



## X ray Fluorescence in the IAEA and its Member States

## Activities in the IAEA XRF Laboratory

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

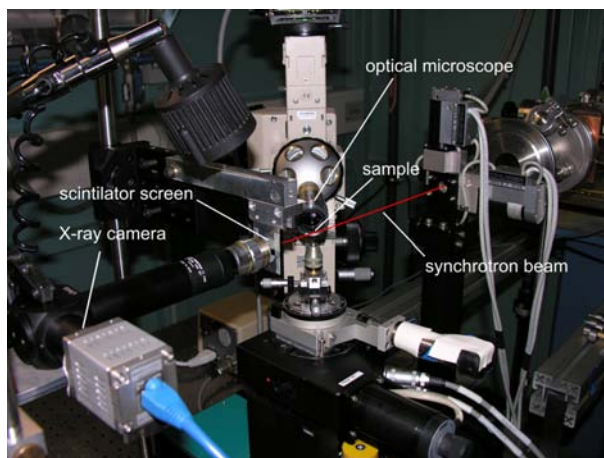
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## Application of X ray imaging techniques to the study of the morphology of malaria mosquitoes

### *Introduction*

X ray phase contrast tomography [1] was applied to examine the morphology of malaria transmitting mosquitoes. This work was carried out in support of a project on development of the sterile insect technique (SIT) for controlling, in combination with other methods, the population of such mosquitoes [2]. The technology is based on the idea of releasing an irradiated, and therefore sterile, population of male mosquitoes to the environment of a given territory. Female mosquitoes mate only once in a lifetime. Therefore if mating occurs with irradiated (sterile) male, the eggs will not hatch, breaking the reproduction chain. The successful implementation of SIT requires that the irradiated population of male mosquitoes is otherwise healthy in order to compete with the non-treated males during the mating process. This requirement implies that the treated insects should not undergo morphological changes due to the irradiation.



*Fig. 1*  
*X ray phase-contrast*  
*imaging set up utilizing a*  
*CCD camera and thin film*  
*scintillator*

## Experimental

The morphology of treated and untreated malaria transmitting mosquitoes (*Anopheles arabiensis* Patton, Diptera: Culicidae) was investigated with the use of X ray phase contrast tomography technique. Two groups of mosquitoes were selected for measurements: irradiated mosquitoes, which had received a dose of 120 Gy from a  $^{60}\text{Co}$  gamma source, and a non-irradiated control population. Pupae and adult populations were studied. The mosquitoes were chemically fixed, their abdomens were sectioned out from the insect body, mounted on graphite rods and scanned in a phase contrast X ray tomographic setup. The measurements were performed at the Fluo-Topo beamline. A monochromatic X ray beam (12 keV) from a multilayer monochromator was used to obtain phase-contrast enhanced projections of the analyzed samples. The projections were collected with X ray film and with a thermally stabilized CCD camera. The experimental set up utilizing CCD camera is shown in Fig. 1. The camera was focused on a thin layer scintillator plate (cerium doped yttrium aluminium garnet – YAG:Ce). Each tomographic scan consisted of 180 projections, each projection was 16-bit grey level image composed of  $1392 \times 1024$  pixels. The 3D morphology of the analyzed samples was reconstructed by applying the filtered backprojection algorithm and by processing the resulting data with specialized volumetric reconstruction software.

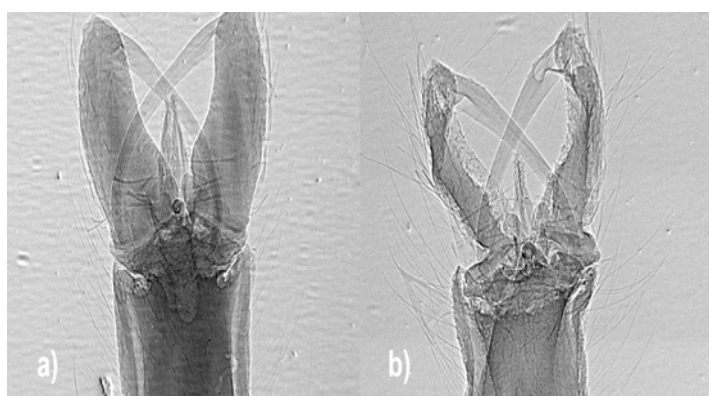


Fig. 2. X ray phase-contrast enhanced projections of the abdomens of irradiated mosquito pupae (a) and adult specimen (b). The collected tomographic scans consisted of 160 such individual projections.

In Fig. 2 two individual projections of the abdomens of irradiated mosquito pupae (a) and adult specimens (b) are shown. The pictures shown in Fig. 2 are the processed images corrected for non-uniform illumination of the object, artefacts

and hot-spots due to camera defects, and for the decay of the primary beam. The corrections were applied to the stack of 180 images collected during the tomographic scan. The corrected stack of 180 images was transformed into a set of 1024 sinograms. Each sinogram contained information required to reconstruct a cross-sectional view of the object at certain position (corresponding to a certain row in the projection image).

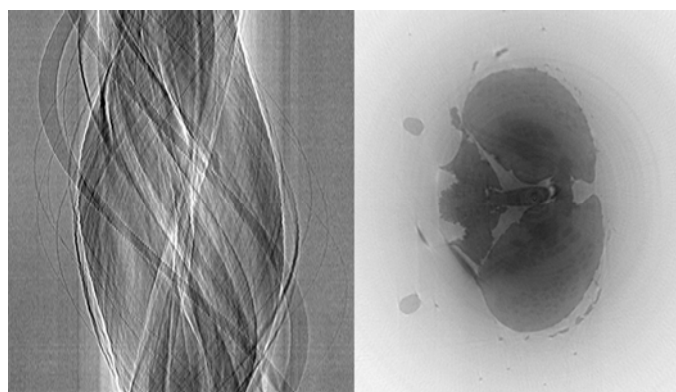


Fig. 3. Left: sinogram corresponding to the 512th row of the projection image shown in Fig. 2a; right: reconstructed cross-sectional view of the sample.

In Fig. 3 a sinogram corresponding to the 512th row of the projection image presented in Fig. 2a is shown. The sinograms were processed by a filtered backprojection algorithm assuming a “pencil beam” measuring geometry. A stack of 1024 reconstructed cross-sections of the object was aligned with the aid of volumetric imaging software to obtain a 3D view of the specimen morphology. The volumetric image of the sample is shown in Fig. 4. Due to low signal to noise ratio in the acquired images the segmentation of the structures based on grey level values could not be performed. However, the quality of the reconstructed images was good enough to visualize differences in the analyzed mosquito specimens.

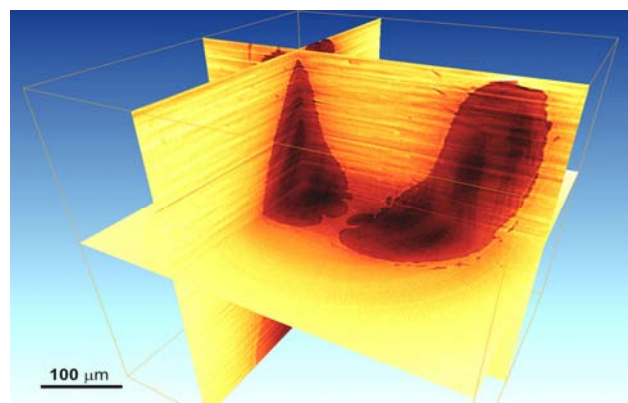


Fig. 4. Cross-sectional view through the volumetric reconstructed image stack.

## Conclusions

The collected data demonstrated the usefulness of X ray phase-contrast tomography for investigating morphological changes in the irradiated mosquito specimens. In the next experiment a cooled CCD camera with specially designed optics will be used. The data acquired with a cooled CCD camera will have a much better signal to noise ratio as compared to the data collected during the first experiment with the thermally stabilized CCD. In combination with phase retrieval algorithms, e.g. [3], this should allow for image segmentation and visualization of the individual morphological details of mosquito specimens.

## References

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## Preparation of insect specimens for micro-XRF analysis

### General considerations

Preparation of insect specimens for  $\mu$ -XRF shares many steps in common with the classic protocols for TEM or SEM. Therefore, the preparation involves the fixation of the tissue, dehydration<sup>(\*)</sup> and infiltration with a medium that can be hardened to give a material suitable for the analysis. A major goal of preparation is to allow a study of the specimen in a form as close as possible to the natural state, in other words, to stabilize and preserve the fine structural details of the organs in a condition similar to that of living tissues. Any artifacts (morphological changes, loss of information or introduction of non-existing structures) should be avoided and the specimen should be stable during examination by using  $\mu$ -XRF.

### Fixation

Fixation is the first step in preparation of tissue and must be accomplished as soon as possible so that postmortem changes are kept at a minimum. Fixation is carried out by treating the specimen with a carefully selected mixture of chemicals under strictly controlled and reproducible conditions.

The features of ideal fixative for  $\mu$ -XRF are as follows:

- prevents tissue autodigestion (autolysis)
- inhibits bacterial and fungal biological activity (preserves the tissue)
- makes the specimen resistant to damage during subsequent processing
- is isotonic with the tissue and thus does not alter the organ's volume
- does not distort any part of the tissue structure
- does not dissolve tissue components
- is not detrimental to the organs under study.

Fixatives are divided into two major groups according to the nature of their reactions with the proteins in the tissue, namely coagulant (ethanol, mercuric chloride, etc.) and non-coagulant cross-linking (formaldehyde, paraformaldehyde, glutaraldehyde).

The main factors affecting the fixation process are:

- The pH of the composition. Chemical fixation is a complex set of oxidative and reductive reactions, where the  $[H^+]$  is constantly changing. A locally excessive acid or alkaline medium may adversely affect the sample. On the other side, at a specific pH, all proteins have a so-called isoelectric point (IEP). Fixation is most effective at the IEP. All fixatives, therefore, have an optimal working pH and are normally applied in a buffered medium. The fixative composition should be chosen in such a way that the optimal working pH is very close to the pH of the cellular medium. Special care should be taken when selecting the buffer solution, particularly in relation to its compatibility with the chemicals to be used in the post fixation processes. For example, phosphate buffer precipitates uranyl acetate, a commonly employed stain.
- Total ionic strength of reagents, osmolarity or tonicity (fixative composition and concentration). Specimens for  $\mu$ -XRF should be fixed under near isotonic conditions. Hypertonic fixatives tend to shrink the organs while hypotonic fixatives are likely to produce swollen specimens.
- Method of application; for  $\mu$ -XRF immersion should be the application method of choice.
- Temperature; this affects the kinetics of fixation. As a rule a slow reaction speed is desirable, thus fixation is carried out at temperatures between 4°C and room temperature.
- Length of fixation; this is one of the parameters to be established empirically. Both under-fixed and over-fixed specimens show typically recognizable artifacts. The whole fixation process is performed in two steps: primary fixation that may last from 30 min to 24 hours, and secondary fixation, typically 1 to 2 hours.

### **Dehydration**

(\*) The term dehydration should not be confused with drying. To dehydrate a tissue in this context means to replace the aqueous intra- and extracellular medium by a non-water containing one. On the other hand, to dry the sample means to remove the liquid from the tissue.

The main aim of the dehydration is to condition the specimen for embedding, that is, to replace the cellular medium by one compatible with the resin

composition to be used for embedding. For  $\mu$ -XRF the specimens are not embedded but only infiltrated with the plasticization mixture, therefore the excess of it should be easily removable from the exterior of the sample. In this way, resins soluble or partially soluble in water or ethanol are the most appropriate ones. For this reason, the monomers of the resin composition are used for dehydrating the samples. Normally an auxiliary infiltration agent is used to speed up the penetration of the monomers. An additional advantage of using water-soluble resins is that a tertiary fixation with uranyl acetate or phosphotungstic acid, that act also as an additional contrasting agent, can be performed during the dehydration stage.

### **Embedding**

Embedding is the process of infiltrating the tissue with a composition that can be subsequently hardened to provide mechanical and environmental stability to the specimen. In the case of  $\mu$ -XRF, resin compositions that polymerize on the air at temperatures below 60°C are preferred. Special care should be taken to follow the recommended proportions on the resin formulation, to prevent the expansion or shrinkage of the polymerized product, as well as on the curing conditions.

### **Preparation of mosquitoes for $\mu$ -XRF**

Preparing whole mosquitoes for  $\mu$ -XRF present several challenges:

- Due to their natural habitat conditions, the animals are hydrophobic. They tend to float on the surface of the aqueous solutions.
- The exoskeleton presents a formidable barrier for the diffusion of solutions into the body of the animal.
- Most of the internal organs are extremely fragile.

To overcome those difficulties, besides the conventional components, to the primary fixation solution two additional substances were added:

Extran neutral; a mixture of anionic and cationic surfactants with strong complexing characteristics. With this addition the air is not trapped inside the external structures of the mosquitoes, which allows the soaking of their carcass.

Dimethylsulfoxide; a generic molecular transporting assistant. DSO easily diffuses across cellular wall caring with it the dissolved molecules of the fixative.

### ***Complete recipe for infiltration with QUETOL 651-NSA***

**NOTE: Most of the chemicals used in this protocol are toxic and some very toxic. Careful attention to the safety data sheets is essential. Perform the operations as much as possible under the extraction hood.**

1. Stun the mosquitoes in CO<sub>2</sub>
2. Place the whole insects in the prefixing solution for 24 hours (primary fixation):
  - a. 2,5% of Glutaraldehyde
  - b. 5% of Sucrose
  - c. 1% of Dimethylsulfoxide
  - d. 0,1% Extran neutral
  - e. 0,1% CaCl<sub>2</sub>
  - f. 0,1 M Cacodylate Buffer
3. Dissect the mosquitoes as pertinent in Cacodylate Buffer.
4. Place the selected organs or parts in fresh prefixing solution for 2 hours
5. Wash in Cacodylate Buffer 5 times for 15 min each.
6. Fix the specimens in 2% Osmium tetroxide 0,1 M Cacodylate Buffer for 2 hours in darkness.
7. Wash in Cacodylate Buffer 2 times for 10 min each.
8. Wash in water 5 times for 15 min each.
9. Dehydrate in graded QUETOL 651 gently shaking (wobbling) the trays:
  - a. 1<sup>st</sup> tray: 25% QUETOL 75% of 2% water solution of uranyl acetate for 30 minutes
  - b. 2<sup>nd</sup> tray: 50% QUETOL 50% of 2% water solution of uranyl acetate for 30 minutes
  - c. 3<sup>rd</sup> tray: 75 % QUETOL 25% of 2% water solution of uranyl acetate for 45 minutes
  - d. 4<sup>th</sup> tray: 90% QUETOL 10% of 2% water solution of uranyl acetate for 45 minutes
  - e. 5<sup>th</sup> tray: 100% QUETOL for 60 minutes.
  - f. 6<sup>th</sup> tray: 100% QUETOL for 90 minutes.
10. Infiltrate with n-Butyl glycidyl ether (n-BGE)
  - a. 1<sup>st</sup> tray: QUETOL 50%, n-BGE 50% for 1 hour.
  - b. 2<sup>nd</sup> tray: n-BGE for 3 hour.
  - c. 3<sup>rd</sup> tray: n-BGE 50% , QUETOL mixture 50 % for 8 hours in darkness:
 

|                                |          |
|--------------------------------|----------|
| i. QUETOL 651                  | 3,5 mL   |
| ii. Nonenyl Succinic Anhydride | 5,4 mL   |
| iii. Nadic Methyl Anhydride    | 1,1 mL   |
| iv. DMP-30                     | 0,175 mL |
  - d. QUETOL mixture for 24 hours in darkness.
11. Place the container under moderate vacuum for 2 hours to degas the samples.
12. Carefully extract the specimens and place them in a soft filter paper. Using small bits of the same paper remove the excess of QUETOL mixture.
13. Place the specimens in a new filter paper and using a micropipette drop some alcohol on each, in order to wash away any remaining QUETOL mixture on the outer surface.
14. Place the specimens in a Teflon capsule and cure in an oven at 50°C for 72 hours.
15. Glue the cured samples on graphite rods for  $\mu$ -XRF analysis.

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## Coordinated Research Project on “Unification of nuclear spectrometries: integrated techniques as a new tool for material research”

During the last years the IAEA supported a substantial number of laboratories in Member States in establishing nuclear spectrometry facilities for various applications. These laboratories are being used in support of applied research, teaching and education in nuclear science and technology as well as for providing analytical services for industry, environmental pollution monitoring, study of cultural heritage, food and agriculture, human health etc. In most cases the nuclear analytical techniques such as X ray fluorescence (XRF), particle induced X ray emission (PIXE), particle induced gamma emission (PIGE), Rutherford backscattering (RBS), etc. are used independently and provide information on a specific characteristic of the analysed materials. In order to characterise the materials in a comprehensive way or to improve the quality of the analytical services provided by the laboratories, a combined use of a few techniques is often required. In recent years advanced laboratories, such as Synchrotron Sources, started to perform measurements by use of the integrated instruments and simultaneous processing of data where outputs of the analytical techniques are used as inputs to a common data processing procedure. In this way a quality of characterisation of various materials by using multiple analytical techniques improves considerably. In order to coordinate the research efforts of the laboratories towards better integration and unification of nuclear spectrometries a new CRP has been initiated under the project D.3.03. “Improvements in nuclear spectroscopy applications”. The CRP should contribute to meet the objectives of the project, i.e. to enhance proper utilisation of nuclear analytical methods in Member States and to help them develop nuclear instruments for special applications.

One of the major areas of integration is application of synchrotron radiation sources for X ray microfluorescence, microdiffraction, X ray microscopy, microtomography, and absorption techniques used for elemental analysis, 2D and 3D imaging and chemical speciation. It is expected that portable XRF spectrometers will be used with other techniques for integrated applications and better utilisation of the equipment. Monte Carlo –

type simulation programmes are also implemented to design integrated nuclear spectroscopy systems to optimise the measurement geometries, detection limits, and spatial resolution. A combination of XRF and PIXE setups and integration of data processing procedure might be used to extend the applicability range of the X ray emission techniques for a better characterisation of the materials. In summary, integration and unification of nuclear spectrometries can be observed at the level of instrumentation including hardware and software components, measurements and data collection, data processing and data interpretation. The results of the CRP will be distributed to all countries having interest in integration and unification of nuclear analytical techniques for comprehensive characterisation of materials in support of new developments and applications.

### ***Overall objectives:***

The CRP will help Member States to improve characterisation of materials by the utilization of nuclear spectrometry instruments and techniques as well as by developing integrated/unified instruments and analytical methodologies. These techniques can be used in small laboratories as well as in state-of-the-art synchrotron sources. It is expected that through the CRP the nuclear spectrometries will be more widely utilised and new special applications implemented in support of environmental pollution monitoring, industry, study of cultural heritage, human health, agriculture. etc.

### ***Specific Research Objectives:***

The specific research objectives may include the following:

- Development (or upgrading) of integrated multifunctional instruments based on nuclear spectrometries and related techniques. Combinations of the various techniques, given in Table I, can lead to new impacts for material analysis, either in their composition, structure, thickness or in the impurities.
- Development of software for handling and operation of integrated multi-functional instruments including data acquisition.

- Development (or upgrading) of integrated analytical approaches/methodologies and software for processing and presentation of data collected by multifunctional instruments.
- Development of new applications of integrated/unified instruments to assist end-users of nuclear spectrometries in various fields.
- Development of synergistic and complementary use of nuclear spectrometries.

### **Expected Research Outputs:**

- The expected outputs of the CRP may include the following:
- Integrated and multifunctional instruments with technical documentation.
- Software packages for handling and operation of multifunctional instruments.
- Integrated software packages for data processing and presentation.

- Improved analytical methodologies based on combined use of various nuclear spectrometries.
- New applications of nuclear spectrometries for better characterisation of materials.
- Research papers, reports, conference contributions and IAEA-TECDOC to be published after completion of the CRP.

End-users and beneficiaries of the developments under the CRP will be researchers and professionals involved in characterisation of materials in the following fields: industry, environmental pollution monitoring, agriculture, human health, study of cultural heritage artefacts etc. The outputs of the CRP will also support the R&D activities in the above mentioned fields.

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## X ray fluorescence in Member States

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

### Argentina

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#### **1. Metal ions diffusion through polymeric matrix: A TXRF study**

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This work proposes the use of X ray fluorescence with total reflection geometry to explore the metal ions transport in aqueous hydrophilic polymer solutions. It is centered in the study of the influence of polymer concentration on ions diffusion. This subject is relevant to various and diverse applications, such as drug controlled release, microbiologic corrosion protection and enhanced oil recovery. It is anticipated that diffusion is influenced by various factors in these systems, including those specific to the diffusing

species, such as charge, shape, molecular size, and those related to the structural complexity of the matrix as well as any specific interaction between the diffusing species and the matrix.

The diffusion of nitrate salts of Ba and Mn (increasing hydrodynamic radii) through water-swollen polymeric solutions and gels in the 0,01 to 1% concentration range, was investigated. The measurements of the metal concentration were performed by TXRF analysis using the scattered

radiation by the sample as internal standard. Results are discussed according to different

physical models for solute diffusion in polymer solutions.

## 2. Total reflection by synchrotron radiation: Trace determination in hydrogels

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Polysaccharides biological growth requires carbohydrates as source of C and micronutrients namely N, F, Na, Mg, P, S, etc, coming from inorganic salts. The presence of these impurities could alter physical properties as adsorption processes in biological tissues. Quantitative trace determination of these low Z elements is rather difficult by using conventional total reflection X-ray fluorescence (TXRF) instrumentation. In this sense, total reflection by synchrotron radiation (SRTXRF) is a promising alternative to be employed for this purpose.

In this work, SRTXRF was investigated in order to

prove its effectiveness for determining impurities in high viscosity polymer solutions. Polymeric solutions of scleroglucan, guar gum, galactomanan, glucomanan, metilcellulose, gelatin and starch were analyzed. Sample quantification was performed with an appropriate internal standard and by a simple calibration procedure with a multielemental standard solution. Measurements were performed in the X ray Fluorescence Line at the National Synchrotron Light Laboratory (LNLS), Campinas, Brazil. After this study, SRTXRF seems to be a powerful tool for the determination of light element traces.

## 3. Trace mercury determination by trapping complexation: application in advanced oxidation technologies

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The extensive use of Hg compounds in agricultural applications as part of pesticides, herbicides, insecticides and bactericides determines its frequent presence in industrial wastewater. It is well known that Hg species possess high noxious effects on the health of living organisms even at very low levels (5 µg/L). Nowadays, quantification of this element is an analytical challenge due to the peculiar physicochemical properties of all Hg species. The regulation of the maximal allowable Hg concentration led to the search of sensitive methods for its determination.

Total Reflection X ray Fluorescence (TXRF) is a proved instrumental analytical tool for the determination of trace elements. In this work, use

of TXRF for Hg determination is investigated. Because of volatilization of several mercury forms, a procedure to capture these volatile species in liquid samples by using different complexing agents is proposed. Ammonium pyrrolidinedithiocarbamate (APDC), ethylenediaminetetraacetic acid (EDTA) and oxalic acid were assayed for trapping the analyte into the solution during the preparation of the sample and onto the reflector during TXRF measurements.

The proposed method was applied to evaluate Hg concentration during TiO<sub>2</sub>-heterogeneous photocatalysis, HP, one of the most known Advanced Oxidation Technologies (AOTs). AOTs are processes for the treatment of effluents in



waters and air that involve the generation of very active oxidative and reductive species. In HP, Hg is transformed to several species under UV illumination in the presence of titanium dioxide.

TXRF is a valuable tool to follow the extent of the HP reaction, by determining non-transformed Hg in the remaining solution.

## Croatia

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### Ten years of cooperation with IAEA Laboratories Seibersdorf – IAEA beam line at the Rudjer Bošković Institute in Zagreb, Croatia

According to the Agreement between the International Atomic Energy Agency and the Rudjer Bošković Institute that has been signed in 1996, IAEA has installed on its own cost a beam line attached to the Tandem Van de Graaff accelerator at the Rudjer Bošković Institute (RBI) in Zagreb Croatia, with possibility to use it up to 30 working days per year. Since that time, numerous activities have been performed in most cases by joint teams of Laboratory for ion beam interactions from Zagreb and staff of XRF Laboratory from Seibersdorf.

The beam line consists of a vacuum chamber (Figure 1) equipped with Si(Li) detector for PIXE spectroscopy positioned at 135 degree port, as well as with particle detector for RBS positioned in the chamber at the 165 degree scattering angle. Intrinsic Ge detector can be positioned for PIGE measurements at the other 135 degree port that is normally used for observing the sample by camera. The beam line is attached to the -15 degree port of the 6.0 MV Tandem. Recently in 2005, a new 1.0 MV Tandetron accelerator has been installed, enabling use of IAEA beam line with any of two existing accelerators (Figure 2).

Since 1996, more than 1000 spectra were recorded and many subjects have been studied. Among these the most frequent studies were of reference and candidate reference materials made by IAEA as well as by other producers. Homogeneity tests performed by PIXE included tests at microscopic level using RBI nuclear microprobe. Several joint intercomparison tests including two with NIST showed excellent capabilities of the beamline PIXE quantification procedure. This has been

achieved by subsequent control of analytical procedure and careful calibration of the system as well as the software being used.



Figure 1. IAEA beam line

Test spectra obtained with IAEA PIXE setup were used also for intercomparison of existing PIXE quantification software. As PIXE is excellent technique for analysis of air particulate matter, this subject has been tackled several times and still is of major importance. In addition to the studies of backing filters for air particulate matter sampling, that should be suitable for all X ray techniques (including XRF) we also studied recently supports that may be used for air pollution monitoring using TXRF.

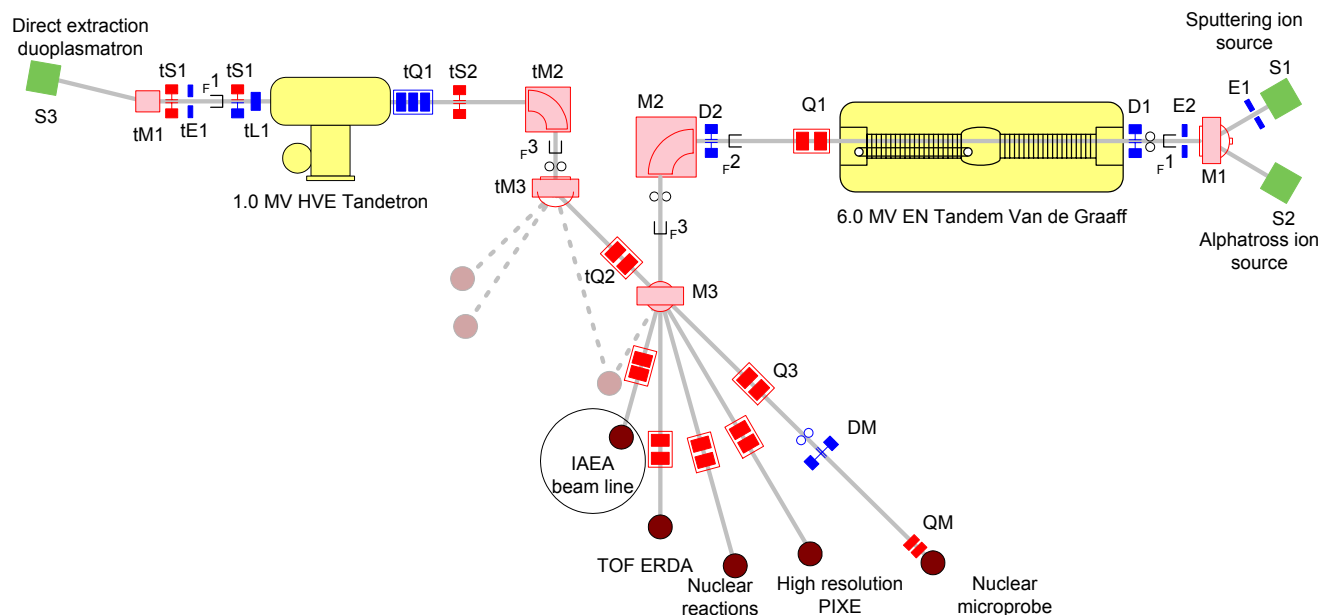


Figure 2. Layout of the Rudjer Bošković Institute Laboratory for ion beam interactions with two accelerators and IAEA beam line.

Several run sessions were dedicated for PIXE studies of samples obtained from other IAEA member state laboratories. These include a series of archeometry samples of ceramics and polychrome pigments, different geological samples, as well as more recently series of timber species. RBS technique was used for analysis of multilayered thin film technological samples.

Not less important was the research connected to the development of analytical techniques. The use of RBS for the analysis of matrix composition was important for testing reliability of PIXE and XRF quantification procedure based on fundamental parameters. Improvements in capabilities of the PIXE/RBS system have been continuously made as well. In order to adapt the PIXE system for the best analytical sensitivity for the particular problem, several different setups can be used. Two different detector positions, 4 different X ray filters that are controlled from outside of chamber, secondary electron suppressor, interchangeable collimators for beams between 2 and 6 mm are possible.

In collaboration with Seibersdorf Laboratories, important improvement has been obtained in development of universal multiparameter data acquisition hardware and software. The software SPECTOR that can control different hardware for data acquisition has, in addition to independent acquisition of spectra from various detectors, possibility to control sample positioning (Figure 3), as well as microbeam scanning (for proton

microbeam at IRB) and sample stage scanning and control (for X ray microbeam at Seibersdorf). Multiparameter data acquisition for IEE and TOF ERDA systems in Zagreb has been also tested and used.

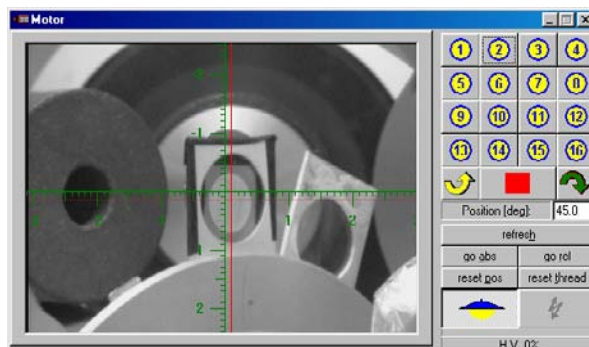


Figure 3. View of the program for sample holder positioning

As a result of many developments, an IAEA-TECDOC – Instrumentation for PIXE and RBS has been also prepared and published.

With collaboration of Seibersdorf staff, quality control procedures were started to be implemented as well. Operation, measurement and quantification standard operation procedures (SOPs) have been completed.

Importance of education through the beam line can be seen from the fact that during the last 10 years, eight trainees from developing countries spent altogether 25 months working at the IAEA beam line in Zagreb. Preparation of learning modules for ion beam analysis is in progress. Furthermore,

remote access to the 'live' PIXE and RBS experiment at beam line will be soon available.

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## Greece

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### 1. Activities in the field of X Ray Spectrometry (2004-2006)

The activities of the Institute of Nuclear Physics at NCSR "Demokritos", Greece, are related with both, basic research and applied research topics in the field of X ray spectrometry.

#### **Basic Research**

##### *a) X ray interactions with matter*

Within the frame of the Greece-Germany IKYDA project (2004-2006) a strong scientific collaboration has been established between the Institute of Nuclear Physics of the N.C.S.R "Demokritos" and the laboratory of the Physikalisch-Technische Bundesanstalt (PTB) at the electron storage ring BESSY II.

The scientific objectives of this common research project refer mainly to the determination of X ray fundamental parameters with low relative uncertainties and to quantification issues (by means of experiments or simulations), regarding the contribution of the photo-electron ionization process to the primary fluorescence intensity, when tunable synchrotron radiation irradiates thin films of low atomic number elements. Summarizing, the results of the project, up to today are the following:

- The experimental determination of the KL Resonant Raman Scattering cross-section for Ni by means of polarized and unpolarized exciting X ray radiation.
- The development of a simulation Monte Carlo code allowing the calculation of the primary fluorescence intensity enhancement for low Z elements due to their secondary ionization by photoelectrons.
- The experimental determination at various incident photon energies (3–12 keV) of the enhancement that it is produced to the fluorescence intensity of thin Mg films (0.5-3  $\mu\text{m}$ ), due to the secondary photo-electron ionization process.

##### *b) Ion-Beam interaction with matter: Polarization properties of the ion-induced X-ray continuum.*

The continuum radiation emitted after the proton bombardment of a solid or gaseous target (PIXE) consists of the main limiting factor for improved detection limits in PIXE analysis of trace elements. The study of the polarization properties of PIXE continuum was motivated from the angular distribution of the ejected SEB electrons, being preferentially emitted in the forward direction. The

degree of the polarization of the PIXE continuum was experimentally determined for the first time in the energy region 3–5 keV for a solid thick carbon target and for 2.5 and 3 MeV proton energies (2004). This work was awarded the best poster award in the PIXE2004 conference.

## 2. Research and Development of Ion Beam and X Ray Methods:

Our research and development activities are related with the implementation of Ion-Beam and X ray Fluorescence methods in various disciplines. New experimental methodologies were developed and the analytical investigations were supported by comparative experimental and theoretical studies.

**a) Chemical state analysis employing sub-natural linewidth resolution PIXE measurement of the  $K\alpha$  diagram line.** In the framework of a Greek-Slovenia bilateral collaboration (2001–2003), sulfur speciation studies were performed in an aerosol filter, by means of high resolution X ray spectrometry. Collaborating part: Jozef Stefan Institute, Ljubljana, Slovenia

**b) Theoretical and experimental study of fluorescent emission in a secondary target tube excited X ray fluorescence set-ups.** A systematic study, both experimentally and theoretically was undertaken towards a full description of secondary target X ray fluorescence set-ups. In particular, fluorescence intensities were measured or predicted for various analytically described X ray tube distributions, operational voltages and combination of anode -secondary target materials.

**c) Non-destructive analysis of Cultural Heritage materials.** During the past few years we have been actively involved in the field of non-destructive analyses of ancient materials, artifacts or artworks. Our applied research activities have been focused so far: 1) to the optimised selection, integration and evaluation of portable milli or micro XRF spectrometers for improving analytical and sensitivity range, including software development and of standardization procedures 2) to evaluate the potential of in-situ XRF analyses to provide specific answers to archaeometrical/conservation problems on various kind of cultural materials and to access their complementarity role in comparison with other techniques recently developed also with portable instrumentation (XRD, PIXE-alpha) and 3) to in-situ XRF surveys of archaeological materials or of historical outdoor monuments for

characterization and conservation purposes. Close collaborations have been established with INFN-LNS, Catania-Italy, Department of Conservation of Antiquities & Works of Art at Technical Educational Institute of Athens, Technical University-Berlin, Institut für Atomare Physik und Fachdidaktik, Public and private museums in Greece, Cultural Foundations and with foreign archaeological schools in Greece (American School for Classical Studies, Wiener Laboratory and the Ecole Francaise of Athens). In particular, a more analytical report will be given to selected activities:

c1) Within the INCO-FP6 STREP project PROMET (project, 1/11/2004-30/10/2007) entitled: “*Innovative conservation approaches for monitoring and protecting ancient and historic metal collections from the Mediterranean basin*”, the Institute of Nuclear Physics has undertaken at first the task to develop and evaluate a portable micro X ray Fluorescence ( $\mu$ -XRF) spectrometer. The portable micro-XRF spectrometer will be applied next (within 2006–2007) to survey metal collections at different countries in the Mediterranean region, like Jordan, Malta and Egypt. Our specific objective would be the development of analytical methodologies/procedures for the identification of corrosion products on the altered surface of metal artifacts.

c2) By the end of 2004, the final version of a prototype portable XRF spectrometer was developed at the Institute of Nuclear Physics by the X ray Spectrometry group. During 2005, a systematic quality control of its performance was undertaken. In the final integrated configuration, specific hardware components were carefully tested with respect to their corresponding specifications; the most frequent malfunctions were recorded and a first draft of an operational guide was prepared. As a next step, standard procedures were developed for the analysis of different types of materials (geological samples, metal alloys etc). In particular, the high voltage setting value, tube current setting and the use of a specific combination of filter materials were optimized according to the type of the analyzed sample and the particular instrumentation units. Two specific calibration procedures were developed, one based on the fundamental parameter approach with a semi-empirical description of the exciting tube spectrum and a

second one based on the internal Compton scattering method. Both calibration procedures were based on experimental measurements of a large set of pure materials and were evaluated for four similarly developed spectrometers. Their accuracy in the quantitative analysis of an unknown sample was evaluated by comparing measured concentrations and certified ones from about ten (10) reference materials, geological and metal alloys as well. The final assessment was that an overall accuracy of better than 10% could be attained for major, minor and traces elements.

c3) Within various frameworks, the EC program "PROMET", the national "Archimedes" and the Pylos Regional Archaeological Project (PRAP) with the Department of Classics of University of Cincinnati, various on-site surveys, on different kind of archaeological materials were conducted during 2005.

- At the Messene archaeological site (South Peloponnesus, Greece), XRF analyses of a large number of excavated metal artifacts was undertaken, as a representative sample of the large bronze metal collection found on this site. Specific analytical procedures were applied for the characterization of a subset of bronze mirrors and in particular of the corrosion layers that appear on their surface.
- In-situ XRF analyses served as one of the diagnostic tools used towards the characterization of the conservation state of an outdoor bronze monument located at Nauplio city in Greece. The study of this outdoor bronze monument, Theodoros Kolokotronis, presented a unique opportunity to enforce the conservation standard for the treatment of cultural property. Since the type of deterioration depends on the exposure environment, all cultural property must undergo a proper documentation and diagnostic analyses before treatment.
- The X ray Spectrometry group was integrated in a multidisciplinary working group including archaeologists, conservators and restorers, which since 2002 within PRAP (Pylos Regional Archaeological Project) aim to study, conserve and preserve the wall-painting fragments from the Palace of Nestor at Ano Englianos in Messenia, Greece, the best preserved of the Mycenaean palaces. Within PRAP, in-situ XRF analyses have been

conducted continuously since 2003 for about 5-10 days/year. The measurements refer to the non-destructive characterization of pigments applied on the pictorial surfaces of the wall-painting fragments. The number of fragments which are kept at the museum storage room is estimated to be equal to 10000. At the final stage of the project a data base will be created including all the documentation and XRF analytical results. In the analytical characterization of the pigments from the Nestor palace wall-painting fragments, the INFN-LNS of Catania has been significantly contributed by means of in-situ XRD and alpha-PIXE analyses using corresponding portable spectrometers.

### 3. Other Activities: Organization of a Training School with the support of TC-IAEA.

Within the Technical Cooperation Project RER/1/006: Nuclear Techniques for Protection of Cultural Heritage Artefacts in the Mediterranean Region, 2005-2007, a regional training course on "Implementation of Portable X-Ray Fluorescence Analysis and Ion-beam Methods in Cultural Heritage Studies in the Mediterranean Region" was organized in Athens (27/3/2006-31/3/2006) by the International Atomic Energy Agency (IAEA) in cooperation with the National Center for Scientific Research "Demokritos", Institute for Nuclear Physics, and the Ministry of Culture, Directorate of Conservation for ancient and modern monuments. The purpose of the course was to provide training for scientists and conservators on the use of XRF analysis and ion beam methods in the field of cultural heritage. The training focused on in-situ analysis with theoretical lectures and practical exercises for real in-situ applications, and it was also included quality assurance/quality control aspects. Two participants from ten (10) regional countries were participated (Albania, Bosnia & Herzegovina, Croatia, Serbia and Montenegro, TFYR Macedonia, Turkey, Cyprus, Greece, Malta and Slovenia) and IAEA experts and other senior and experienced researchers from Greece and abroad were kindly contributed as lecturers.

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## Poland

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The X rays fluorescence group is a part of Department of Nuclear Methods, Faculty of Physics and Applied Computer Science, University of Science and Technology AGH, Cracow, Poland. Staff members include M. Lankosz –chief of the group, L. Samek, B. Ostachowicz, J. Ostachowicz, M. Boruchowska, A. Ostrowski and PhD students: J. Chwiej, S. Wójcik, M. Kastyak, A. Mańka.

The XRF laboratory is equipped with the multifunctional energy dispersive x-ray fluorescence system. It consists of micro-beam x-ray fluorescence spectrometer with capillary x-ray optics, the broad X ray beam from Mo secondary target for XRF analysis of bulk samples and total reflection X ray technique. Chemical laboratory is

equipped with pressure digestion system used for the sample preparation.

The group takes part in the investigations leading in few directions. The main topics concern on the environment, cultural heritage conservation, medical researches. L. Samek works on applications of fluorescence technique in cultural heritage and environment. She is doing analysis of aerosol samples (indoor, outdoor) collected on filters in the museums, historic churches. The aim of these researches is selection and modification of conservation methods of art objects. She was doing bilateral Flemish–Polish project untitled: “ Proper heating of historic wooden churches-characterization of air flows, particulate deposition and strains in the wood”. This project was conducted in cooperation with Department of Chemistry at University of Antwerp and Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, Krakow. B. Ostachowicz is working with TXRF technique applied in trace elements determination in the biological and environmental samples. Now she is involved in the project concerning the fitoslabilisation of postindustrial slug heap.

Next of the research activity of the Department of Nuclear Methods is application of the selected techniques based on synchrotron radiation to investigation of human central nervous system (CNS) tissue. The researches are conducted in cooperation with Institute of Neurology and Institute of Medical Biochemistry, Collegium Medicum, Jagiellonian University in Cracow. The studies are focused on the investigation of the role of trace

elements as well as main biological molecules in pathogenesis of selected disorders of human CNS, i.e. Parkinson’s disease, amyotrophic lateral sclerosis and brain gliomas. Thick tissue slices taken from selected areas of CNS were used in the experiments. The measurements were performed at ESRF in Grenoble, HASYLAB in Hamburg and LURE in Orsay. The synchrotron microbeam X ray fluorescence (SR-XRF) was applied to analysis of both topographic and quantitative differences in the accumulation of selected elements CNS tissue structures. However, the X ray absorption near edge structure spectroscopy (XANES) was used in order to determine the oxidation states of iron, copper and sulfur either in pathological or control cases. The analysis of main biological molecules in CNS tissue was carried out with the use of Fourier transform infrared microspectroscopy (FT-IRMS). The typical beam size of synchrotron radiation used in the measurements is between 1 and 15 micrometers, therefore it is possible to carry out the investigations at the single cell level. The obtained results enabled to find both topographic and quantitative differences in the distributions of selected elements as well as biomolecules between the samples representing neurological disorders and the control group. The research point out that the techniques based on synchrotron radiation allowed to determine elements and biological molecules at micrometer spatial resolution. Therefore they enable study of potential abnormalities in chemical composition of tissue micro areas, that may be involve in neurodegenerative disorders as well as carcinogenesis.

## Sri Lanka

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### Current Activities of ED-XRF Laboratory in Sri Lanka.

ED-XRF facility was established in the analytical laboratory of the Atomic Energy Authority in 2001 under the technical assistance received through IAEA TC project “ Development and Utilization of Nuclear Analytical Technology” (SRL/2/005). The facility comprises of X ray tube (Rich – Seifert), sample holder with secondary target assembly and a Si (Li) detector. The laboratory has also got

necessary facilities to analyze water samples by co-precipitating technique using APDC.

Our XRF laboratory has already established analytical procedures to use emission transmission method (AXII-QAES, P. Kump), back-scatter fundamental parameter method (QXAS-BFP), APDC co-precipitation method and thin and thick sample analysis method. We take part in research activities and provide analytical services to some industries, national institutes and Universities, research groups etc.

Selected activities carried out by the XRF Laboratory:

1. Research study on heavy metal concentration levels in crow feathers collected from different environments.
2. Research study on heavy metal concentration levels in industrial effluents released to a main water body (i.e the Kelani River).
3. Research study on hyper accumulating capacity of flora in Ussangoda area (Serpentine mineral deposited area)
4. Study on the possibility of removing heavy metals in liquid waste by bricks (low cost waste water treatment method).
5. Study on heavy metal contamination in soil collected from Tsunami affected areas.
6. Elemental analysis of air particulate matter to identify pollutants and pollution sources.
7. Provision of analytical services to archeological studies (qualitative information only)
8. Alloy analysis for technical evaluations

Our Group is involved in several projects in the fields of mineral exploration by using XRF techniques. In mineral sand analysis, we use back-

scatter fundamental parameter method (BFP). This method was first learnt by the national group during the expert mission of Dr. D. Wegrzynek, XRF Group, Seibersdorf Laboratory, IAEA.

The results thus obtained from some minerals and deposits are shown in Table 1. The concentrations of all elements except Zr were obtained from the measurements performed with Mo secondary target with X ray tube operated at 40 kV. Zr was measured with Ag secondary target.

In Sri Lanka, there is a rising demand for this analytical service as it can provide the customer relatively fast and reliable results at low cost. AEA has decided to upgrade the existing facility to TXRF through the IAEA technical assistance to meet the demand for the services to analyse water and other liquid samples.

In addition, Quality Assurance and Quality control procedures have been implemented for validation of analytical methods and check of accuracy of analytical results obtained. We have participated in the proficiency tests organized by the XRF Group of Seibersdorf laboratory and AQCS programme. Our Laboratory has demonstrated satisfactory performance in these exercises. As we recognize the importance of having national and international recognition for the analytical results generated by the laboratory, we initiated steps to fulfil the requirements of ISO/IEC 17025 standards.

Table 1: The Concentration elements determined in some minerals using quantitative analysis of AXIL-BFP

| Element | Iron ore   | Zircon (coarse) | Zircon (fine) | Rutile (Coarse) | Rutile (fine) | Garnet      | Ilmenite    | Monazite      | Quartz       | Raw sand     |
|---------|------------|-----------------|---------------|-----------------|---------------|-------------|-------------|---------------|--------------|--------------|
|         |            |                 |               |                 | Wt. %         |             |             |               |              |              |
| Ca      | –          | 3.3 ± 0.1       | 0.9 ± 0.2     | –               | –             | 2.4 ± 0.1   | 0.23 ± 0.04 | 0.6 ± 0.1     | 11.9 ± 0.1   | 1.0          |
| Ti      | 1.1 ± 0.2  | 11.0 ± 0.1      | 12.2 ± 0.1    | 67 ± 1          | 65 ± 1        | 6.08 ± 0.05 | 36.0 ± 0.2  | 1.05 ± 0.03   | 0.66 ± 0.03  | 26.0 ± 0.3   |
| Mn      | –          | –               | –             | –               | –             | 1.1 ± 0.1   | 0.59 ± 0.02 | –             | 0.013 ± 0.02 | 0.31 ± 0.01  |
| Fe      | 67.5 ± 0.3 | 1.49 ± 0.02     | 1.24 ± 0.02   | 0.48 ± 0.01     | –             | 21.3 ± 0.2  | 20.8 ± 0.2  | 1.12 ± 0.03   | 0.98 ± 0.02  | 12.40 ± 0.05 |
|         |            |                 |               |                 | ppm           |             |             |               |              |              |
| Zn      | 503 ± 30   | 257 ± 16        | 216 ± 10      | –               | –             | 2905 ± 30   | 170 ± 12    | 1690 ± 30     | –            | 309 ± 7      |
| Pb      | –          | –               | –             | –               | –             | –           | 190 ± 8     | 1640 ± 40     | –            | 97 ± 10      |
| Sr      | –          | 76 ± 5          | 25 ± 6        | 8.1 ± 0.5       | –             | 31 ± 4      | 44 ± 2      | 80 ± 3        | 347 ± 2      | 93 ± 3       |
| Y       | –          | 278 ± 10        | 560 ± 10      | 12 ± 2          | –             | 2020 ± 10   | 21 ± 2      | 4180 ± 30     | –            | 102 ± 3      |
| La      | –          | –               | –             | –               | –             | –           | –           | 91560 ± 1400  | –            | –            |
| Ce      | –          | 2580 ± 500      | 11580 ± 950   | –               | –             | –           | –           | 166400 ± 1600 | –            | –            |
| Pr      | –          | –               | –             | –               | –             | –           | –           | 16560 ± 490   | –            | –            |
| Nd      | –          | –               | –             | –               | –             | –           | –           | 50940 ± 1900  | –            | –            |
| Sm      | –          | –               | –             | –               | –             | –           | –           | 6052 ± 440    | –            | –            |
| Gd      | –          | –               | –             | –               | –             | –           | –           | 3816 ± 500    | –            | –            |
| Hf      | –          | 3730 ± 500      | 7690 ± 100    | –               | –             | 800 ± 50    | –           | 1353 ± 50     | –            | 1350 ± 40    |
| Th      | –          | 234 ± 10        | 1180 ± 20     | –               | –             | 1730 ± 20   | 51 ± 4      | 29040 ± 100   | –            | 140 ± 7      |
| U       | –          | –               | 160 ± 10      | 37 ± 2          | –             | 81 ± 7      | –           | 873 ± 20      | –            | 35 ± 7       |
| Zr      | –          | 120900 ± 300    | 266600 ± 570  | 5260 ± 12       | 3624 ± 10     | 19560 ± 34  | 1380 ± 4    | 53580 ± 210   | 92 ± 4       | 30560 ± 52   |



**IAEA**  
International Atomic Energy Agency

**XRF Newsletter No. 11**

July 2006

The XRF Newsletter is prepared twice per year by the IAEA Laboratories in Seibersdorf. Correspondence and materials to be considered for publishing should be sent to:

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Printed by the IAEA in Austria,  
August 2006