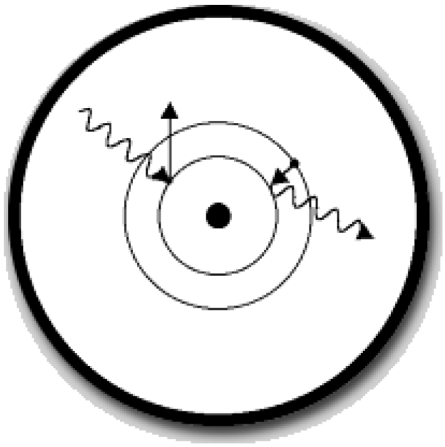
X-ray Fluorescence in the IAEA and its Member States

XRF NEWSLETTER



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CONTENTS

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Activities in the IAEA XRF Laboratory

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

Evaluation of the results of a Proficiency Test for X-ray fluorescence laboratories

The proficiency test scheme has been initiated in order to provide a cost-free service for the XRF laboratories that are interested in controlling and improving their analytical performance. Systematic participation in the proficiency tests provides the XRF laboratories with a possibility to fulfil the requirements of the QA/QC protocols as well as to monitor and sustain good analytical performance during a long period of time. The results can be used as guidance on the problems encountered and the actions to be taken in order to improve the analytical process. Keeping these benefits in mind, the IAEA Laboratory at Seibersdorf decided to organise proficiency tests for the XRF laboratories on a regular basis. The first run of the proficiency test included distribution of the IAEA certified material Lake Sediment (IAEA-SL-1) to 37 laboratories applying XRF technique for elemental analysis (the test samples were repacked and unknown to the participants). The results were submitted to the IAEA by 22 XRF laboratories. The results are kept fully anonymous and each participating laboratory received a code number known only to the organisers. The analysis was performed with different modes of XRF (including EDXRF with radioisotope and X-ray tube excitation, TXRF and WDXRF) combined with different sample preparation procedures (including pelletizing, fusion and digestion). The participants applied different software for X-ray spectrum deconvolution and quantification (including commercial versions, in-house developed and the Agency's package AXIL/QXAS). The results were grouped versus analytes/laboratories and compared with the assigned values of the analytes by calculating z-scores and u-scores and using three different fit-for-purpose criteria. The final reports of the proficiency test exercise together with certificates of participation were distributed to the participating XRF laboratories in July 2002. A relevant paper will be published in a peer-reviewed journal. By mentioning the proficiency test exercise we invite and encourage other XRF laboratories to take part in the next runs to be announced soon.

Extending quantitative analytical capabilities of EDXRF technique for environmental samples

It is well known that EDXRF technique has limitations in the quantitative analysis of light elements ($Z<10$). To circumvent this problem, different approaches have been used including improvements in EDXRF instrumentation and applying fundamental or semi-empirical methods for quantification. Lack of reliable information on the concentrations of the light elements (present in the so-called dark matrix) affects also the accuracy of the results obtained, e.g., for the environmental, geological or biological materials. A method proposed by the IAEA XRF Laboratory (in co-operation with the Ruder Boskovic Institute, Zagreb, Croatia) is based on an *a priori* determination of the low Z analytes in selected types of materials which next can be used in the XRF analysis of similar samples. To implement and test this idea, Rutherford Backscattering Spectrometry (RBS) was used to characterise the major elements in dark matrix of some plant-based samples (including biomonitors) and to apply the information as input to

EDXRF analysis. The derived stochiometry and mass ratios for the moss, lichen, and cotton cellulose samples were used as input to the quantitative routines included in the QXAS software (Simple Quantitative and Full Fundamental Parameter methods) applied for the analysis of plant-based IAEA reference materials. The results of the work were presented during the European Conference on EDXRS, Berlin, Germany, 16-21 June 2002.
Further information is available from Samuel Akoto Bamford (S.A.Bamford@iaea.org)

Application of the Backscatter Fundamental Parameter method for in-situ element determination using portable EDXRF spectrometer

The Backscatter Fundamental Parameter (BFP) algorithm was modified and adapted (with assistance of Mr. Wegrzynek, Poland, currently IAEA staff member and Leader of the XRF Group at Seibersdorf) for the use with portable EDXRF spectrometer. The method is based on coherently and incoherently scatter peaks of primary radiation applied to estimate the dark matrix of the analysed sample. To improve the accuracy of the correction procedures the differential mass scattering cross-sections are used. The BFP method requires a simple calibration by using only one standard sample. The algorithm takes also into account the secondary excitation effects. The method was tested for element determination in various matrices, including samples with minimum sample preparation. The measurements were performed in the laboratory for homogeneous samples prepared from standard reference materials as well as in the field for *in situ* element determination. In both cases a portable XRF spectrometer based on ^{109}Cd radioisotope source and Si-PIN photodiode detector was applied. The BFP algorithm performed well for the analysis of loose powder samples with unknown and variable fraction of dark matrix. The results of the direct *in situ* analysis of soil were in a satisfactory agreement with those obtained in the laboratory after processing the soil material (except for zirconium). The physical principles of the BFP method and the results obtained will be published in the X-RAY SPECTROMETRY journal.
Further information is available from Dariusz Wegrzynek (D.Wegrzynek@iaea.org).

Acquisition and installation of a new (commercial) EDXRF spectrometer

Most of the EDXRF spectrometers available in the XRF Laboratory at Seibersdorf were constructed and/or set up by the IAEA staff. As home-made instruments they appeared to be extremely flexible and ideal for training and research. However, they do not fully meet the requirements of a QA/QC scheme. In order to provide good quality analytical services to other research groups at Seibersdorf and Member States, the XRF Group decided to acquire a commercial EDXRF spectrometer. It is based on an X-ray tube (max. power 400 W) that allows excitation of a sample with optimum and reproducible geometry. Modification of the primary X-ray radiation is done by (Bragg) polarisation on crystals or by using secondary targets (typically Mo and Co). Quantification is based on empirical (Lucas-Tooth, Price; extended Compton scattering), Fundamental Parameter (FP) and hybrid models. The diversity of excitation conditions and quantification procedures available allows optimising the analytical procedure for

various analytes and materials. The new spectrometer was installed and thoroughly tested in the first half of 2002.

Redesign and rebuild of sample positioning system attached to the Agency's beam line at the Ruder Boskovic Institute, Zagreb, Croatia

As mentioned in the third issue of the XRF Newsletter, January 2002, the IAEA has its own PIXE/RBS beam line at a Tandem van de Graaff accelerator, Ruder Boskovic Institute, Zagreb, Croatia. In order to provide automatic control and improve fine positioning of the analysed samples, the sample positioning system placed in the vacuum chamber was redesigned and rebuilt (with assistance of Mr. M.Bogovac, Croatia). The work included the installation of a video camera, the development of an interface and computer program to control the video camera, its movements and the detector bias. The sample positioning system has already been tested.
Further information is available from Samuel Akoto Bamford (S.A.Bamford@iaea.org)

X-ray Fluorescence in Member States

During the last months we received contributions from a number of Colleagues on the current XRF activities in the Member States. Below there are short communications received from Argentina, Mongolia, Peru, Portugal, Spain, Thailand and Venezuela (this section is based on the original submissions, with minor editorial changes only).

Argentina

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The following projects carried out by the XRF Group are described below:

1. ***"Iridium Detection by Total Reflection x-Ray Fluorescence in Samples Providing from The Cretaceous-Tertiary Boundary and Experimental Amphibian Embryos "***
   The main purpose of this study is to report the high sensitivity of TXRF for Ir measurements in mineral and biological samples. Mineral samples originate from different horizon deposits in the Neuquén basin, Argentina. Ir anomaly seems to be related to diderophite material provided to Earth in large quantities most probably by a 10 km asteroid that impacted earth 65 Myears ago. Sample preparation procedures and multielemental information are available. In a particular case of amphibian embryos, the detected level of Ir was in the order of 1 part per million (bioconcentration factor of 9).

2. ***"Polymer Solutions on Glass: Adsorption Study by Total Reflection x-Ray Fluorescence"***
   Equilibrium properties of a polymer solution in the vicinity of a solid-liquid interface are locally altered relative to the properties in bulk. This is caused probably by attractive or repulsive interactions adsorbed fraction-adsorbent. In this way, theoretical models for flexible and rod-like polymers were published.The present work is related to study of the adsorption of 0 to 0.05 % aqueous scleroglucan solution on 400 to 600 μm glass microspheres. This study was possible by labeling the macromolecule by means of a chemical reaction with iodine, and then detecting by TXRF. Results show that for dilute concentration polymers behave in an anomalous way near the interface, contrary to the previous theoretical predictions. A first attempt to explain this behavior is presented.

3. ***"Total Reflection x-Ray Fluorescence Polymer Spectra: Classification By Taxonomy Statistic Tools"***
   The aim of this work is to explore the use of chemometric tools for the classification of synthetic and natural polymers with a mean molecular weight greater than 106. Spectra

obtained by TXRF technique were analysed in order to retrieve matrix information and to illustrate calibration procedure. The intensities of coherent and incoherent scattered peaks were selected for this purpose. The investigated polymers were aqueous solution of scleroglucan, polyacrilamide, polyoxyethylene oxide, glucomannan, 2,3,6-tri-o-ethylamylose and xhantan, between 0 to 1% (m/m) concentration range. MATLAB software is used for numerical calculations. Using a combination of principal component analysis and cluster analysis, the differences in composition of various analyzed polymers can be visualised.

4. ***Project: "Sampling extended areas: planning analytical protocols".***
Part of Co-ordinated Research Project: In-situ applications of X-Ray fluorescence (XRF) techniques. ARG11632
This project describes a methodology for sampling large areas taking into account QA and QC requirements (to ensure representativeness of samples). The proposed methodology offers a general approach to planning field investigations that could be useful for any type of environmental studies. In a report we describe the criteria for sampling planning, sampling protocols checklist, sampling devices, transportation and blank sample requirements. The final objective is to design a sampling strategy that will eventually allow the use of portable EDXRF instruments for *in situ* analysis of soil. This methodology will be applied for soil characterization in the zone of Campana, Argentina, by evaluating data coming from an EDXRF spectrometer with a radioisotope excitation. The samples were obtained from two different layers: A) the top soil (0-15 cm depth) and B) at 50-60 cm, covering an area of approximately 80 km^2.

Mongolia

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The Nuclear Research Center for Research and Training has got three EDXRF systems with isotopic sources (Cd-109, Am-241), secondary target and total reflection module. These facilities are used for training, research and analytical services. Training is provided for students of Physics and Chemistry at both undergraduate and postgraduate levels. The samples analyzed consist essentially of environmental, geological, biological, steel and alloy materials. Research activities are carried out in the areas of mineral resources and environmental studies (including analysis of air, water, biological and geological materials) and optimization of the measurement protocols.

Since 1976 the Radioisotope EDXRF system has been used for the determination of major and minor elements in copper-molybdenum and polymetallic ores samples in support of effective exploitation of mineral resources. With Cd-109 excitation the Cu-Mo ores and tail samples are analyzed for the contents of major elements, such as Cr, Mn, Fe, Cu, Zn, As, Pb, Zr and Mo. With Am-241 excitation the Cu and Mo concentrates are analyzed for major elements such as Cu, Mo, Fe and minor toxic elements such as As, Sb and Ag.

Since 1993 the TXRF system has been used for determination of toxic heavy metals (Ti, Cr, Mn, Fe, Cu, Zn, As, Pb, Cd, Hg) and other trace elements in water, alcohol, products of fermentation and food samples. This technique appeared to be extremely useful for environmental studies and pollution monitoring.

The analytical services are provided for research institutions, universities, governmental agencies, geological and environmental assessment companies, prospectors and miners. Other analytical services include the determination of trace elements in soil, sediment and rock samples.

The XRF group has also participated in different projects organized by the IAEA. Under a Co-ordinated Research Project (CRP) on "*In-situ* Applications of X-Ray Fluorescence Techniques" a portable XRF analyzer will become available. This instrument will be used for certification analysis of Gods of Buddha Church, works of art and other archaeological objects.

It is worth mentioning successful monitoring of the technological processes in the Cu-Mo ore processing plant by using XRF technique (3 single and 7 multi-element probes to analyze Cu-Mo ores, tails and Cu-Mo concentrates were installed in 2001).

Currently, two other XRF groups from the Central Geological Laboratory and Central Laboratory of Science and Technological University are working on the applications of EDXRF technique in the geological studies.

Peru

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Under the IAEA TC project PER/2/018 (1993-1994) on "Nuclear Training" an EDXRF spectrometer based on Cd-109 radioisotope source was installed. Then in 1998 a Total Reflection X-ray Fluorescence module was attached under the project PER/9/020 on "Analytical Nuclear Techniques for Environmental Control".

The XRF laboratory belongs to the Chemistry Department of Science Direction - General Direction for Promotion and Technical Development of the Peruvian Institute of Nuclear Energy and is located at the auxiliary laboratories of the Nuclear Centre.

The following three thesis for degree in chemistry were completed:

1. "Multielemental Analysis of archeological bones by X-ray Fluorescence for the reconstruction of diets at First Period in the Lima Culture"
2. "Determination of heavy metals in water of the Rímac river by Total Reflection X-Ray Fluorescence"
3. "Multielemental Analysis of sea water samples by Total Reflection X-Ray Fluorescence, using pre-concentration with APDC/MIBK" (in edition)

Currently we are working on another thesis dealing with "Multielemental analysis of biological marine samples by Total Reflection X-Ray Fluorescence and pre-concentration after microwave digestion".
In order to get the accreditation, we are working on validation of the analytical methods for determination of Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se and Pb in natural water by using Total Reflection X-Ray Fluorescence technique. The EDXRF spectrometer with Cd-109 excitation is used for the analysis of clays, potteries, soil, sediments, geological materials, bones, etc., mainly for the archaeological applications.

Portugal

Environmental Analytical Chemistry (QAA) Group, Instituto Tecnológico e Nuclear
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The Energy Dispersive X-Ray Fluorescence (EDXRF) laboratory of the QAA Group utilises a commercial XRF spectrometer with a 200 W rhodium X-ray tube, secondary targets and filters, and a Si(Li) detector, with a resolution of 160 eV at 5.9 keV. The analysis can be made using the direct or secondary excitation modes and an automatic 16-position sample holder. The most important studies carried out in our laboratory relate to the geochemistry of riverine sediments and adjacent coastal zones, in order to investigate the origin, dispersal pathways and modification of the sediment load carried by the large Iberian rivers into their estuaries and into the Portuguese shelf. The influence of anthropogenic activities on the elemental composition of sediments is also studied in order to assess the final destination of these anthropogenic trace elements in the costal marine environment. The current projects in this area are financed by the Portuguese Science Foundation under: *"Consequences of River Discharge Modifications on Coastal Zone and Continental Shelf"*, *"Late Quaternary Environmental Changes from Estuarine and Continental Shelf Sediments"*.

Another field of study is to assess the application of marine organisms from coastal areas as environmental monitors; due to their way of feeding based on filtering large amounts of water, these benthic organisms are particularly exposed to pollutants. The chemical compositions of sediments and marine sponges collected in the same area indicate a clear selective bioaccumulation of some trace elements in some species. Current work: *"Selective Elemental Bio-accumulation in Marine Sponges from Portuguese Coast"*.

The transfer of pollutants from soils to plants in contaminated mining areas has also been one of the study areas; mining activities produce large amounts of waste rock and tailings, containing chemicals and residues with high concentrations of heavy metals. The EDXRF analysis of soils and plants from different mining areas allows the identification of the plants with potential capacity to be used in phytostabilization procedures that may possibly reduce the risks of metals mobility and decrease the subsequent environmental degradation.
Work recently developed in this area: *"Elemental Uptake and Root-leaves Transfer in Cistus ladanifer L. Growing in a Contaminated Pyrite Mining Area (Aljustrel, Portugal)"* and *"Dispersal Pathways of Contaminants and Transfer to Plants System in the Urgeiriça Uranium Mill Tailings"*.

Due to the non-destructive nature of the EDXRF technique, it has also been used in study of artefacts with museum and archaeological interest. This includes the investigation of the production, circulation and consumption of metal artefacts from the Calcolithic to the Late Bronze and Iron Age Culture groups. We aim to contribute to the understanding of the technical conditions of the metallic artefacts production and circulation. The large amount of artefacts available with variable chemical compositions, the findings of foundries and the identification of distinct metallurgical operations (e.g. crucibles) may give a significant contribution to the understanding of the historical dynamics of a long period in the Pre-History in Portugal. The current projects are: "Metallurgy of *Penedo do Lexim* in the Nearshore Platform Northward from the *Serra de Sintra"* and "Natives and Phoenicians in *Almaraz"*.

The International Atomic Energy Agency has recently financed a project, which made possible to upgrade the XRF spectrometer, under the project "Environmental and Marine Studies Using Nuclear Related Techniques (IAEA TC/POR/7/003)".

More details about our research work, equipment, laboratories and staff are available at www.itn.pt.

Spain

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The Archaeometry Unit (UA) of the Material Science Institute of the Valencia University (ICMUV) has got portable EDXRF spectrometers with small X-Ray tubes and thermoelectrically cooled semiconductor detectors (Cd(Zn)Te and Si-PIN). In June 2002 a new facility based on Total Reflection X-Ray Fluorescence (TXRF) analysis will also be in operation at the UA.

The research activities of the UA include in situ EDXRF analysis of art objects from the Spanish Cultural Heritage. The following examples are worth mentioning:

- Identification of the underglaze and overglaze cobalt blue decoration of painted ceramics from Valencia (XIV-XIX centuries). This project is carry out in collaboration with Dr. Jaume Coll from the National Ceramic and Luxurious Arts Museum "González Martí".
- Reconstruction of the original tonality of blue degraded smalts on canvas from the measurement of the relationship of Co/Pb.
- Analyses and comparative studies of engravings elaborated with different techniques (etching and heliogravure) from the XVII - XX centuries, and EDXRF analysis of ancient and currently commercialised inks. This project is developed in collaboration with Dr. Rosa Vives from the Barcelona University.
- Identification and characterization of forged works of art.
- In the near future: quantitative and semi-quantitative multi-element microanalysis of solid and liquid samples by TXRF.

References:
1. J.L. Ferrero, C. Roldán, M. Ardid, E. Navarro. X-ray fluorescence analysis of yellow pigments in altarpieces by valencian artists of the XV and XVI centuries. *Nucl. Instr. and Meth.* A 422 (1999) 868-873
2. Ferrero, J.L., Roldán C., Navarro E., Ardid M., Marzal M., Almirante J., Ineba P., Vergara J., Mata C. Applications of the X-ray fluorescence analysis to the cultural parimony of the Comunidad Valenciana (Spain): painting, metal and paper. *Journal of Radioanalytical and Nuclear Chemistry.* Vol. 240, No 2 (1999) 523-528.
3. Ferrero J.L., Roldán C., Juanes D., Morera C., Rollano E. EDXRF analysis of pigments of works of art from the Spain's cultural heritage. *Advances in X-ray Analysis*. ICDD, Vol. 44 (2001) 425-430.

Thailand

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The EDXRF spectrometer used in our laboratory is the tube type (Rh) with LN cooled Si(Li) detector. Our research concentrates on chemically upgrading local ores and presently we are working on ilmenite, the low grade titanium-containing ore. EDXRF has been our main equipment in this kind of chemically upgrading treatment [1,2]. Recently, we are also interested in using EDXRF for gems analyses, due to its unique advantage in non-destructive nature which is important for gems or any other valuable objects. Besides usage in research, being the teaching institution, we also use it as part of the teaching courses mostly in the form of demonstrations of its analytical capability to students of mining engineering and chemistry.

References:
[1] Sumpun Wongnawa, Phadoong Boonsin, and Thippawan Sombatchaikul, "Determination of Impurities in Ilmenite Ore and Residues After Leaching with HCl-Ethylene Glycol by Energy Dispersive X-Ray Fluoresence (EDXRF)".
[2] S. Wongnawa, P. Boosin, D. Kongkaew, S. Yodbutra, and S. Pisuttanawat, "Preparation of Standards Mimicking the Ore Matrix for EDXRF Spectrometry of Ilmenite Ores", J. Trace Microprobe Techn., 17(1) 25-37 (1999).

Venezuela

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Staff: Prof. Eduardo Greaves, PhD and Prof. Laszlo Sajo-Bohus, PhD

Instrumental facilities include three PC-based XRF spectrometers:

TXRF system with a modified Wobrauschek's module fitted with a multilayer

monochromator,
Radioisotope excited (Cd-109 and Am-241 annular sources) with Si(Li) detector
Radioisotope excited (Cd-109 point source) with large plannar HP Ge detector.

Other facilities include chemistry laboratory with variety of sample preparation equipment. Our all PC-based spectrometers use the ageing Canberra S100 MCA with 286, 386 and 486 PC. For spectrum analysis the DOS version of the QXAS analysis package is used. All this equipment and software, although operational, require renewing.

We may also make measurements, through collaboration, with an ICP-AES on campus and a modern wavelength dispersive system (Siemens) located in an external laboratory (IVIC).

Currently we have 6 Thesis students and one Postdoctoral (3 undergraduates in chemistry, 1 undergraduate in physics, 2 PhD candidates in chemistry and one PhD in archaeology) working in various projects.

Current projects and techniques applied:

1) Measurement of lead as a contaminant in the environment. Matrices are: aerosols, street dust, automotive brake pads and calcium pills manufactured by the Venezuelan Pharmaceutical Industry (techniques applied are ICP-AES and XRF with Cd-109 to excite Pb K-alpha lines)
2) Determination of Platinum in blood of cancer patients by using TXRF
3) Analysis of the electrolytic bath in Hall-Herault aluminium reduction cells (wavelength dispersive XRF, ICP-AES and PGNAA)
4) Measurement of trace elements in Antique Ceramic figurines from the Archipielago Los Roques, Venezuela. A study of the origin of the ceramics (TXRF, XRD and Scanning Electron Microscopy)
5) Trace elements in Spirituous liquors (TXRF)
6) Direct analysis of trace elements in biological fluids (TXRF)

Publications produced by our group in the field of XRF during the year 2001:

L.M.Marcó P., T.Capote, E.A.Hernandez C. and E.D.Greaves, Feasability study on an in-situ microwave digestion prior to analysis of biological samples by total reflection X-ray fluorescence, Spectrochim. Acta 56B (2001) 2187-2194

L.M.Marcó P., E.Jimenez, E.A.Hernandez C., A.Rojas and E.D.Greaves, Determination of Zn/Cu ratio and oligoelements in serum samples by total reflection X-ray fluorescence spectrometry for cancer diagnosis, Spectrochim. Acta 56B (2001) 2195-2202

J.I.Bermudez, E.D.Greaves. P. Nemeth and L. Sajo-Bohus, Determination of Technetium by total reflection X-ray fluorescence, Spectrochim. Acta 56B (2001) 2247-2252

L. Bennun, E. D. Greaves and J. J. Blostein, New procedure for intensity and detection limit determination in spectral trace analysis: application for trace mercury by TXRF, Accepted for publication in X-ray Spectrometry (2002)

Publications of potential interest to the XRF community:

1. Sampling, storage and sample preparation procedures for X ray fluorescence analysis of environmental materials, IAEA-TECDOC-950, IAEA, June 1997
2. Industrial and environmental applications of nuclear analytical techniques, IAEA-TECDOC-1121, IAEA, November 1999
3. RM Report, The Newsletter for Reference Materials and Proficiency Testing Users, http://www.rmreport.com/

XRF Newsletter

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