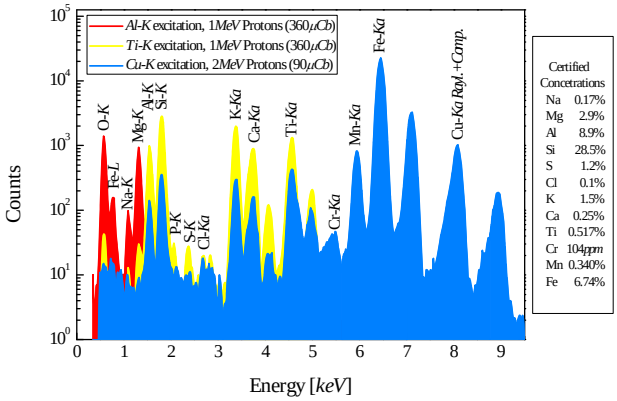


Fig. 5. XRF spectra of the IAEA standard reference material SL-1, measured by means of three different proton induced X ray beams (Al-K, Ti-K and Cu-K excitations, respectively)

The first experimental results obtained showed optimum analytical performance (analytical range, sensitivity) in the low and sub-keV energy region, a critical development in comparison with the conventional tube excited XRF and PIXE modes. The perspectives for further applications are numerous and significant towards: Systematic measurement of RRS cross sections, polarization measurements of the PIXE continuum, the improvement of the detection limits for certain trace elements in metallic matrices (e.g., Pt in Au), evaluation of secondary processes in XRF spectrometry and for applying and assessing reference-free quantification using unpolarized excitation.

d) Development and validation of a quantification methodology for a portable micro-XRF spectrometer

In micro-XRF analysis a polycapillary X ray lens is usually utilized in the excitation channel to collect very efficiently, propagate and focus down to few tens of micrometers the exciting X ray beam radiation. On the other hand, the X ray lens introduces major difficulties in quantification, since its transmission efficiency highly depends on the transmitted X ray energy which results in a significant and not easily predicted modification of the energy distribution of the primary spectrum from the x ray tube. Furthermore, in particular for metal alloys, the quantification involves many practical problems such as presence of the interfering diffraction peaks and the heterogeneity of the alloys at the micro-scale.

A methodology that provides an analytical description of the lens transmission efficiency was developed and validated in the case of a set of standard reference glasses. Furthermore, for the case of metal alloys an experimental strategy was suggested to minimize the interference of diffraction peaks and the heterogeneity of the alloys [5]. The methodology for the determination of the lens transmission efficiency implied at first that the parameter can be expressed as a polynomial of 5th degree versus energy. Next, measured and theoretically predicted characteristic K- or L- line intensities emitted by a large set of pure single element or compound targets (1.5 - 25 keV), with “infinite” or infinitely thin thickness, were compared through a X^2 minimization procedure with the fitted parameters. For the measurements, a micro-XRF set-up consisting of an Rh microfocus X ray tube operated at 50 kV, a polycapillary X ray lens and a Si-drift X ray detector was used. The agreement between theoretical and experimental pure elemental thin/thick target intensities at the convergence minimum was equal or better than 10%. The developed quantification model was validated in reproducing elemental concentrations of ten NIST and BAM standard reference glasses. The observed deviations from the corresponding certified values were in general less than 10%-15%.

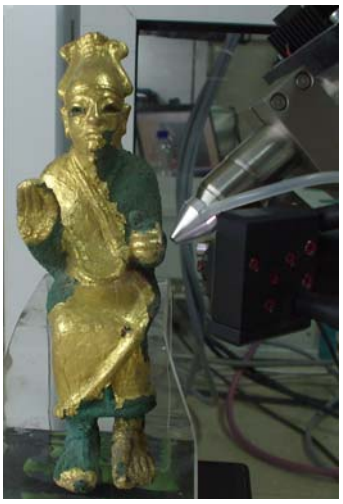
The proposed analytical approach has the advantage that it does not require any removal of the X ray lens from the μ-XRF setup configuration and it can be adapted in commercial micro-XRF spectrometers supporting a FPA methodology towards quantification in micro-XRF analysis.

e) The PROMET analytical campaign in the Syrian Arab Republic and Jordan, Oct/Nov 2007

The morning of 26 October 2007 marked the beginning of a two-week analytical campaign of EU FP6 PROMET (PROtection of METals, www.promet.org.gr) to the Syrian Arab Republic and Jordan. Scientists from the Institute of Nuclear Physics at NCSR Demokritos and the Institute of Electronic Structure and Laser (IESL) - FORTH at Herakleion, Crete left Athens to land to Damascus, where they spent a week working with colleagues at the National Museum of Damascus before continuing the campaign to Yarmouk University in the city of Irbid, Jordan.

The aim of the campaign was to demonstrate the use and potential of two new mobile instruments, in analytical investigations of archaeological and historical metal objects in collaboration with PROMET colleagues in the Syrian Arab Republic and Jordan. The Demokritos group, Andreas Karydas, Vicky Kantarelou and Makis Bistekos, carried to Damascus their state-of-the-art micro-XRF spectrometer, while the IESL-FORTH group, Annie Giakoumaki and Demetrios Anglos, brought their portable LIBS instrument.

During the first week the measurements were carried out in the conservation laboratory at the basement of the National Museum in Damascus. Luda Mahfoud and Abeer Kurdab, Mayada El Saadi and Kasem Yahya from the Damascus Archaeological Musuem, Prof. Ahmad Almansour and Prof. Jameel Alshehne from the Aleppo University, the Syrian Arab Republic, presented a number of really unique objects (mostly gold and gilded copper alloys) from the impressive metals collection of the museum. These objects were examined for the first time by the XRF and LIBS spectrometers with the aim to characterize raw materials, in order to improve the knowledge about the manufacture technology and to identify surface corrosion products. The measurements were attended by Dr. E.H. Bakraji, D. Halloum, H. Eassa and M. Al Waze from the Syrian Atomic Energy Commission.



Eail God, Gilded copper alloy, Late Bronze Age 1400-1300 B.C., Museum No. 3573, Archaeological museum of Damascus, the Syrian Arab Republic, October 2008, Micro-XRF analysis of gold foil composition, thickness, identification of surface corrosion products, © PROMET

On 28 October a special event was organized by the Directorate General of Antiquities and Museums of the Ministry of Culture, which is a partner in the PROMET consortium. Maher Azar, advisor of the Minister of Culture and engaged in PROMET, offered a welcome address. The activities of the PROMET project and the aims of the campaign were presented by the project's coordinator Prof. Vasilike Argyropoulos, Greece, and Prof. Ahmad Almansour, representing the Syrian partners, followed by a discussion session with conservators, archaeologists and historians.

In the second week people and instruments traveled to Irbid, Jordan, for the second part of the campaign and settled at the Numismatic Museum of the Yarmouk University. In collaboration with colleagues from the Department of Conservation of the Faculty of Archaeology and Anthropology, Manar Bani Hani, Lina Khrees, Prof. Mohammed S. Shunnaq, Prof. Zaidon Al Muhasin and Prof. Ziad Al-Saad, and the curator of the Umm Qais Museum, Mohammad Bashabsheh, the LIBS and Micro XRF groups focused on the analysis of a series of gold and copper coins among the unique collection of the museum. This collection spans a broad time period from the Roman, Byzantine, Arabic and Ottoman times.

Furthermore, in collaboration with Abeer Arafat from the Royal Scientific Society in Jordan several copper objects from the Umm Qais site were examined by both techniques in order to characterize their bulk composition, to identify surface corrosion products and thus to assess their preservation state. In addition, together with Manar Bani Hani and Abeer Arafat naturally weathered coins and coupons treated with different coatings were also examined in order to evaluate their protection performance and in some cases their reversibility. The university environment provided a nice opportunity to the groups from Greece to interact with students and teaching staff at the Department. Following two special lectures by A. Karydas and D. Anglos, students came by and had a chance to see and use the analytical instruments.

Lion, Headed Eagle lapis lazuli and gold, 3000 B.C. Early Bronze Age, Archaeological museum of Damascus, the Syrian Arab Republic, October 2008, Micro-XRF analysis of the golden head and tail, compositional analysis of the lapis-lazuli stone, © PROMET

Andreas Karydas (left, INP-NCSR) and Demetrios Anglos (right, IESL-FORTH) presenting the principle of microXRF and LIBS operation respectively, at the Department of Conservation of the Faculty of Archaeology and Anthropology at Yarmouk university, Irbid, Jordan, during the PROMET 2007 campaign.

f) Dissemination activities

During October 2008, the INP-NCSR Demokritos in collaboration with the Department of Applied Research of the Directorate for Conservation of Ancient and Modern Monuments, Ministry of Culture, Greece, organized in the framework of the IAEA regional TC project RER/1/006 two events: A regional workshop entitled "*Harmonization Procedures related to Nuclear Analytical Techniques for characterization and preservation of cultural heritage artifacts*" (13-17 October 2007) and a national workshop (20-22 October 2007) with the aim to promote the use of modern, non-destructive analytical techniques (XRF, Ion-Beam Analysis, LIBS, μ-Raman, FTIR, etc) in the field of conservation science. About 100 professional conservators and researchers in the conservation science attended the three days workshop with great interest.

Acknowledgements

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Spain

Main activities of the X ray Laboratory at the Institute of Earth Sciences 'Jaume Almera', CSIC, Barcelona

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Introduction

The Laboratory of X ray Analytical Applications (LARX) in the Institute of Earth Sciences "Jaume Almera" (ICTJA) belongs to the Spanish Council for Scientific Research (Spanish acronym CSIC), the major Scientific Research Organization in Spain. Since its creation, the ICTJA has actively been engaged in X ray experimental activities mainly using X ray diffraction for crystallography studies. The LARX was established in 1982 and since then has been continuously equipped with instrumentation for both X ray diffraction (XRD) and X ray fluorescence (XRF) spectrometry. During 1982-1990, the laboratory work was mainly focussed on the study of crystal structures, including single-crystal X ray diffraction analysis or "ab initio" resolution from powder X ray diffraction spectral data. WDXRF instrumentation was used to provide complementary geochemical data generated under the research projects of the ICTJA as well as for a precise quantification of light elements and their molecular state in geological samples.

From the beginning of the 1990's the LARX started several activities and research projects on environmental issues including studies of the dispersal of heavy metals in soils and sediments combining the results from X ray diffraction and X ray fluorescence techniques. The XRD instrumentation was replaced by a new Siemens D-5005, thus enabling improvements in quantification of mineralogical composition of atmospheric particulates. From 1995 - 2002, the laboratory was equipped with a portable EDXRF system using three radioisotope sources (Fe-55, Cd-109 and Am-241) and a Si(Li) detector, which was used for field analysis or in-situ characterization of mineral pigments in paintings ("frescoes") of Romanesque Age.

During the last eight years, the WDXRF instrumentation has been renewed (Bruker S4, Rh target tube, five analyzing crystals, three collimators and two detectors). A new EDXRF spectrometer with primary beam focusing capability was purchased to perform analysis of small regions, determination of layer thickness and elemental mapping (XDAL, Helmut Fischer GmbH including W target tube and motorized XYZ stage; spatial resolution of 0.1-2 mm).

Research& development activities

From the beginning of LARX, the laboratory has participated in collaborative projects together with

other Spanish CSIC Institutes, Museums and Universities, funded by the National Science Programme or by the European Framework Programmes. During the last decade the LARX collaborative links with scientists from European X ray laboratories (CFAUL, Lisbon, Portugal; INETI, Lisbon, Portugal, MITAC, Antwerp, Belgium) or from South America X ray laboratories (CENA, Sao Paulo, Brazil; CEPROCOR, Cordoba, Argentina) have been strengthened. Since 1997 several specialized courses and seminars on X ray analysis have been organized. The main research activities are focussed on a study of dispersal of metallic pollutants at different environmental compartments (water, air, soils and biota) and their intake by the vegetation from abandoned old metal-mining areas. Some of these research projects are:

A. Determination of metal contaminants in vegetation by means of XRF techniques

Determination of chemical composition of vegetation materials in environmental and industrial field has become an urgent and important need. In this context, the development and application of X ray fluorescence methodologies and quantification strategies for the determination of major, minor and trace elements has become one of the research topics in the last years. The XRF has successfully been applied as a multi-elemental technique for a rapid quality control of bulk raw medicinal plant materials [*1*] and environmental pollution monitoring to perform simultaneous analysis of vegetation species collected in polluted mining areas surrounding abandoned waste landfills. In the later case, diverse configurations of XRF spectrometers using different excitation sources (EDXRF [*2-4*], WDXRF [*5*] and HE-P-EDXRF [*6*]) have been tested to assess the analytical performance in specific applications. Data obtained confirmed the benefit of using polarized x-rays sources (primary beam scattered by a secondary target) to reduce a high scattering of the primary photon beam by vegetation samples which resulted in an improvement of the detection limits, especially for heavier elements (low $mg \cdot kg^{-1}$ range), as compared to conventional instrumentation. Recent experimental work using bench-top focusing EDXRF instrumentation concentrates on study of the elemental distribution and mapping of contaminants (see Fig. 1) in different parts of vegetation (roots, leaves, branches, stems) growing on the wastes of the abandoned Pb-Zn mines.

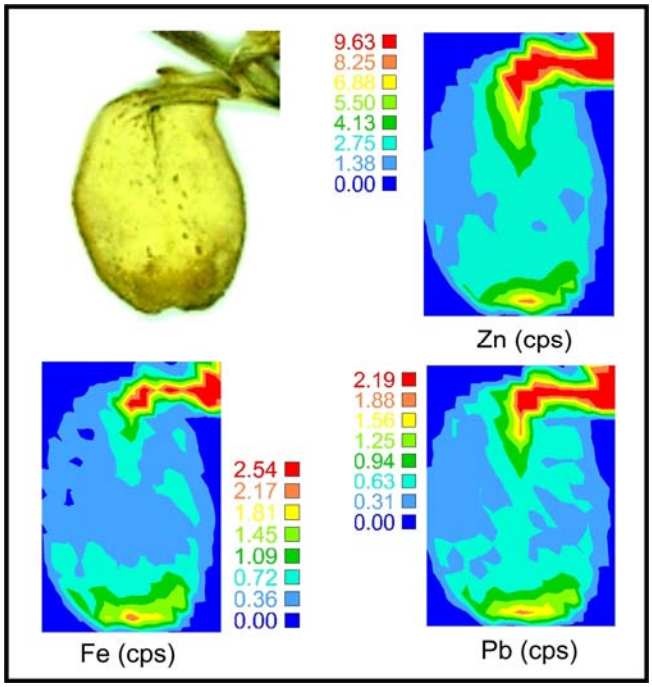

Fig. 1. Elemental mapping (Zn, Pb, Fe) of leaves from plants growing on mining waste residues (Osor Mine, Girona province, Spain)

B. Characterization of solid residues from mining activities and metal scrapping industry

The usefulness of EDXRF for a fast characterization of residues from abandoned mines or active metal industry was demonstrated [7, 8]. Likewise the combined use of XRF and lixiviation tests was a promising tool for the pre-evaluation of potential environmental risk of this kind of residue samples [7].

On the other hand, the small-spot EDXRF instrumentation at LARX has been applied for the determination of heavy metals in soil cores and the complementary mapping (see Fig. 2) provide interesting data for the assessment of metal mobility along the unsaturated soil zone of metal polluted areas [9, 10].

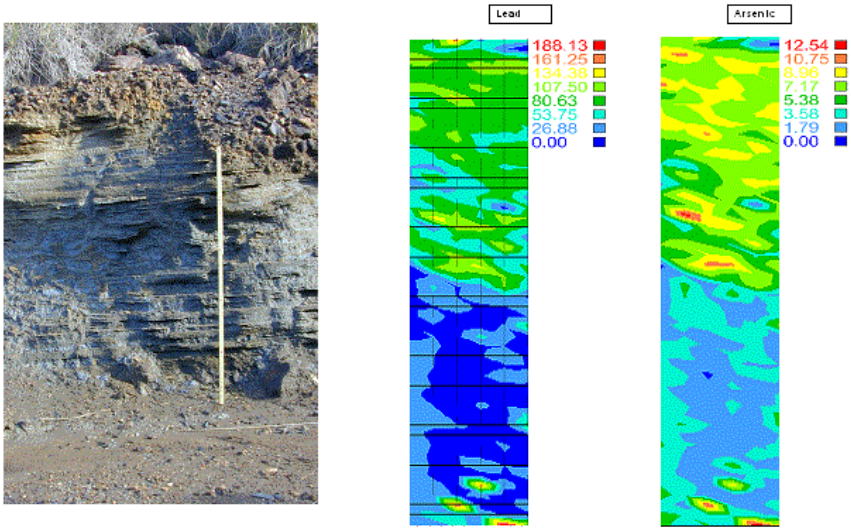

Fig 2. Elemental mapping (lead and arsenic) in soil cores of overbank sediments of a polluted creek (Cartagena-LaUnión mining district, Murcia province, Spain). Vertical scale is 50 cm.

C. Determination of hexavalent chromium and total cadmium in aqueous solutions by means of preconcentration and XRF analysis

Direct analysis of complex industrial and environmental liquid samples presents some difficulties due to the matrix-effects, and a dilution of the sample or preliminary separation/pre-concentration step may be required prior to the atomic spectroscopy analysis. A promising alternative is a combination of pre-concentration and X ray fluorescence (XRF) analysis. In particular, the feasibility of the combined use of a simple and inexpensive activated thin layer pre-concentration procedure with different configurations of XRF spectrometers (EDXRF, WDXRF, HE-P-EDXRF) for the determination of trace amounts of some metallic pollutants in complex environmental liquid samples has been one of the recent research fields of the LARX.

The proposed analytical methodology is based on the metal preconcentration using activated thin layers containing the commercial anion-exchanger tricaprylylmethylammonium chloride (Aliquat 336). This is a particularly convenient method of separation for the XRF technique because the activated thin layer can be mounted directly in the spectrometer and thus offering a novel and easy alternative to the complicated pre-concentration methods applied for such matrices. Moreover, the use of activated thin layers eliminates a need for correction of the matrix and thickness effects. Since the attenuation of the primary and secondary radiation is almost negligible in this case, the XRF signal is proportional to the metal concentration. The detection limits for the metals under investigation are in the μgL^{-1} range. This methodology has been successfully applied for the determination of Cr(VI) in industrial waters [11], a direct determination of Cd in sea water samples [12, 13] and precious metals (Pd, Pt) in synthetic liquid samples.

D. Application of combined XRD and XRF for the study of materials used in cultural heritage

Simple lime wash or lime painting are the most common exterior surface coating of old buildings, that are part of our urban cultural heritage. The color was usually obtained by adding to the lime, red and yellow natural minerals (yellow and red ochres -almagres and terra rossa, black earths and treated mineral pigments; red, yellow, black iron oxide pigments and synthetic ultramarine blue). The traditional colors were, and still are, the yellow and red, the black or grey and the blue. The old lime paintings made of mineral pigments exhibit properties of transparency, depth and softness of color. Today, their aesthetic value is again recognized, and the efficiency and quality of the chromatic studies in the conservation and rehabilitation of architectural heritage relies on the understanding and retrieving of these traditional materials. The combined use of WDXRF and XRD spectrometry is a useful methodology for the characterization and precise identification of such pigments [14, 15].

Within the framework of a join project between LARX and the National Museum of Fine Arts of Catalonia (MNAC, Barcelona) a portable EDXRF (XAN, Helmut Fischer GmbH) has recently been used for study and analysis of Greek, Roman and Middle Age coinage (see Fig. 3) from archaeological excavations in Catalonia [16, 17].

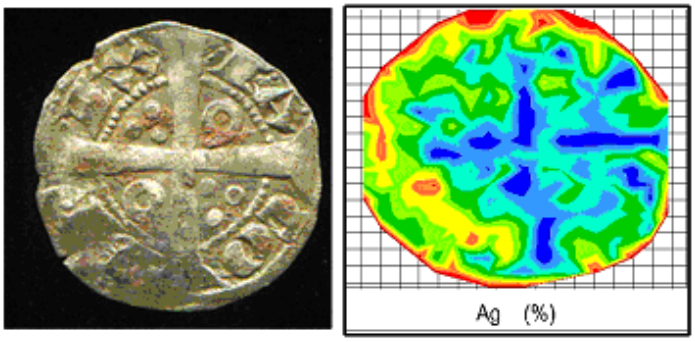

Fig 3. Distribution of silver on surface of a coin of Middle Age (13th century, from National Art Museum of Catalonia, MNAC, Barcelona, Spain)

Other activities

The X ray instruments are also used to provide relevant data for basic geological work carried out by other scientific groups at the ICTJA. Geochemical and mineralogical assessment based on XRF and XRD data allows characterization of geological materials in order to determine the origin and sources of rocks, correct interpretation of geological processes [*18, 19*], and supports mineral exploration [*20*].

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Philippines

XRF activities at Analytical Measurements Research Group, Philippine Nuclear Research Institute

Analytical Measurements Research Group, Atomic Research Division, Philippine Nuclear Research Institute

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XRF analysis and activities of the Analytical Measurements Research (AMR) Group (see Fig.1) of the Philippine Nuclear Research Institute (PNRI) focus on both research and analytical services. Air pollution research, in particular source apportionment studies, requires multi-elemental data for a substantial number of samples (see Figs. 2 and 3).

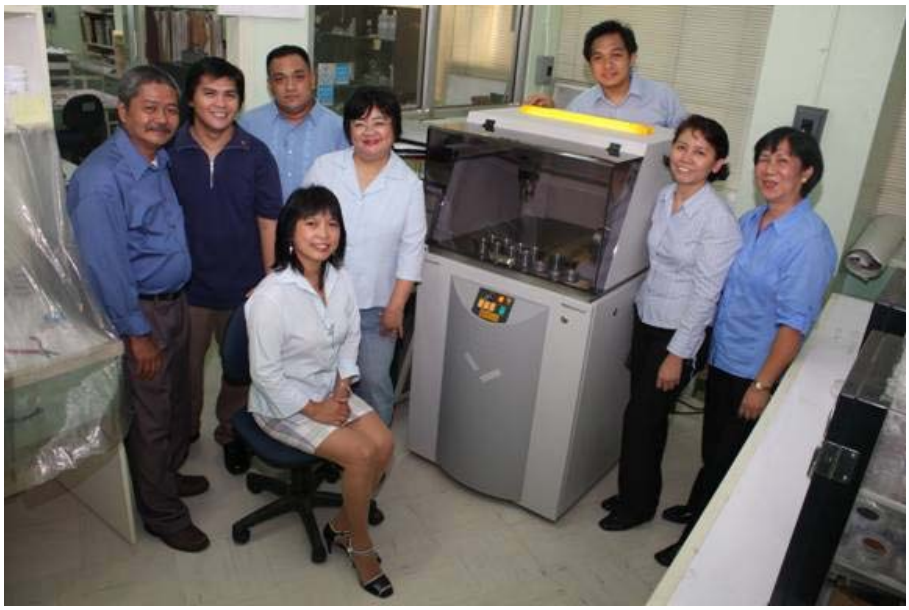

Fig. 1. Analytical Measurements Research Group, Atomic Research Division, Philippine Nuclear Research Institute (credit: Alan Borras)

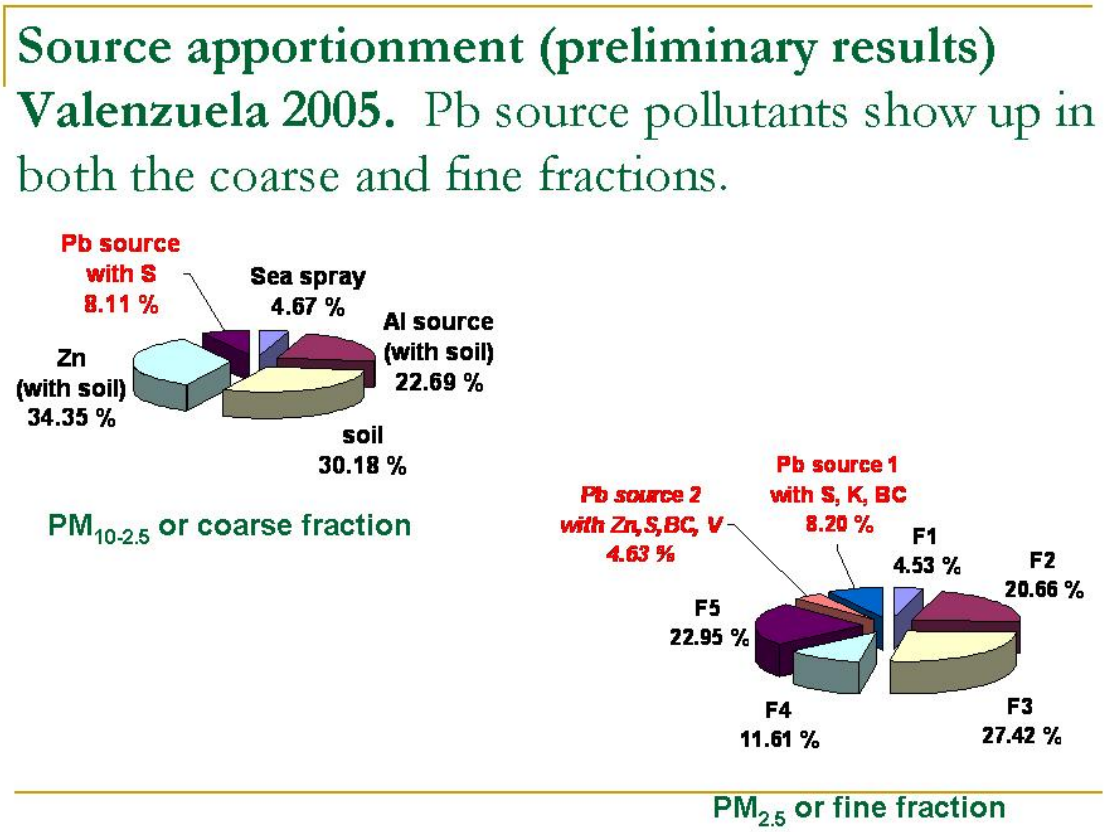

Fig. 2. Preliminary results for source apportionment (credit: Alan Borras)

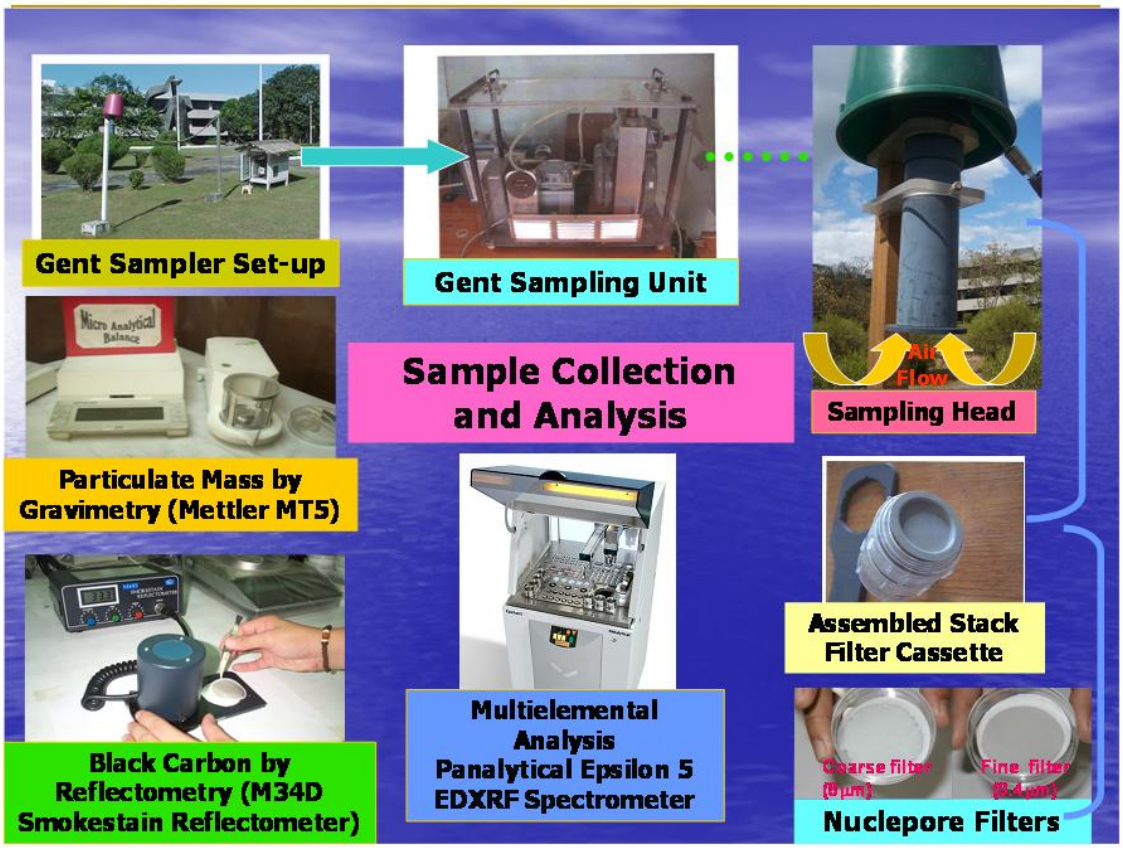

Fig. 3. Sample collection and analysis for air particulate matter (credit: Alan Borras)

In the PNRI, energy-dispersive X ray fluorescence (EDXRF) has been used as an effective tool for providing such multi-elemental data. With the latest acquisition of the Panalytical Epsilon 5 (E5) EDXRF system, the process of quantification has become easier and faster with the auto-quantify method. Other research involvements of the group are in the analysis of samples in relation to mineral explorations and the elemental characterization of water in support for isotope hydrology research.

The AMR group, as part of its function to provide analytical services, offers qualitative or semi-quantitative analysis of solid samples using the auto-quantify method, quantitative analysis of environmental samples using the emission-transmission method and quantitative analysis of air particulate matter collected on filters. Telephone wire materials sold in junkshops (alleged to have been pilfered from installed telephone lines of a major telecommunications company in the country) and materials being assessed in relation to patent claims are other examples of samples submitted for analytical services.

As mentioned, a useful feature of the E5 system is the use of the auto-quantify (AQ) method. Calibration lines used for this type of application are obtained using the fundamental parameter (FP) model. For AQ applications, accurate results are obtained for samples prepared as fused glass beads in which the whole matrix is known. However, only qualitative or semi-quantitative analysis can be applied for other types of solid samples. The AQ method was adapted for the multi-elemental analysis of air particulates using the MicroMatter standards to set up the AQ calibration and using an assigned compound for making up to 100% composition.

A new application of E5 system was analysis of water. In this case 30 µL of water samples (pre-concentrated) and multi-element standards were dried on a membrane filter and samples were analyzed by using the conventional calibration curves; for majority of the elements a good linear response for the standards was obtained.

Publications of potential interest to the XRF community

Portable X ray Fluorescence Spectrometry: Capabilities for *in situ* Analysis, P.J. Potts and M. West, Eds., The Royal Society of Chemistry, Cambridge, UK, 2008

Atomic and Nuclear Analytical Methods. XRF, Mössbauer, XPS, NAA and Ion-beam Spectroscopic Techniques, H.R. Verma, Springer, 2007

D. Wegrzynek, R. Mroczka, A. Markowicz, E. Chinea-Cano and S. Bamford, Experimental evaluation of X ray optics applied for microanalysis, X ray Spectrom. 37, 635-641, 2008

Analytical Instrumentation: A Guide to Laboratory, Portable and Miniaturized Instruments, G. McMahon, Wiley-Interscience, 2008

P. K. Hopke et al., Urban air quality in the Asian region, Science of the Total Environment, 404, 103-112, 2008

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