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X ray Fluorescence in the IAEA and its Member States

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Activities in the IAEA XRF Laboratory

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

Micro-XRF analysis of metal alloys: Addressing the problem of micro-scale heterogeneity

The introduction of high performance X ray focusing devices in XRF spectrometers opened new analytical possibilities for fast, spatially resolved elemental analysis of surface details by either desktop or portable scanning X ray microprobes. Heterogeneities having the same size or being even larger than the exciting X ray beam spot size are expected to produce misleading quantitative results when the analysis of single spot or comparable size area is performed. To tackle this problem an analytical methodology was investigated with the aim of defining the size of an analysed area or equivalently the mass to be analysed that will eventually produce representative results of the metal alloy bulk composition.

Experimental

The scanning micro-XRF measurements were performed by a modular XRF spectrometer developed in-house consisting of a high power (1-3 kW), line focus, Mo-anode, diffraction X ray tube and a silicon drift X ray detector (SDD)[1]. The spatial resolution of the monolithic polycapillary X ray is 25 µm @ W-Lα with a filtered exciting X ray beam (50 µm Ni + 12.5 µm Mo). A computer controlled, motorized stage allows 3D translation and rotation of the sample. A laser triangulation position sensor and an optical microscope coupled to a CMOS camera provide real time image of the analysed region of the sample. The scanning and the data acquisition is controlled by the Spector/Locator software.[2]

The micro-XRF area scan measurements were performed on certified reference alloys such as the BCR-691 copper alloys, reference gold alloys produced and distributed by Helmut Fischer GmbH and silver alloys. In all the measurements, the experimental conditions were as follows: 45 kV high voltage, 30 mA tube current, 5×5 mm^2 scanned area, 50 µm step size and 20 s measurement time per step, whereas the QXAS software was used for the extraction of net characteristic X ray peak areas. Tin was determined by using the characteristic X ray Lα line and lead via Lβ line.

Methodology for data analysis

An optimum methodology to experimentally study the influence of sample heterogeneity on the precision of the micro-XRF analytical results is based on variation of the spot size of the exciting X ray beam. This approach requires the availability of different X ray lenses. An alternative way followed at the IAEA Nuclear Spectrometry and Applications Laboratory incorporated an exciting X ray beam with fixed spot size, but individual spot measurements were properly summed so that the final result represents effectively the analysis with an exciting beam of a larger spot size. In order to demonstrate the procedure applied let us assume, for example, a sample area of 600 μm × 600 μm scanned by 12 ×12 spot measurements with a step size of× 50 μm (Fig. 1a). The data analysis was based on the following steps:

1. The relative standard deviation (RSD) was calculated by considering all individual elemental intensities from all spot measurements (Fig. 1a).
2. All possible combinations of four not overlapping spot measurements were added up and the respective RSD was determined (Fig. 1b).
3. The procedure was continued by considering nine (Fig. 1c) and sixteen (Fig. 1d) not overlapping spot measurements and again the respective RSDs were calculated for all possible combinations.

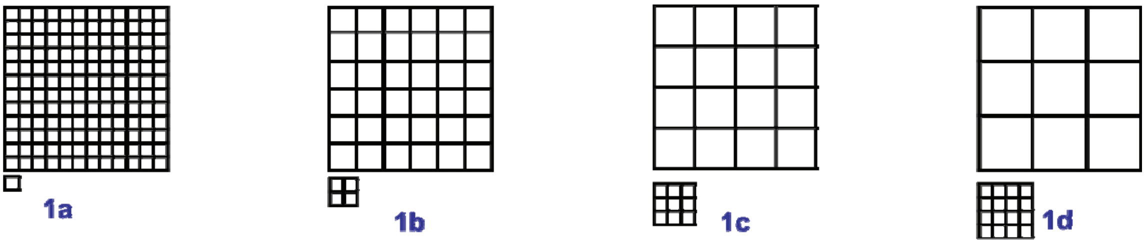

Fig.1: a) *Each square stands for a single spot measurement.* ***b,c,d)*** *Four, nine and sixteen neighbouring non overlapping areas/pixels are summed resulting in a new unit shell*.

It is expected that the RSD of the mean values will decrease as the analysed area/mass increases. In this case, the homogeneity level is defined at the corresponding area/mass where the overall RSD falls within a particular threshold (for example at 2%), comparable with the one produced by taking into account the instrumental and statistical precision only.

Results

For the major elements of the standard reference alloys the RSD's of the characteristic X ray intensities as measured after the irradiation of the multiplicity of the different increasing areas is presented. The results show that the gold and silver alloys are rather homogeneous at the level of 1-2% (see Fig. 2a), but leaded bronze alloys exhibit strong inhomogeneities (Fig. 2b). The report of BCR-691 series of copper alloys emphasizes the heterogeneity of the samples regarding the lead content and suggests that an analysis area with a diameter of at least 5 mm should be used in order to attain a precision better that 4%. Additionally, it is known that lead is insoluble inside a copper matrix alloy, creating globules. This is very well presented in the histogram in Fig. 3 showing the net intensities of lead Lβ lines from a bronze alloy (BCR A) containing 7.9% Pb.

Conclusions

The variability of micro-XRF results in the case of metal alloys has been investigated with the aim of defining the minimum area that represents the bulk composition. The methodology applied included large area micro-XRF scans and an appropriate data treatment to simulate exciting X ray beams of variable spot size. The gold and silver samples showed good degree of homogeneity for all elements even for a small analysis area (about 0.2 mm^2), whereas leaded copper alloys showed considerably larger heterogeneity for lead (about 4% for a 7 mm^2 analysed area) and less than 1% for Cu, Zn and Sn.

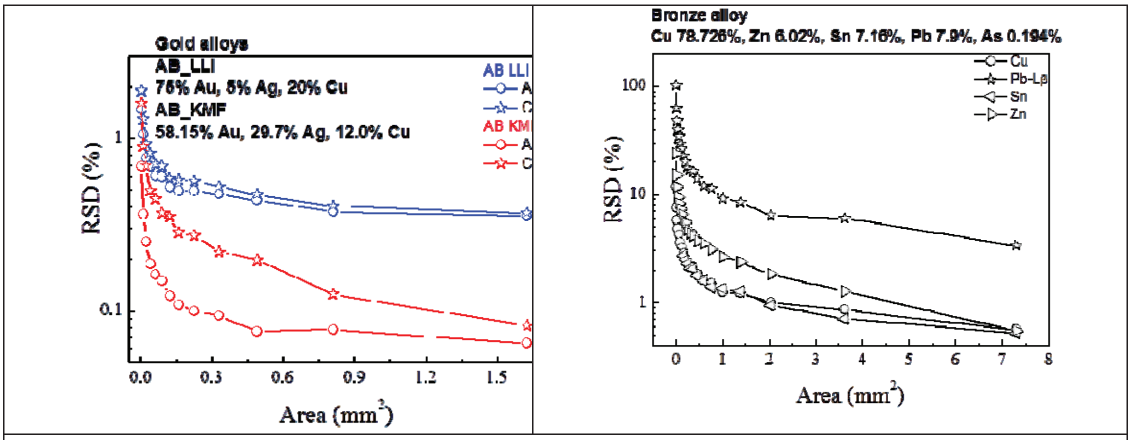

Fig. 2: Left: The gold alloys are homogeneous which means that even single spot analysis would provide representative results of the alloy composition. Silver is absent in the graph because of poor statistics. For the BCR-691 A copper alloy an equivalent analysed area of about 7 mm^2 is required in order to get precision in determination of lead better than 4%.

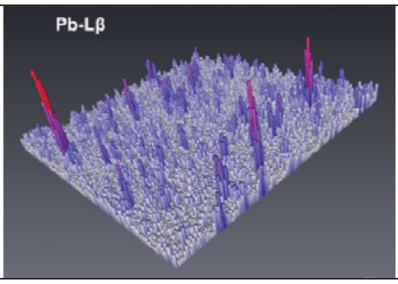

Fig. 3: Variation of PbLβ intensities for the quaternary BCR A certified copper alloy within the scanned area (6.5 mm × 6.5 mm).

References

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Meetings

Second Research Co-ordination Meeting (RCM) of the Co-ordinated Research Project (CRP) on Micro-analytical Techniques Based on Nuclear Spectrometry for Environmental Monitoring and Material Studies, Vienna, Austria, 10-14 October 2011

Considerable progress has been observed in recent years in the development and applications of micro-analytical techniques based on nuclear spectrometry. Major reasons for this include a possibility to use new excitation sources such as synchrotron radiation (SR) facilities, and low power compact X ray tubes designed to offer optimum excitation geometry, combined with the availability of advanced thermoelectrically cooled semiconductor detectors and of miniaturized or large scale X ray optics with improved performance. Consequently, the quality of characterization of various materials has improved considerably not only in large scale laboratory facilities (ion beam and SR sources) but also with in-house experimental set-ups, and new applications have become possible in support of applied research, teaching and education in nuclear science and technology in a wide variety of fields.

The following nuclear spectrometry (and related) techniques can be used for microanalysis: (i) X ray fluorescence (XRF), (ii) X ray diffraction (XRD), (iii) total reflection X ray fluorescence (TXRF), (iv) ion beam analysis based on applications of low energy particle accelerators (including particle induced X ray emission – PIXE, particle induced gamma-ray emission – PIGE, Rutherford backscattering spectrometry – RBS, nuclear reaction analysis - NRA), (v) synchrotron based techniques including extended X ray absorption fine structure spectroscopy (EXAFS) and X ray absorption near edge spectroscopy (XANES), (vi) X ray fluorescence micro-tomography, (vii) scanning electron microscopy (SEM), etc. The techniques are usually used for elemental analysis, 2D and 3D microscopy imaging and chemical speciation.

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

- Industrial applications including microelectronics, study of corrosion and corrosion protection (measurement of the composition and thickness of coatings), study of catalytic materials, waste characterization, characterization of thin films.
- Testing and quality assurance of materials for power generation (e.g. nuclear power, fusion).
- Environmental investigations including individual aerosol particle elemental analysis, fingerprinting and source apportionment, characterization and understanding hyper-accumulating plant systems.
- Applications to archaeology, art and conservation science (e.g. non-invasive characterization of raw materials, investigation of objects related to the cultural heritage, authentication and provenance studies of museum objects, characterization of corrosion and alteration products of artefacts in support of their conservation, restoration and future preservation).
- Mineralogy, geology and geochemistry.
- Food quality and agriculture studies.
- Biomedical and human health related studies (e.g. biomonitoring, trace element mapping of tumour tissues, bone, biocompatibility of implants, elemental deficiencies and/or anomalies in human tissues related to specific diseases, interactions and effectiveness of pharmaceutical products).

Taking into account the importance of the micro-analytical techniques based on nuclear spectrometry in different fields, the IAEA initiated a Coordinated Research Project (CRP) on special configurations and new applications of micro-analytical techniques based on nuclear spectrometry in order to coordinate and support the research efforts of the relevant laboratories.

The second RCM under the CRP was held in Vienna from 10-14 October 2011. The purpose of the meeting was to:

- Review the current status of research activities in the field of micro-analytical techniques based on nuclear spectrometry in the participating laboratories.
- Update information on the available instruments and facilities that can be used for the purposes of the present CRP and beyond.
- Coordinate the research activities of the present CRP and identify potential synergies and possible new collaborations among the participating groups.
- Refine and define a work programme for the remaining duration of the CRP and particularly for the period until the next RCM planned in April-June 2013.

PHOTO 1. Session of the meeting

Representatives of fifteen laboratories involved in the CRP (eight research contract holders and seven research agreement holders) from Argentina, Australia, Austria, Belgium, Croatia, Greece, Italy, Poland, Portugal, Slovenia, South Africa, Sri Lanka, Ukraine, United Arab Emirates and United

States of America participated in the meeting (see Photo 1) and presented their research activities in the relevant field, including details on the achievements to date (see Table 1) and future work plans of their research projects.

TABLE 1. Current status against expected outputs of the CRP (as of October 2011)

Expected output	Country
Improvements in the analytical performance of micro-analytical techniques	AUL – new 3-DRUM PM2.5 strip sampler has been developed, two 3DRUM sampling units have been operational for 12 months, monitoring dust storm events across east and northwest coasts of Australia AUS – improved GI-XRF software: combined GIXRF and XRR fitting CRO – measurements of uncertainty budget for PIXE analysis of particulate matter (PM) samples in collaboration with the IAEA Laboratories Seibersdorf GRE – sample preparation and investigation procedures for alloys of biomedical interest developed and adopted ITA – portable XRD measurements for high d-spacing minerals completed PL – software for chemical composition evaluation of individual particles with micro-XRF based on Monte Carlo simulation, Monte Carlo simulation based software for determination of element concentration profiles in layered samples with confocal XRF microscopy SLO – developed preliminary quantification software for low energy micro-XRF UA – improvement of the high brightness electron source providing submicron spot sizes, improvements of the high brightness ion source USA – new hiRX technique with high sensitivity and selectivity for elemental characterization
Standardized analytical procedures including optimized sample preparation techniques	ARG – analytical procedures for soil analysis. Optimization of sample preparation for soils and hair analysis. Improved QA/QC procedures. Soil analysis and cultural heritage in collaboration with other countries. AUS – development of GI-XRS and XRR measurement protocols CRO – calibration for particulate matter analysis by using new PIXE/RBS chamber GRE – IBA and corrosion procedures for alloys of biomedical interest tested and adopted ITA – improvement of the combined use of the portable PIXE and portable XRD for the pigments analysis confirmed by applications PT – analytical procedures for paper standard reference samples, samples analysed by EDXRF tri-axial spectrometer, PIXE and TXRF analyses performed, analysis by portable spectrometer ongoing SA – sample preparation and measurement procedures adapted for specific applications indicated in the plan of work SRL – developed procedures for microwave digestion for rice analysis by TXRF technique USA – novel small volume (pL) deposition for instrument calibration and sample characterization
Modern (upgraded) instruments and analytical methodologies including software packages for data acquisition and data evaluation	AUL – upgraded laser absorption techniques for black carbon strip filter measurements, developed automated laser absorption techniques for black carbon measurements on DRUM strips AUS – GI-XRS-EXAFS tested for As implants CRO – New gas ionization detector for heavy ion RBS re-installed at a dedicated chamber GRE – continuation of the Tandem vdG facilities improvement – proton microprobe arrived but not yet commissioned

	ITA – developed new high intensity 210-Po, constructed and tested new modular portable-XRD spectrometer PL – developed confocal 3D micro-XRF spectrometer and software for data processing, developed measurement control and data acquisition software for the 2D/3D micro-XRF spectrometer SLO – quantification of low energy micro-XRF scanning for light elements was improved SRL – analysis of soil samples by EDXRF, analysis of rice samples by TXRF, collection of rice samples UA – development of the anode cooling system in proton-beam induced X ray source, development of X ray optics with polycapillary lens UAE – Raman system installed and applied on environmental and cultural heritage USA – developed hiRX prototype instrument utilizing MWDXRF technology
New applications in support of research, innovation and technological development	AUL - combined HYSPLIT wind speed and direction data with IBA elemental data for the first time to identify dust sources from agricultural regions ARG – procedure for toxic element determination in biological tissues AUS – SRTXRF XANES of Fe in human cells, Sr uptake in trabecular bone of Sr-treated rats BE – preventive conservation studies finalized in two museums, continued in 13 museums and cathedrals (of which 4 in cooperation with Argentina and Portugal) and study of atmospheric particle interactions with ancient pigments CRO – ray tracing study of downsized HR PIXE spectrometer design for chemical speciation performed GRE – testing of electrochemical resistance of surface modified alloys of biomedical interest PL – investigation of matrix effect in XRF analysis of individual particles, investigation of the homogeneity of titanium layers in support to biomedical research, quantitative determination of the concentration profiles of Fe and Cu in multilayer polymer samples – validation of the Monte Carlo simulation software PT – upgrade the portable X ray micro-EDXRF system, new detector with thin Be window for detection low-Z elements installed SLO – influence of CuO nanoparticles in nutrient solution on plant growth tested SA – biofortification of staple food crops continued; plant-yeast symbiosis completed, study on fruit disorders completed, Ni speciation in hyperaccumulators continued UA – calculation and measurement of proton-induced X ray yield UAE – study of ‘dust particle’ effect on museum showcases started; study of size resolved elemental distributions of aerosol particles, study of composition of aerosol particles variation with time (hourly, weekly, monthly and yearly resolution) in collaboration of Dubai government USA – hiRX application for actinide element characterization
Publications including IAEA-TECDOCs and papers in national/international scientific journals	Published articles, conference proceedings and other reports: 31 + 5 + 5 (total: 41) Conferences contributions (poster and oral presentations): 54

The RCM also identified the following key synergies among the participants, which should lead to further interactions and collaborations:

Accomplishments to date:

A) AUL, CRO – fine particle sampling using the ASP cyclone system and IBA characterization techniques, construction of new PIXE chamber in Croatia design based on experience of Australia (fine particle sampler was provided to Croatia by Australia);

B) BE, ARG, PT – characterization of indoor and outdoor air pollution in the context of preventive conservation (3 museums in Buenos

Aires and 1 in Cordoba (ARG) and 1 in Lisbon (PT));

C) PT, BE – study of the deterioration process of ceramic tiles;

D) AUS, ARG, UAE – measurements and determination of Cd and As in soil samples;

E) ARG, SRL – chemical separation and speciation of As followed by AAS analysis, analysis of total As content;

F) AUL, PL, GR-S – confocal systems specification and Monte Carlo simulation;

G) PL, GR-S – investigation of stratified materials, quantitative analysis by Monte Carlo simulation and analytical algorithms.

Conclusions

To date this CRP has applied nuclear methods and related techniques to produce new outcomes not reported previously in areas related to characterization of air pollution, elemental pollution of soils and water, cultural heritage and preventive conservation, material analysis, microanalysis of biological and biomedical materials, actinide elements in nuclear materials and confocal X ray systems.

Some of these new outcomes include:

- New information on relative contributions of agriculture and farming to extreme desert dust storm events experienced annually along the east coast of Australia;
- Applied new high resolution X ray methodology for direct actinide element characterization of nuclear materials;
- Development of a new high efficiency portable PIXE-alpha spectrometer and new modular portable-XRD system;
- Application of complementary portable techniques (PIXE-alpha and modular XRD) for the non-destructive characterization of ancient mineral pigments;
- Construction of 2D/3D confocal XRF microscope and the measurement control/data acquisition software for the characterization of multi-layered multi-element samples;
- Size and elemental concentration of aerosol particulate matter in the United Arab Emirates was determined for the first time
- By combining complementary analytical methods available in different countries more complete characterization of critical pollutants in residential areas was achieved in Argentina
- Ion beam analysis techniques in combination with electrochemical corrosion testing led to the assessment of the corrosion resistance of surface modified metallic alloys for biomedical applications and contributed to the improvement of their properties.

The participants have created new and enduring collaborations across nuclear spectrometry based micro-analytical techniques and their applications that will benefit both the participants and other Member States.

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Technical Meeting on Applications of Synchrotron Radiation Sources for Compositional and Structural Characterization of Objects in Cultural Heritage, Forensics and Materials Science, Vienna, Austria, 17-21 October 2011

Synchrotron radiation (SR) has been exploited and explored as an analytical research tool over the past three decades leading to an exponential growth of research in many scientific disciplines. Nowadays, more than 50 synchrotron light sources have been built worldwide, whereas others are in the construction or planning phase. Synchrotron radiation offers remarkable analytical capabilities based on its unique properties such as a high brilliance, wide spectral range from the infrared to the hard X ray energy region, the wavelength/energy tunability, high degree and selection of polarization and the well defined time-space structure and coherence. These SR features coupled in a dynamic way with the various modes of photon matter interactions lead to the development and application, of a wide range of analytical techniques, with high elemental sensitivity and chemical specificity, 3D spatial resolution at the micrometer scale or below boosting analytical applications in a diverse range of scientific disciplines such as the materials science, energy research, protein crystallography, environmental science, chemistry, life sciences/biosciences, microelectronics, geological sciences including studies of extra-terrestrial matter, paleo-environment and industrial applications such as pharmaceuticals and biotechnology, automotive, semi-conductors, metallurgy, cosmetics, bulk chemical synthesis, characterization and study of cultural heritage objects and forensic applications.

Major objectives of the technical meeting included the following:

- To review the current status, developments, and trends in SR applications with emphasis on the fields of cultural heritage, forensics and characterization of energy related materials.
- To identify and map the needs for further improvement in SR instrumentation and analytical methodologies to achieve improvement and optimization of the analytical performance of the SR based techniques.
- To explore means to exchange and sharing of cross-cutting information and know-how between scientists in the relevant SR applications.
- To provide recommendations to the IAEA and its Member States on how to enhance and support training of young scientists in SR applications.

Participants from 13 IAEA Member States (Austria, Australia, Belgium, China, France, Germany, India, Italy, Lebanon, Portugal, Serbia, Thailand and Ukraine) as well as IAEA staff members participated in the meeting (see Photo 1).

PHOTO 1. Participants of the technical meeting.

Specific recommendations of the participants related to the role of the IAEA and the future developments and applications of SR sources are included in the final meeting report to be published soon.
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Technical Meeting on Sharing Experience, Current Status and Guidelines for Effective Utilization of Nuclear Instrumentation for Environmental Applications in Developing Member States, Vienna, Austria, 17–21 October 2011

In recent years considerable progress has been made in development and application of nuclear instrumentation and associated analytical methodologies for environmental monitoring. A wide range of possible applications includes the following areas:

- Determination of radioactive pollutants in environmental materials and radioecology;
- Remediation of contaminated sites;
- Monitoring of inorganic pollutants in air, water and soil by using nuclear analytical techniques;
- Determination of distribution of toxic elements in support of study of environmental impact (e.g., study of mechanisms of intake and/or release of toxic elements by biological species);
- In-situ characterization of environmental materials (e.g. mining sites, hazardous waste sites);
- Nuclear safety, nuclear security and nuclear safeguards.

Recently many IAEA Member States have expressed their interest in launching a nuclear power programme which would generate additional interest and needs in the field of environmental monitoring related to operation of the new nuclear power plants. In addition, some countries which already have nuclear power plants (about 30) are considering expanding their nuclear power programmes. Such programmes involve not only the construction and operation of nuclear reactors, but also a large set of associated activities such as mining, fuel fabrication, site assessment, environmental monitoring, radiation protection, commissioning/decommissioning, and waste management.

All of these activities require the utilization of equipment for environmental monitoring including equipment for sampling and nuclear spectrometry based analytical instruments. These instruments should be properly selected, installed, operated and maintained. Moreover, standardized procedures have to be applied to generate meaningful and reliable analytical data.

The Technical Meeting on '*Sharing experience, current status and guidelines for effective utilization of nuclear instrumentation for environmental applications in developing Member States*' was held at the IAEA headquarters in Vienna. The overall objective of the Technical Meeting was to review the relevant experience and current status of nuclear instrumentation for environmental applications and to prepare a report/guidelines for its effective utilization in developing Member States, including technical and managerial requirements and any possible role for the IAEA. The meeting's specific objectives included the following:

- To review state-of-the-art nuclear instrumentation for environmental monitoring including analytical methodologies;
- To identify capabilities and shortcomings for effective operation, maintenance and repair of state-of-the-art instrumentation used for environmental studies;
- To define practical training in operation and effective utilization of nuclear instrumentation for environmental applications including development of conventional and e-learning tools;
- To review tools and methodologies for meaningful sampling of air particulate matter, water, soil and sediments including installation of environmental monitoring stations;
- To review QC/QA for nuclear instrumentation and analytical methodologies;
- To define the role of national and international networks;
- To define the role of the IAEA in support of the developing Member State activities to effectively apply nuclear instrumentation for environmental monitoring.

The meeting was attended by participants from Bangladesh, Costa Rica, Cuba, Ghana, Greece, Kenya, Lebanon, Madagascar, Malaysia, Mexico, Morocco, Montenegro, Philippines, Serbia, Slovenia, Sudan, Tunisia, United Republic of Tanzania and the United States of America (see Photo 1).

PHOTO 1. Session of the meeting.

The participants formulated the following conclusions:

- Participation in this technical meeting was beneficial and provided an opportunity to share and exchange experience on state-of-the-art instrumentation and new applications of nuclear techniques for environmental monitoring.
- There is a continuous need to improve analytical laboratory services by using skilled and qualified personnel, well maintained equipment, and application of QC/QA standards (ISO 17025, ISO 9000, etc.); IAEA provides important support to meet these requirements in the developing countries.
- In order to ensure that reliable results are obtained NI and associated instruments should be operational throughout economic lifetime and the users in Member States must be well trained in both the operation and maintenance of the systems. Since most developing countries have limited resources and expertise as well as inadequate support from manufacturers due to geographical location and limited market, IAEA is expected to continue its assistance of Member States in this area.
- The participants agreed that their participation in the Nuclear Instrumentation Laboratory NETwork (NILNET) should provide a good framework to enhance their activities related to

nuclear instrumentation applied for radiation environmental monitoring.

- The participation in technical meetings, workshops, intercomparison exercises and in the forum of NILNET should contribute significantly to the dissemination of knowledge and sharing experience.

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Consultants Meeting on Review and Update of Training Programmes for Nuclear Instrumentation for Environmental and Other Applications, Vienna, Austria, 27–30 September 2011

A key factor in successful nuclear technology transfer is the availability of local personnel with skills to apply the technology and techniques appropriately and to ensure safe handling and maintenance of sensitive instruments. Therefore, the IAEA attaches a special emphasis on training of scientists, engineers and technicians from Member States, in particular from developing countries, in the field of nuclear instrumentation with respect to installation, operation, calibration, maintenance, refurbishing and repair as well as in methodology and applications of nuclear spectrometry.

The training is provided through various modalities including on-the-job individual fellowship training (IFT), group fellowship training (GFT), national, regional and interregional training courses, in-service training or e-learning (distance learning) by using Information and communication technologies (ICT) based training/learning materials. One must mention that all training programmes have been provided upon direct requests of MS or in response to their needs formulated under relevant Technical Cooperation (TC) projects.

The major objectives of the consultants meeting included the following:

(i) To review and update training programmes for nuclear instrumentation installation, operation, calibration, maintenance, refurbishment and repair as well as for methodology of environmental and other applications of nuclear spectrometry.
(ii) To discuss, review and assess teaching methods and aids required for effective training.
(iii) To identify modalities for effective implementation of the updated programmes in developing Member States.

The participants of the meeting from Austria, Canada, Germany, Poland and United States of America reviewed the training programmes for nuclear instrumentation and nuclear spectrometry provided by the IAEA Nuclear Spectrometry and Applications Laboratory (NSAL) at Seibersdorf in the form of group and individual fellowship training for the fellows from the developing Member States. The use of nuclear related technologies in the areas of human health (diagnosis and therapy), monitoring and assessment of environmental pollution (air quality management, toxic waste processing, remediation of contaminated sites), agriculture (food testing, food sterilization, plant breeding, insect pest control) and in many other fields related to characterization of materials and non-destructive testing, is increasing worldwide. This generates a growing demand for skilled and well trained personnel. The developed countries have the ability to effectively address this problem, but the developing Member States expect assistance by the IAEA with high quality training activities. The worldwide availability of training facilities in the areas of nuclear instrumentation and spectrometry are limited. Therefore, there is a well justified need for the training of fellows from the developing Member States at the IAEA NSAL which has appropriate facilities, well qualified personnel and access to the best experts in the field, coupled with long experience of training in a multicultural environment.

It was concluded that the training programmes carried out at the IAEA NSAL should be periodically reviewed by external experts through, e.g. consultants meetings the recommendations related to the update of its training programmes should be followed up on. In view of the growing demand for training of IAEA fellows the laboratory space for the training, in particular in the area of nuclear spectrometry should be increased.

The Member States should collaborate with the IAEA on effective implementation of their own training programmes including regional and national training courses and proper maintenance of the local training facilities.

The participants of the CM formulated a number of specific suggestions and recommendations to provide guidance on future training activities in support of effective utilization of analytical nuclear spectrometry based methodologies and related instrumentation.

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Conferences

International Conference on Development and Applications of Nuclear Technologies (NUTECH-2011), Cracow, Poland, 11-14 September 2011

NUTECH-2011 was a medium size international conference which covered topics related to nuclear energy, management of radioactive wastes, radiation chemistry, dosimetry and radiation protection, radiotherapy and radiometric measurements, radiopharmaceuticals and radioisotope production, applications of nuclear techniques in support of environmental and biomedical studies, agriculture and food processing, characterization of materials and protection of cultural heritage objects. The conference was attended by more than 150 participants from 22 countries: the majority of the participants were young scientists coming from various national research institutions and universities. The presented contributions including invited talks, oral presentations and posters were of a high standard. The venue of the conference (Faculty of Physics and Applied Computer Science, AGH University of Science and Technology, Cracow) appeared to be excellent for such an event. The IAEA representative delivered an invited talk on Review of the IAEA Activities to Support Applications of Nuclear Techniques for Characterization and Protection of Cultural Heritage Objects (authors: A.Markowicz, A.G. Karydas and R. Padilla-Alvarez). The nuclear techniques include XRF, IBA and NAA, dating techniques and gamma and electron irradiation methods. Various modalities of IAEA support were outlined including Technical Cooperation (TC) projects used for establishing basic infrastructure and development of skilled personnel, Coordinated Research Projects (CRPs) used for coordination of research and development carried out by the laboratories in both the developed and developing countries, and applied/adaptive research and development activities carried out in the IAEA Nuclear Spectrometry and Applications Laboratory at Seibersdorf. Emphasis was on the recent results and selected applications of X ray fluorescence (XRF) techniques including laboratory and in-situ measurements. XRF techniques provide information on the elemental composition of the CH objects which can be used in support of conservation and restoration, determination of production technology and origin, evaluation of alteration processes, diagnosis of previous modifications, and for preventive conservation. Major features of these techniques such as simplicity, speed of operation, immediate generation of multi-element analytical data, non-destructive character of analysis, portability and flexibility in terms of analysis of various objects made XRF techniques extremely attractive and fully recognized by the CH community. The facilities of the Nuclear Spectrometry and Applications Laboratory applied in support of the CH field, include various laboratory and (trans)portable EDXRF spectrometers, micro-beam XRF and absorption techniques integrated in one instrument, SEM as well as access to IBA techniques and synchrotron radiation sources in the laboratories in Croatia and Germany. Most of the applications of the XRF techniques were performed in cooperation with the Museum of Fine Arts in Vienna for characterization of paintings, coins, sculptures, bronzes, etc. Selected results of the applications for the elemental compositional characterization and 3D imaging were presented. The next conference in this series will be held in Warsaw, Poland in 2014.

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Support to Technical Co-operation projects

Final Coordination Meeting of the Regional IAEA/RCA TC project on Characterization and Source Identification of Particulate Matter in the Asia Region (RAS/7/015), Vienna, Austria, 14–18 March 2011

The meeting was attended by participants from Australia, Bangladesh, China, India, Malaysia, Mongolia, Myanmar, New Zealand, Pakistan, Philippines, Republic of Korea, Sri Lanka, Thailand and Vietnam. Its major objectives were: (i) to review and summarize the results obtained under the project in 2007-2011, (ii) to review and assess the role of the lead- (and assisting lead-) country coordinators and the contribution of regional resource units, (iii) to discuss the role and contribution of end users, (iv) to identify actions to ensure sustainability of air pollution monitoring activities, (v) to identify and discuss the regional priorities and activities in 2012-2013, and (vi) to review and agree on a preliminary regional work plan for 2012-2013.

Conclusions

The project has contributed to the improvement of air quality in the RCA region by the application of advanced nuclear analytical techniques (NATs) for the assessment of air particulate matter (APM) pollution. This has been achieved in particular through:

1) The characterization and use of long term data on fine and coarse APM to identify anthropogenic and natural ambient air particulate matter pollution sources across 14 countries in the Asian region. Their impact has been assessed.

2) Sufficient high quality elemental data was obtained by all Member States within the region covering the time frame from May 2000 to December 2009 to study large scale trans-boundary pollution and long range transport of air particulate matter. In particular:
    - Long range wind blown soil was identified in Australia, Bangladesh, China, India, New Zealand, Pakistan, Republic of Korea, Sri Lanka and Vietnam.
    - Smoke from biomass burning in Bangladesh, China, India, Indonesia, Malaysia, Pakistan, Republic of Korea, Sri Lanka, Thailand and Vietnam.
    - Emissions from coal fire power stations including secondary sulphate emissions in Australia, Bangladesh, China, India, Thailand and Vietnam.
    - Long range transport of sea spray hundreds of kilometers inland from coast lines in Australia, Bangladesh, India, Pakistan and Vietnam.
    - Volcanic emissions in New Zealand and the Philippines.

3) A reliable high quality database spanning 14 countries and five years between 2003 and 2008 was created and disseminated to all Member States in November 2010. This world-first elemental database is already assisting air quality managers to make informed decisions on pollution abatement and control strategies. The data from this database has resulted in over 95 refereed journal publications from ten Member States.

4) Greater use and investment in nuclear analytical techniques for air pollution studies is evidenced by the increased investment in facilities and resources. For example, new XRF systems in Indonesia (2011), Mongolia (2011), Myanmar (2008), Philippines (2007) and a new particle accelerator in Bangladesh (2011). New long term spin-off projects and collaborations have been developed involving significant extra resources within Member States in Australia, Bangladesh, India, Indonesia, Mongolia, New Zealand, Pakistan and Philippines.

5) The project has transferred knowledge and training related to NATs and air pollution to pollution managers and industry groups.

6) A significant end user community has been established throughout the region. End users in environmental monitoring are using the data that has been generated by nuclear analytical

techniques for monitoring, state of the environment reporting and mitigation purposes. End users ranging from local government, EPAs, and industry groups have been actively engaged by the project contributing both financial and in-kind support for the project activities.

The project discovered difficulties in engaging with external human health body and there was limited success in relating visibility to fine particulate concentrations.

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Final Regional Coordination Meeting of the TC Project RER/0/0815 on Using Nuclear Techniques for the Characterization and Preservation of Cultural Heritage Artefacts in the European Region,

Warsaw, Poland, 7–9 September 2011

The meeting was attended by participants from Albania, Azerbaijan, Bosnia & Herzegovina, Bulgaria, Croatia, Cyprus, Greece, Hungary, Former Yugoslav Republic of Macedonia, Malta, Montenegro, Poland, Portugal, Romania, Slovenia, Serbia, Turkey and Ukraine (see Photo 1). Its major objectives were: (i) to review and summarize the results obtained under the project in 2009-2011, (ii) to identify and discuss the regional priorities and activities in the coming years, and (iii) to review and consolidate a regional work plan under a new TC project proposal RER/0/034 on Enhancing the Characterization, Preservation and Protection of Cultural Heritage Artefacts for 2012-2013.

The objectives of the meeting were fully met. The results presented by the participants confirmed that the project was successful and the activities included in the regional work plan were implemented as scheduled. Major achievements of the project RER/8/015 include:

PHOTO 1. Session of the meeting.

- Sub-regional network, improved quality of study and characterization of CH objects, standardized procedures;
- Extensive exchange of experience; better understanding, cooperation and communication between cultural heritage and nuclear science communities at a national level; improved cooperation and communication at the regional level; promotion and popularization of NATs;
- High quality coordination meetings, regional training courses and workshops; national workshops; bilateral cooperation; scientific visits/missions; conference participation;
- Established intra-country collaborations between nuclear specialists and conservators in all participating Member States;
- Established bi- and multi-lateral collaboration between participating Member States;
- Substantial improvement in analytical capabilities in all Member States though national TC project and national contributions to acquire up to date equipment; enhanced utilization;
- Increased integration of different nuclear techniques for radiation treatment and better characterization of CH objects;
- Close to 100% participation in the regional group events with a large participation of early-stage researchers resulting in increase of skilled personnel in the region;
- Recognized importance of national seminars to raise awareness on the advantages of nuclear techniques in support of CH field.

The participants confirmed their interest in continuation of the activities related to applications of nuclear techniques in CH field and agreed on a slightly modified 2012-2013 regional work plan. During the meeting visits to the Museum of Marie Sklodowska-Curie (see Photo 2), the National Museum (see Photo 3) and the Archaeological Museum were organized.

PHOTO 2. Visit to the Museum of Marie Sklodowska-Curie.

PHOTO 3. Visit to the National Museum.

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Final Coordination Meeting of the IAEA Technical Cooperation Project RER/2/005 on Characterizing Seasonal Variations in Elemental Particulate Matter Concentrations in European Urban and Rural Areas under Different Climatic Conditions

Vienna, Austria, 5–7 October 2011

The meeting was attended by representatives from nuclear science and environmental protection institutions from Albania, Bosnia and Herzegovina, Bulgaria, Croatia, Greece, Hungary, Lithuania, Malta, Montenegro, Poland, Portugal, Moldova, Serbia, The Former Yugoslav Republic of Macedonia and Turkey (see Photo 1). The major objectives were: (i) to review and summarize the results obtained under the project in 2009-2011, (ii) to identify and discuss the regional priorities and activities in the coming years, and (iii) to review and consolidate a regional work plan under a new TC project proposal for 2012-2013.

PHOTO 1. Participants of the Final Coordination Meeting (credit: M. Bernatonis).

Major achievements of the project include the following:

1) Infrastructure for collection of APM and meteorological measurements was established and effectively utilized;
2) APM was collected systematically and according to the agreed work plan of the project.
3) Collaboration was strengthened between participating countries (scientific visits and fellowships);
4) QA/QC for APM sampling and PM10 determination through intercomparison exercise of sampling equipment was performed in Greece;
5) Knowledge of source apportionment methodologies was improved;
6) Knowledge of methodologies on assessment of APM impact on human health was improved;
7) Students were involved in the project;
8) Collaboration was fostered with other institutes, universities and decision makers including dissemination of the results through conferences and peer review papers.
9) WHO guidelines and EU regulations were applied;

10) The relation of APM with meteorological conditions was established;
11) Sources of APM identified and reports were sent to the relevant authorities in five countries.

For more information please contact A. Markowicz (A.Markowicz@iaea.org).

Regional Training Course (RTC) on Characterization of Cultural Heritage (CH) Objects by Using Nuclear Techniques (RER/8/015), Ankara, Turkey, 11–15 July 2011

The RTC was organized and hosted by the Turkish Atomic Energy Authority and the Museum of Anatolian Civilisations in Ankara in co-operation with the IAEA. Twenty-seven participants from fifteen countries involved in the regional TC project RER/8/015 on Using Nuclear Techniques for the Characterization and Preservation of Cultural Heritage Artefacts in the European Region attended the event (see Photo 1).

PHOTO 1. Participants of the Regional Training Course.

The programme of the RTC covered a wide range of nuclear and related analytical techniques with a special emphasis on portable XRF spectrometers and in-situ analytical methodologies applied for characterization of cultural heritage objects. The lectures delivered by the local and external experts were of a high standard and provided the participants with adequate background, principles and limitations of the analytical techniques including XRF, micro-XRF, X ray imaging, Raman spectroscopy and FT-IR spectroscopy. The lectures were supplemented by practical demonstrations of the equipment and experiments carried out for the real cultural heritage objects. Two technical visits organized to the laboratories of the Turkish Atomic Energy Authority and the Museum of Anatolian Civilizations were a valuable component of the RTC. The participants were very active during the lectures and practical demonstrations, and shared their experience on the applications of nuclear analytical techniques in the home institutions. The analytical facilities of the host institutions were of a very high standard and quality, and access to the facilities was generously provided to the participants (see Photo 2).

PHOTO 2. Practical demonstration of a micro-XRF spectrometer at the Museum of Anatolian Civilizations.

For more information please contact A. Markowicz (A.Markowicz@iaea.org).

X ray Fluorescence in Member States

Austria

A Low Power Mo Tube TXRF Spectrometer with Electrically Cooled Silicon Drift Detector

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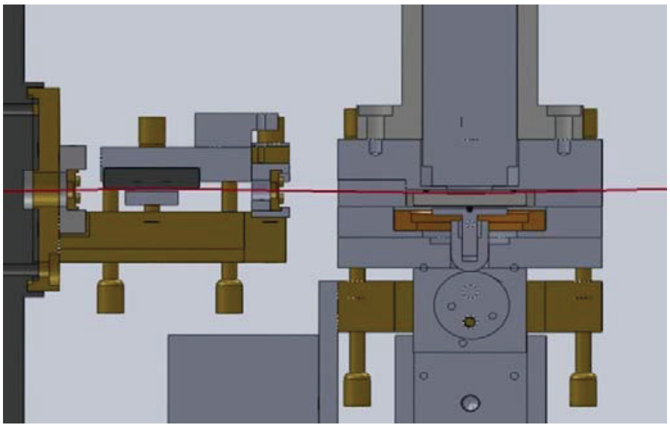
The design of a compact TXRF spectrometer yielding detection limits in the range of 10 pg for the medium Z elements like Rb is described. An air cooled Mo anode X ray tube (Oxford XTG), focus size approximately 100 × 120 µm² and 50 W power, thus operational with 50 kV and 1 mA from a compact small light weighted X ray generator (Matsusada, JAPAN) forms a compact source for TXRF.

High voltage and tube current of the generator is computer controlled and allows remote adjustment according to dead time depending on the sample. The beam is emitted from a point like focus and is collimated by a slit of 50 µm width, resulting in a rectangular shape 20 mm × 50 µm. Next in the beam path is the multilayer monochromator of 25 mm diameter on a quartz substrate (Mo-Si with a 2d spacing of d=4 nm, AXO Dresden), which delivers an intensive monoenergetic beam due to the high reflectivity offered. The multilayer is seated in an Al holder (see Fig. 1) and can be adjusted by means of a combination of rotator and translator stages to easily find the Bragg condition for the high intensive Mo-Kα which is then the excitation energy.



Multilayer

xcenter and sample reflector
from top downlooking detector

Fig 1. Schematic of the TXRF setup

High voltage and tube current of the generator are computer controlled and allow remote adjustment according to dead time depending on the sample. The beam is emitted from a point like focus and is collimated by a slit of 50 µm width, resulting in a rectangular shape 20 mm × 50 µm. Next in the beam path is the multilayer monochromator of 25 mm diameter on a quartz substrate (Mo-Si with a 2d spacing of d=4 nm, AXO Dresden), which delivers an intensive monoenergetic beam due to the high reflectivity offered. The multilayer is seated in an Al holder (see Fig. 1) and can be adjusted by means of a combination of rotator and translator stages to easily find the Bragg condition for the high intensive Mo - Kα which is then the excitation energy.

As it is a monoenergetic beam the spectral background is sufficiently low in the entire region except for the inelastic, elastic scattered primary radiation around 17.5 keV. Inserting a 0.5 mm Al filter in the primary beam path guarantees a low background in the low energy region also (5 keV and below). Here without Al filter, scattered primary radiation is observed, which results from total reflection of the direct beam on the multilayer mirror surface, increasing the background. The Bragg angle for Mo Kα corresponds to a certain cut off energy of a total reflecting mirror and thus the low energy part of the full spectrum from the tube is reflected and in the monoenergetic spectrum, but absorbed in the Al filter. The distances from source focus to sample (140 mm) and sample to detector (5 mm) are designed to be as short as technically possible. The sample reflector with 30 mm diameter and 3 or 4 mm thickness and the dried sample in the center, is placed horizontally with the sample on the top surface in a plastic holder serving as the carrier unit for in/out motion. This carrier fits in the main support which can be adjusted to total reflection conditions by means of a rotator/translator and tilter stage. The tilter is necessary to adjust the reflector parallel into the plane of the reflected beam to guarantee full illumination. The main support with the sample carrier unit also hosts the silicon drift detector SDD with 80 mm² active area (KETEK Munich). The AXAS system of KETEK, a compact electronic box supplies the bias, power for the Peltier and comprises the signal processing and MCA. The detector is mounted looking down versus the horizontally lying reflector. The reflector is moved up/down from the loading position by a drive. The reflecting plane is defined by 3 steel balls, against which the reflector is gently pressed manually by a spring. The correct adjustment is controlled by a lenseless CCD camera and a monitor or PC-TV board making the X ray beam directly visible and allowing the correct position after a sample change. The mechanical reproducibility is achieved without problems. The result is a compact easy to operate TXRF spectrometer which can be operated just from the mains, no LN2 for the detector, no water cooling for the tube and enough flux to achieve detection limits of 7 pg for Rb after 1000 s collection time with operating conditions of 50 kV and 1 mA. Results from measurements of various samples are shown together with a comparison of experimental and certified values from standard reference materials. Further improvements are planned with a curved Multilayer and a 75 W Mo anode tube with a focus smaller than 120 µm (the slit width used).

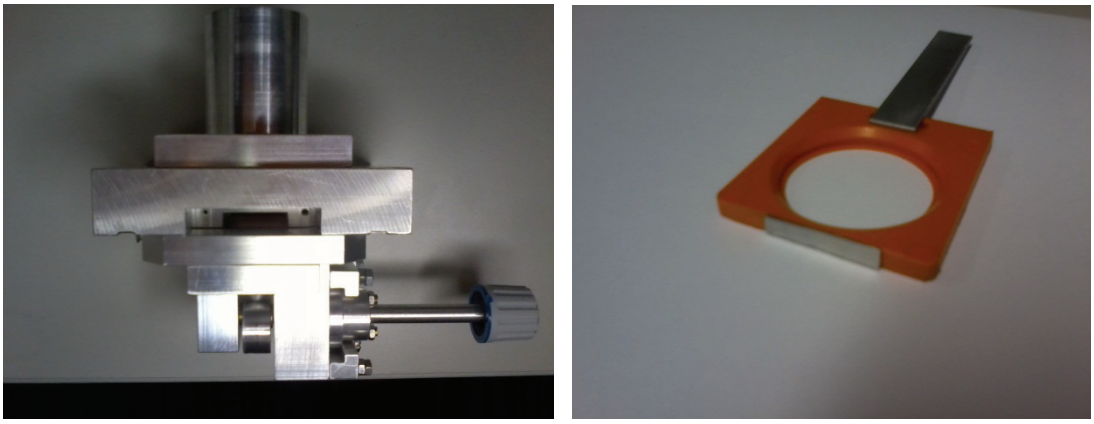


Fig. 2. Photo of lifting unit (left) and sample reflector holder (right).

Figures 2 and 3 show the design in detail for sample lifting and positioning as well as the overlook of the complete design with the X ray tube monochromator and detector.

The results of the analysis of a metal multielement standard sample and a reference sample of a 7 ng Rb single element standard, prepared from an aqueous solution using a 1 µL sample volume are shown in Figs 4 and 5.

The equipment is produced in cooperation with a Viennese mechanical workshop and uses commercially available components for the rotation and translation stages (ORIEL). It will be a pleasure to cooperate with the interested institutions and researchers of the IAEA Member States to design and produce this spectrometer at lowest costs.

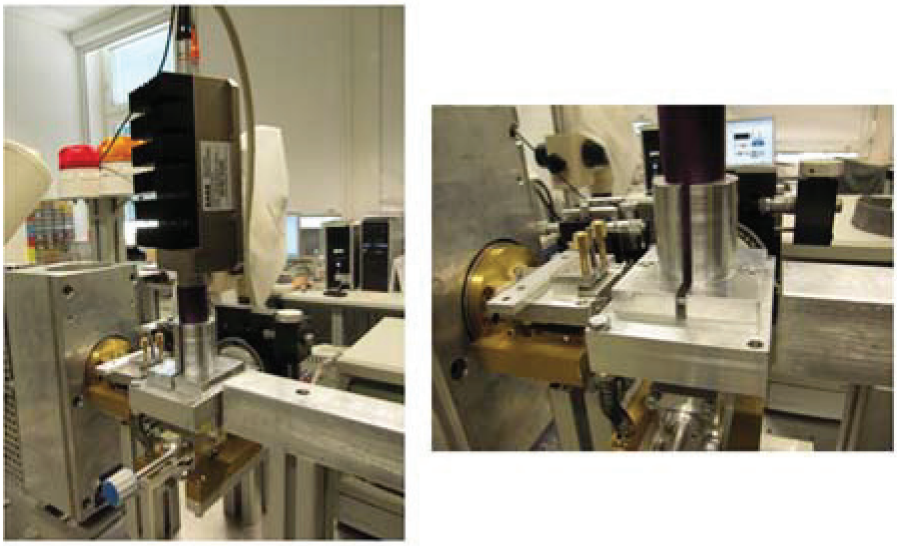
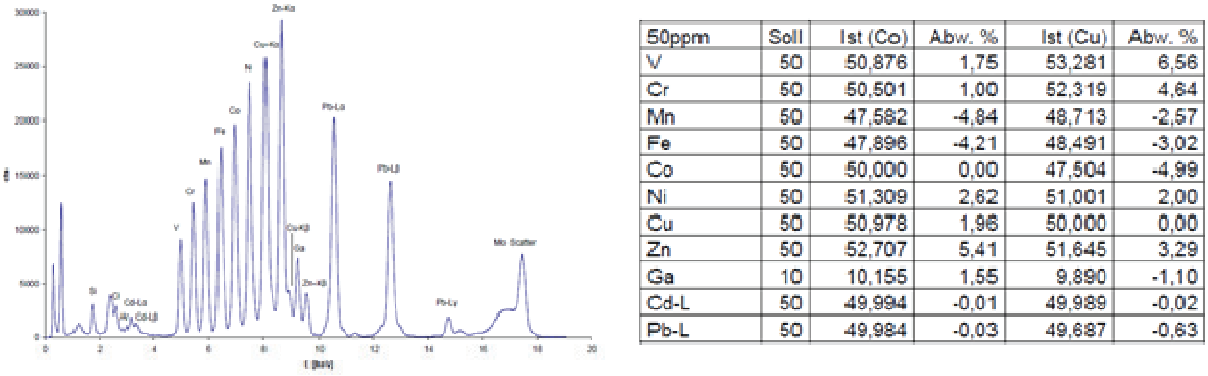


Fig. 3 Photos of the recently developed setup.



50ppm	Soll	Ist (Co)	Abw. %	Ist (Cu)	Abw. %
V	50	50,876	1,75	53,281	6,56
Cr	50	50,501	1,00	52,319	4,64
Mn	50	47,582	-4,84	48,713	-2,57
Fe	50	47,896	-4,21	48,491	-3,02
Co	50	50,000	0,00	47,504	-4,99
Ni	50	51,309	2,62	51,001	2,00
Cu	50	50,978	1,96	50,000	0,00
Zn	50	52,707	5,41	51,645	3,29
Ga	10	10,155	1,55	9,890	-1,10
Cd-L	50	49,994	-0,01	49,989	-0,02
Pb-L	50	49,984	-0,03	49,687	-0,63

Fig. 4 Spectrum of a multielement sample containing 50 ppm of various elements.

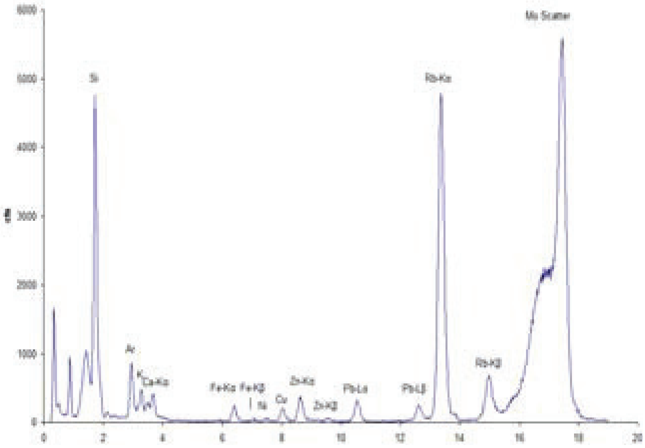


Fig. 5 Spectrum of a single element reference sample containing 7 ng of Rb (with impurities).

Acknowledgement: We want to thank KETEK Munich, Germany (www.ketek.net) for their generous loan of a recent model of the large area 80 mm² detector for our experiments.

Literature

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C.Felling, J.Prost and P.Wobrauschek, TXRF Spectrometer – Design with 50W Mo tube and SDD, Lecture 18, TXRF Conference 2011 Dortmund Germany June 07-09, 2011.

Poland

Activities carried out in the Department of Medical Physics and Biophysics, Faculty of Physics and Applied Computer Sciences, AGH University of Science and Technology

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Abstract

A confocal micro-beam X ray fluorescence microscope was constructed. The system was assembled from commercially available components, namely a low power X ray tube source, polycapillary X ray optics, silicon drift detector, and a video camera coupled to an optical microscope. The hardware and the developed software can already handle spatial as well as volumetric confocal scans. The spectrometer is controlled by software developed in-house. The graphical user interface as well as the communication protocols between hardware components was created by using the National Instruments LabVIEW. The camera image calibration and scan area definition software were also based entirely on the LabVIEW code. Presently, the main area of application of the newly constructed spectrometer is 2-dimensional mapping of element distribution in environmental, biological and geological samples with micrometer spatial resolution. The spectrometer was applied to homogeneity testing of titanium layers. A Monte Carlo simulation based quantitative analysis computer code for confocal XRF microscopy was also developed and tested with the use of a layered sample.

Introduction

Micro-beam X ray fluorescence spectroscopy (μ-XRF), also referred to as XRF microscopy, is a non-destructive technique of local elemental analysis. The technique is used in many fields of research for the determination of element distributions in alloys, geological, environmental, biological, and biomedical samples, where information about distribution of elements with micrometer spatial resolution, in originally heterogeneous samples, can provide clues about biological, chemical, and physical process under investigation. In confocal XRF set up the analysis is performed with the primary X ray beam focused by a polycapillary lens and the X ray fluorescence radiation emitted by the sample is focused by a polycapillary half-lens. The primary beam focusing lens sits in the path between the X ray source and the sample, whereas the polycapillary half lens is located between the sample and the X ray detector. The foci of both polycapillaries overlap creating an ellipsoidal testing (probing) volume through which the sample can be translated independently in the 3 spatial directions (3D). Typical spatial resolution of a tabletop set up is of the order of a few tens of micrometres. 3D XRF microscopy is a relatively new technique. It requires further development of procedures for quantitative element mapping.

This work describes a newly established laboratory facility for 2D/3D X ray fluorescence microscopy and its application to quantitative analysis of Ti layers were presented. Also presented were the results of testing of a newly developed Monte Carlo simulation based approach for quantitative depth profiling of elements in multielemental layered samples with the use of confocal XRF microscopy.

Construction of a 2D/3D XRF microscope

A photograph of the experimental set-up (configuration without the half lens) was shown in Fig. 1. The spectrometer consists of: (1) Mo-anode, side window X ray tube, 50 W air cooled, 50 m Mo filter in the primary beam path; (2) silicon drift detector (SDD), active area 10 mm^2, effective thickness 450 μm, 8 μm Be window, FWHM = 140 eV at 5.9 keV; (3) motorized XYZ detector stage, (4) motorized XYZΦ sample stage; (5) optical microscope coupled to a CCD camera; (6) polycapillary X ray lens, nominal focal distance 4.4 ± 0.3 mm, nominal focal spot diameter, FWHM = 13 μm at Mo-Kα; (7) polycapillary conical collimator (PCCC), nominal focal distance 3.4 mm, nominal focal spot diameter, FWHM = 28 μm at Fe-Kα. All the system components came with LabVIEW drivers or well defined software interfaces to be integrated and controlled by the developed virtual instrument (VI). The system was assembled on a stable optical breadboard table. As a radiation source, we utilized an X Beam from XOS (X ray Optical Systems Inc.) with integrated X ray tube and polycapillary optics. The tube was operated with air cooling and was powered by a maximum current of 1 mA at a maximum voltage of 50 kV. A silicon drift, Peltier cooled X ray SDD, AXAS-V system from Ketek, with a resolution of 144 eV at 5.9 keV was positioned perpendicularly to the photon beam. The AXAS-V system comprised of the detector, preamplifier, shaping amplifier, temperature control, internal power supply, ADC/MCA with USB PC interface. The accompanying software, running under the MS Windows operating system, provided full control of the ADC/MCA to initiate the measurements and to transfer data. It facilitated a DDE (dynamic data exchange) server and used the DDE standard protocols to communicate with other Windows programs, such as a LabVIEW client of the DDE server. The X ray detector together with the AXAS signal processing electronics were mounted on a stack of three (XYZ) translational stages 8MT167-25 (Standa) driven by stepper motors. The nominal spatial resolution of this device was 0.156 μm and the travel range 25 mm. The XYZ stages were controlled by personal computer (PC) via USB interface with software access through a NI VISA driver. The 8SMC1-USB motor controller came with a suite of software tools (VIs) for controlling the motors and software development by using the NI LabVIEW.

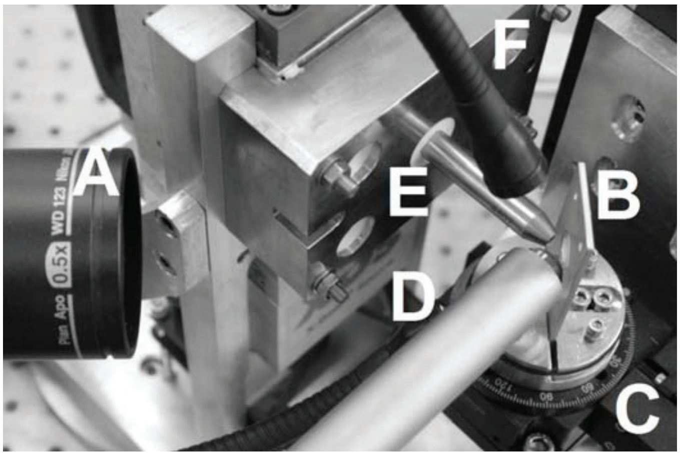

Fig. 1. Photograph of the experimental set-up: A – optical microscope lens, B – sample holder, C – XYZφ sample stage, D – detector snout, E – X ray tube with integrated polycapillary, F – light source.

The sample table translation axes XYZ were driven by the Physik Instrumente (PI) M-405CG stages with a travel range of 50 mm. All stages were equipped with high resolution DC motors and gear head drive providing 0.1 μm minimum incremental motions (unidirectional repeatability 0.2 μm). They were controlled by a C-848 PI motor controller. Additionally, a motorized rotation stage 8MR-151 from Standa was employed. The C-484 and the sample stages were commanded from a host computer via an RS-232 serial connection with ASCII commands to set operating modes, to transfer motion parameters, and to query the system and motion values. A collection of LabVIEW VIs was used to communicate with the C-848 and its connected axes. A video camera (Bosh LTC0455) coupled to an optical microscope focused on the sample was utilized to display details of the area excited by X ray beam. It was connected to a frame grabber ad-in card (NI-1411) installed in PC running MS Windows XP operating system. Camera control development was carried out in LabVIEW, by using NI Vision Acquisition Software with NI-IMAQ camera driver.

Analysis of the homogeneity of titanium layers

The spectrometer has been used to determine the homogeneity of titanium coatings on polymer substrate. 2D XRF mapping, without the use of the confocal half lens, was carried out. Two samples with different titanium coating thickness were examined. The aim of this measurement was to find the mean surface mass of titanium, MTi, and its relative uncertainty due to the layer intrinsic heterogeneity. Both samples were measured in several thousands of points (with 50 μm step). The X ray tube was operated at 50 kV/L mA. Time of measurement was 5 s per point for the 'thick' sample and 60 s for the 'thin' sample. For each sample, the observed relative variance of the sample surface mass was determined from the set of n measured points.

$$s^2_{observed} = \frac{1}{\left(M_{Ti}\right)^2} \frac{\sum_{i=1}^{n} \left(M_{Ti,i} - M_{Ti}\right)^2}{n-1} \qquad (1a)$$

$$M_{Ti} = \frac{\sum_{i=1}^{n} M_{Ti,i}}{n} \qquad (1b)$$

There were two main contributions to the observed variance: (1) the measurement methodology - s_{method}^{2}, and (2) the sample intrinsic homogeneity - s_{homog}^{s}:

$$s_{observed}^{2} = s_{method}^{2} + s_{homog}^{2} \qquad (2)$$

The uncertainty of the method was estimated and then Eqn. (2) was used to determine Ti layer thickness uncertainty:

$$s_{homog} = \sqrt{s_{observed}^{2} - s_{method}^{2}} \qquad (3)$$

The results of the measurements were presented in Table 1; additionally histograms of surface masses (with calculated Gauss shape) for both samples were shown (Fig. 2).

TABLE 1. Results of homogeneity testing of Ti layers

Sample	number of points	mean surface mass, M_{Ti} [µg/cm2]	$s_{observed}$ [%]	s_{method} [%]	s_{homog} [%]
1 (thin)	1491	4.63	5.78	3.18	4.83
2 (thick)	2555	2519	9.42	0.48	9.41

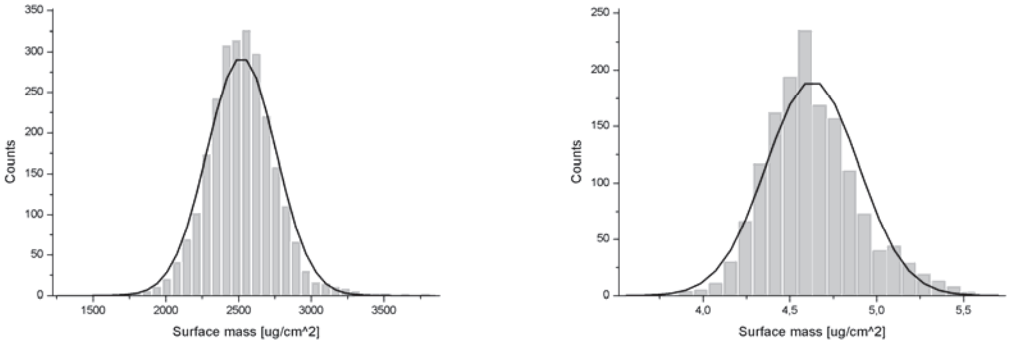

Fig. 2. Surface mass histograms for 'thick' sample (left) and 'thin' sample (right).

Validation of the Monte Carlo quantitative analysis code

The data collected during confocal XRF measurements of a stratified sample were modeled with a newly developed Monte Carlo (MC) simulation code [1]. This code simulates interactions between X ray photons and matter. The fate of every individual photon is being tracked from the point where it leaves a focusing optic through sample penetration until its detection in the X ray detector. The primary and secondary fluorescence and multiple photon scattering effects, taking into account photon polarization, are considered in this code. The analysed sample consisted of two layers made of Fe/Ni alloy foil (64% Fe, 36% Ni) separated by polypropylene foil. The experimentally measured intensity profiles and the Monte Carlo calculated concentrations of Fe and Ni versus depth were shown in Fig. 3.

Conclusions

The newly constructed 2D/3D XRF microscope can already handle aerial and confocal mapping of element distributions. Further expansion of the spectrometer hardware as well as the development of instrument control and acquisition software will be carried up. There is also a need for further validation of the Monte Carlo code with new stratified reference materials.

[1] M. Czyzycki, D. Wegrzynek, P. Wrobel, M. Lankosz, X-Ray Spectrom., 2011, 40, 88–95.

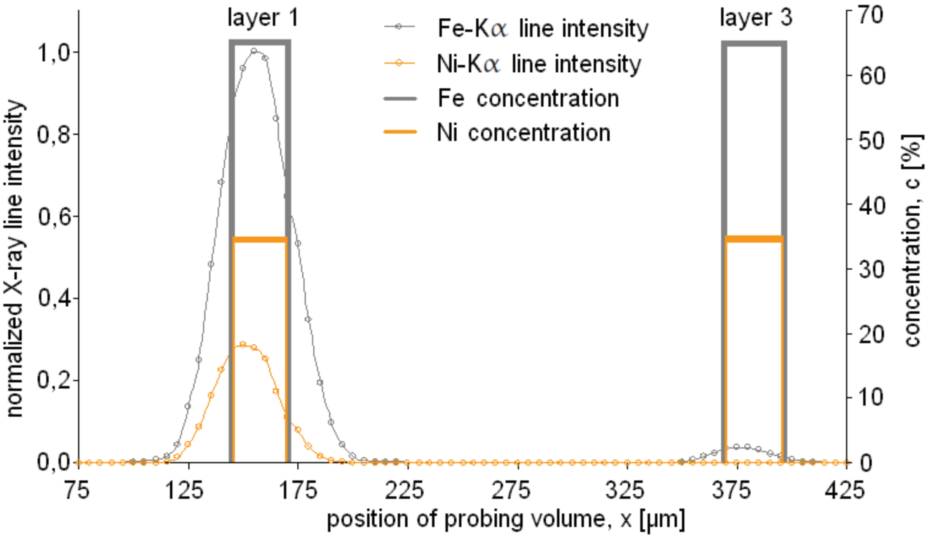

Fig. 3. A three layered sample. Depth related Fe-Kα and Ni-Kα line intensities were plotted. Chemical composition and layer thicknesses were reconstructed with the MC simulation.

Characterizing seasonal variations in elemental particulate matter concentrations in European urban and rural areas under different climatic conditions

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The regional IAEA Project (2009-2011) Characterizing Seasonal Variations in Elemental Particulate Matter Concentrations in European Urban and Rural Areas Under Different Climatic Conditions has been coordinated. The aim of the project is to evaluate concentrations of PM10 and concentrations of elements in PM10 collected during warm and cold seasons of the year and to find correlations with meteorological conditions. Several urban and rural areas were chosen in Eastern, Southern and Western Europe (Albania, Bosnia and Herzegovina, Croatia, Greece, Hungary, Montenegro, Poland, Portugal, and Serbia) for sampling air particulate matter. Such investigations can help to explain the possible sources of air pollution in these areas and may help to take actions to reduce the emissions in the future. Such investigations also help to compare the results from different areas and to apply the relevant EU guidelines. For the determination of the elements The energy dispersive X ray fluorescence method was used.

Source apportionment of particulate matter (PM10) collected in Cracow, Poland

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Samples of PM10 were collected in the winter of 2010 at two different sites in Krakow, Poland. One was located nearby a steel factory (in the Nowa Huta district) and the other was situated at a distance of 10 km from the first one (in the Krowodrza district). The detected mass concentrations varied between 33 and 320 μg/m^3. The daily limit value is equal to 50 μg/m^3. The Environmental Protection Agency was in charge of both - collecting the samples and providing the mass concentrations. Measurements of elemental concentrations and statistical analyses were performed at the University of Science and Technology. Mean concentrations of Ti, Cu and Br were almost the same for both sites. Mean concentrations for K, Ca, Cr, Zn and Pb were two times higher for the site in Nowa Nuta district than in the Krowodrza district. For Mn three times and for Fe four times higher values of mean concentrations were found for the Nowa Huta district. Source contributions to ambient PM10 were determined by factor analysis (FA) and multilinear regression analysis (MLRA) based on PM10 composition data which included elemental concentrations. During winter time the main contributors to pollution of PM10 have been local combustion, industry and other non-identified sources such as secondary aerosols. For the Nowa Huta district 53.1% of sources were identified as combustion, 28.5% as industry and 18.3 % non-identified. For the Krowodrza district, industry contributed 50.4%, combustion 46.1% and non-identified 3.5% to pollution of PM10. These results are presented in Fig. 1. While looking at the meteorological parameters we saw that concentration of K was inversely correlated to temperature for both sites. Potassium could come from wood combustion. Similar percentage contributions of pollution sources have been observed at both sites.

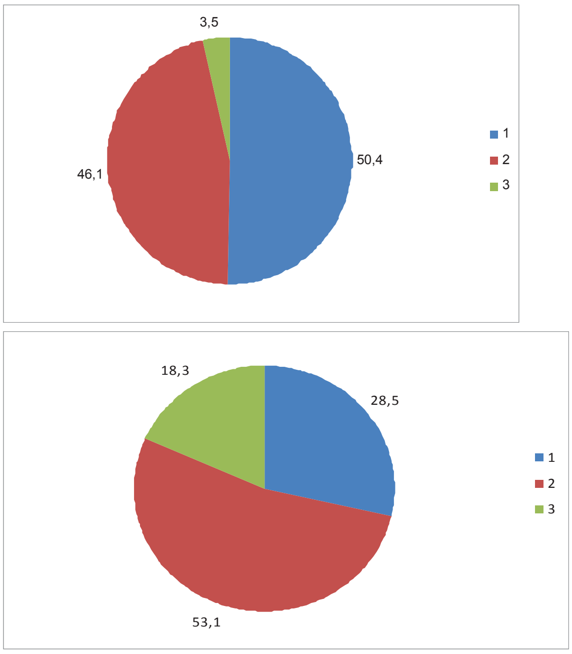

Fig.1. Source contributions at Krowodrza (left) and Nowa Huta (right); 1 - Industry, 2 - Combustion, 3 - Not identified.

Serbia

Recent activities of the XRF group formed in the Chemical Dynamics Laboratory of the VINCA Institute of Nuclear Sciences, Belgrade, Serbia

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The VINCA Institute of Nuclear Sciences was founded in 1948 as a centre for natural sciences, although it was first called Institute of Physics. Later the Institute became truly nuclear after installation of a nuclear reactor in 1956 when an emphasis on nuclear sciences was observed. The Government nuclear program was terminated in 1968 and consequently a number of multidisciplinary laboratories were formed. The Chemical Dynamics Laboratory was one of them.

Application of XRF spectrometry in our Laboratory started in 1992 with acquisition of a Si(Li) semiconductor detector and radioisotopes for excitation. Even then, the experimental set-up without sample compartment and annular geometry allowed measurement of samples with different sizes and shapes. Non-destructibility was also used as a benefit for different types of analysis including a small number of CH artifacts.

A breakthrough in this field was participation in the IAEA TC project RER/1/006 in 2005 when actually this XRF group was connected with the National Museum in Belgrade and possibilities of different analytical techniques were presented to the community of CH professionals. Also, cooperation with similar institutes in Europe was established and this enables exchange of the knowledge and experience. Activities in the IEAE TC projects RER/1/006 and RER/8/015 made a significant contribution to the work of the scientists from both communities.

With the help of the IAEA (cost sharing project in the scope of RER/1/006) a portable XRF spectrometer dedicated to CH analysis was constructed in our Laboratory, providing access to the interested CH professionals. In the past 5 years many small projects involving application of NAT in CH characterization and conservation, especially XRF spectroscopy, were implemented. We will mention some of them here with emphasis on various statistical tools for data processing which we initiated this year.

I. Investigation of the Cinnabar usage in Neolithic period on the Territory of Serbia

Museums in Serbia have a large collection of the Neolithic pottery excavated from different archaeological sites on the territory of today's Serbia. Our work placed particular emphasis on investigation of red colored ceramic with the idea of identifying the possible origin and usage of the red colouring pigment cinnabar. Samples from the (cinnabar) ore source at Mt. Avala, which is the nearest one to the archaeological sites, were also investigated. Equipment from three laboratories (Institute of Nuclear Physics, NCSR Demokritos, Athens, Greece, University of Ljubljana, Faculty of Mathematics and Physics and Jožef Stefan Institute Ljubljana, Faculty of Mining and Geology, and VINCA Institute for Nuclear Sciences, University of Belgrade) was used and PIXE, XRD, milli- and micro-EDXRF techniques were applied on several samples. The majority of samples, consisiting of red coloured shreds, figurines and altars were analysed only by milli XRF in Vinca Institute. Samples were excavated from three different neolithic sites: Vinča, Pločnik and Bubanj.

Analysis of the cinnabar ore from Mt. Avala showed the presence of trace element Cr a few times higher than in the Earth's continental crust which can be considered as a fingerprint characteristic element for that locality.

The XRF analysis of the red decorations on figurines and pottery shreds showed the presence of ochre pigments. Cinnabar as a coloring pigment was identified in only a few ceramic fragments and red decorations on figurines from the site of Pločnik. But no Cr traces were found in recorded XRF spectra.

Investigation of cinnabar powder found in a ceramic vessel at the Pločnik site, provided additional information regarding cinnabar usage and production in Vinca culture. XRD analysis of the powder revealed quartz crystals and illite besides cinnabar, so the conclusion was drawn that this cinnabar powder was produced by crushed ore and not by some kind of thermal process. A lack of Cr content in cinnabar powder gave us several possibilities for the origin of the powder although Mt. Avala cannot be excluded since the presence of illite was detected.

A final conclusion can be drawn that cinnabar was used as a pigment for figurine decoration at the Plocnik site in the Gradac phase of the Vinča culture, which is the oldest confirmed use of cinnabar mineral in the Vinča culture (end of the VI and the beginning of the V millennium BC).

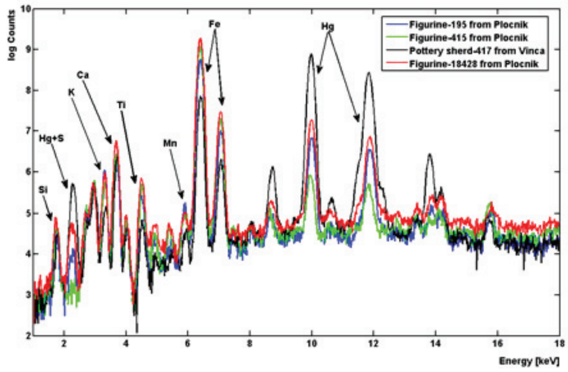

Fig. 1. EDXRF spectra of figurines.

Fig.2. Analysed ceramic shreds.

II. In-situ Investigation of the paintings in National Museum in Belgrade

Appropriate construction of the portable XRF spectrometer allows us to offer our analytical services to museums for in-situ non-destructive analysis of the pigments on different objects as well as metal and ceramic objects. As a part of the conservation process of Robert Hubert's painting Staircases in the Park of Villa Farnese in Caprarola we were asked to perform nondestructive analysis of the pigments on this large scale painting on canvas (148 × 217 cm) dated in the XVIII century (see Fig. 3). Restorers noticed that the original dimensions of the painting were changed and a question of the time of the modification arose. Analysis of selected cross-sections from the original painting and added strips under an optical microscope showed presence of different ground layers on the original painting and stripes. The primer of the original consists of two layers, first made of bolus (iron alumina silicates) and the second made of lead white pigment mixed with carbon and ochre. The primer on added strips is lead white.

Fig.3. Robert Hubert 's painting Staircases in the Park of Villa Farnese in Caprarola.

Analysis of the pigments on this painting was performed with a portable EDXRF spectrometer specially designed for analysis of paintings, icons and frescoes, equipped with a motorized platform with measuring head which consists of an AMPTEK X123 spectrometer with Si-Pin Detector (6 mm2/500 µm, Be window 0,5 mil/12,5 µm thickness and 1.5 inch detector extension) and pin hole focused air cooled X ray tube (Oxford Instruments, Rh-anode, max 50 kV, 1 mA). Two laser pointers were used for proper positioning of the measuring head on the painting. No filter and 40 kV, 300 µA and 120 s measuring time were selected as experimental parameters, and kept constant during all measurements. Also, distance between the sample and the X ray tube was 21 mm, between sample and detector was 16 mm while the angle between detector and X ray tube was 45°.

Earth green pigment, red ochre, orpiment yellow (As), vermilion (Hg), and Prussian blue (Fe) pigments were identified on the original size painting. Identified pigments on the original painting correspond to the known period in which painting was created (1768-69). Analyses of the pigments are also performed on the other painting by the same artist from that period which allows us to draw some conclusions about author’s palette.

Determination of the range of pigments on the strip and its dating proved to be complex because of the two layers of pigments separated by varnish. Analyses have shown the presence of some elements characteristic of specific pigments only on the strips such as zinc, copper, chromium, cobalt and arsenic (see Fig. 4). Since copper and arsenic show linear correlation in some points it is concluded that one of the green pigments on the strips is emerald pigment dated not earlier than 1814.

Unlike the original, green pigment visible in cross sections of strips, is composed of copper pigment identified as malachite or verdigris. Both copper-based pigments were known in antiquity. Blue pigments on the stripes are also different from those from the original painting and permanent presence of Zn peak in all spectra recorded on added strips indicates usage of zinc white pigment which has been used as an oil pigment since 1848, although it was synthesized in 1746.

So for the first layer of the strip compared with the original may be said to have originated from the period close that of the original, although it is not likely that the artist himself undertook the intervention as the combination of pigments on the strip is different than that of the original.

We are certain that the second layer of pigments on the strip was made after the author’s death due to the presence of zinc white.

These results were confirmed with optical microscopy and SEM-EDS measurements on several cross-sections from the painting.

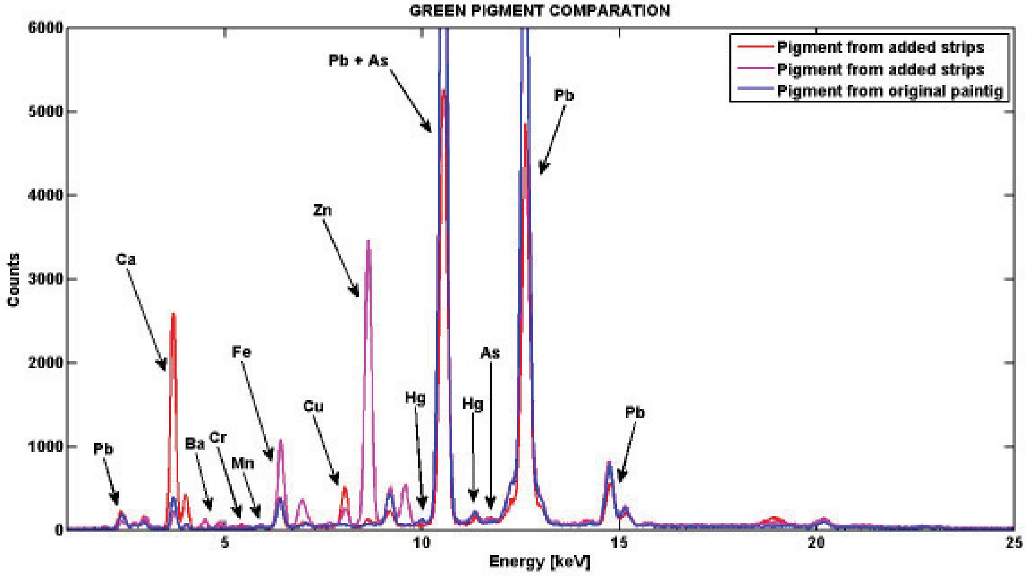

Fig.4. EDXRF spectra of the green pigments.

III. Classification of Neolithic ceramics using EDXRF spectrometry and Multivariate Statistical Analysis

Principal component analysis (PCA) and discriminant analysis (DA) are often used for multivariate statistical analysis in order to classify examined ceramic pieces. The aim of classification is to distinguish the local production and ceramics from those produced in other locations. The 32 ceramic shreds from the Vinca site, 22 figurines or fragments of figurines, four fragments of altars, two fragments of vessels from the Plocnik site and fifteen fragments of vessels from the Bubanj site were analysed.

The dimensions of ceramic shreds from the Vinca site range from 10 × 5 cm (large pieces) to 5 × 3 cm (the small ones) and the average thickness is approximately 4-6 mm. The clay body is homogenous in structure with a very shiny surface and glaze free. The figurines from the of Plocnik site (which belongs to the Vinca culture) are generally about 10-15 cm high and 5 cm in diameter. There are some larger but also much smaller ones looking like amulets. Figurines and altars were made of black ceramic. The smooth surfaces were painted with red and white pigments.

The pottery shreds that belong to the Starcevo culture from the Bubanj site have black ceramic bodies with red pigmented lines or spiral decorations on the exteriors. These ceramics are mainly painted with red pigments and/or decorated with graphite in different combinations of lines. The average dimension of fragments from the of Bubanj site is 5 × 5 cm and their thickness is 5-10 mm. All ceramic pieces were cleaned and the measured areas were polished before analysis. Each sample was analysed in three points.

The results obtained by EDXRF measurements of 67 investigated samples were used to form a training data set in this work. The training data set comprised average values for net peak area for ten elements: Si, K, Ca, Ti, Mn, Fe, Zn, Rb, Sr and Zr. These elements were chosen because for them the measurement uncertainty was less than 15% in the chosen experimental setup.

Principal component analysis

Principal component analysis (PCA) was performed in order to achieve dimension reduction. The original ten-dimensional space was transformed into a new two-dimensional one. The first direction in new space was chosen in the direction of maximal variance, determined by the first principal component (PC1). The other direction is determined by the second principal component (PC2) and is orthogonal to the first one. Auto scaling was chosen for data pretreatment. The influence of variable to sample grouping could be analysed from the loadings plot.

Discriminant analysis

The aim of discriminator analysis is to perform dimension reduction in such way to preserve class separability as much as possible. In the other words, it is necessary to find a transformation matrix M (Z=MX) which will reduce the original data space dimensionality in the lower one, but the prior knowledge about class membership must be retained as much as possible. In this analysis we used J=tr(S2-1 S1) as class separability criteria, where S1 and S2 are between and within the class scatter matrix, respectively. The next step is to design the proper classifier and calculation of the prediction ability for test data set. No pretreatment of the training set is necessary to perform DA.

From the Eigen analysis of the correlation matrix it can be noticed that the first three PCs explained 76.91% of the total variance among ten variables, where the first component (PC1) contributed 49.87%, the second one (PC2) contributed 14.12%, and the third (PC3) 12.91% of the total variance. The scores plot of PCA for investigated ceramic samples is shown in Fig. 5. It can be noticed that class separability is not so obvious. Even the autoscaling performed before PCA for the samples from Bubanj did not result in satisfactory separation of investigated samples into particular groups. The recognition ability for PCA is 72%, 83%, 23% for Pločnik, Vinča i Bubanj, respectively.

The loadings plot in Fig. 6 shows that values for Si, K, Ti, Fe and Rb are very similar variables (variables are very close to each other on the plot). These are the variables which load most significantly into the first PC since they are the furthest from zero in the right direction on the plot. They load with the same signs, which indicates that they are strongly correlated. Sr and Zr are strongly correlated. Along the second PC direction, one can see that Mn and Ca load positively and are correlated. Only Zn has small loads in both PC directions.

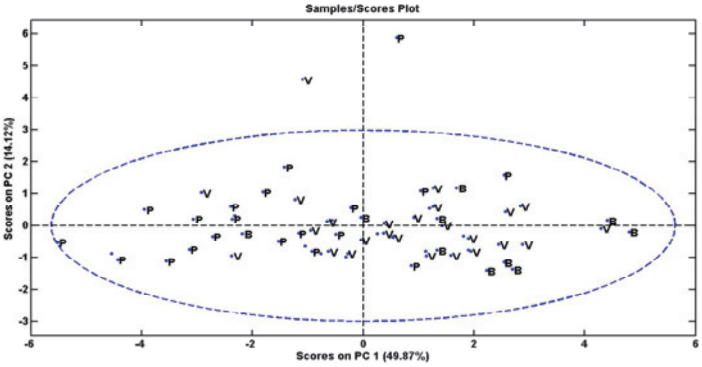

Fig. 5: Scores plot of first two PC components of ceramic samples.

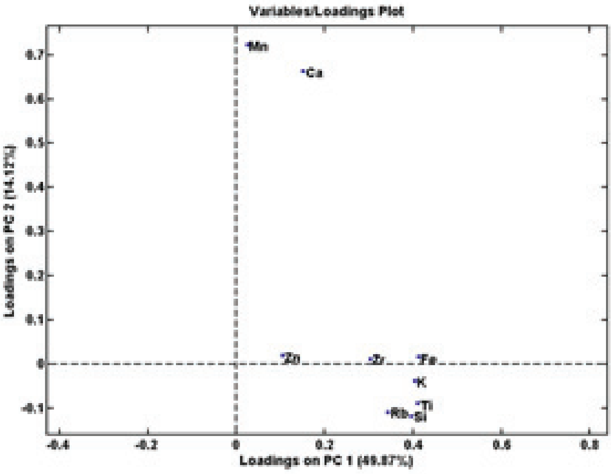

Fig. 6. Loadings on first two PC components.

The result of DA is shown in Fig. 7. It can be noticed that:

- DA was performed on raw data set;
- The choice of discriminator criteria was good;
- Good class separability was achieved even though *prior probability* was equal for all three classes.

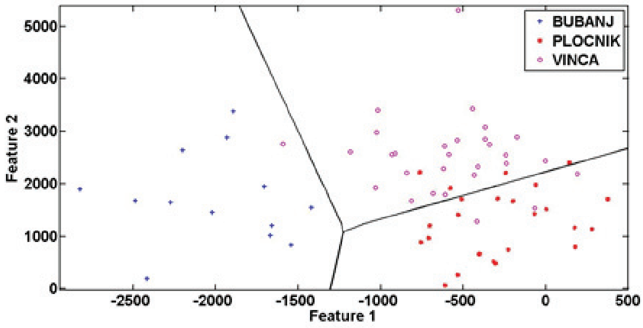

Fig 7. DA results and three linear classifiers plot.

As a conclusion we can emphasize that recognition ability of PCA is smaller than of DA.

By applying DA it is possible to perform the dimension reduction in a way to preserve the *prior* knowledge to class membership as much as possible. The DA performed on the training data set allowed us to project the linear classifier-most desirable for classification of unknown samples.

By comparing DA and PCA results we concluded that DA is a much more useful multivariate analysis tool for this purpose and this type of samples.

IV. XRF Analysis of the pigments on the paintings of famous Serbian artists from the XIX and XX centuries

As an attempt to create adatabase of used pigments for some famous Serbian painters, XRF non-destructive analysis of the pigments (see Fig. 8) was performed on more than 20 paintings which are attributed to Katarina Ivanovic, Nadezda Petrovic and Sava Sumanovic. Analysis of the preliminary results revealed some new information regarding usage of titanium white at the beginning on the XX century. This activity is still ongoing.

Fig.8. EDXRF analysis of Katarina Ivanovic's painting in the National Museum in Belgrade.

V. In situ non-destructive analysis of icons in Krusedol monastery

The latest and perhaps the most important activity was carried out in the last week of October 2011. A multidisciplinary team consisting of professionals from several institutions spent five days in the monastery of Krusedol at Fruska Gora, Serbia (see Fig. 9) in an attempt to perform characterization of icons which originate from four different periods from the XVI to XX centuries with numerous conservations in between. IR and UV Reflectography and EDXRF spectrometry were applied. Since these icons can be considered to be representative objects, the results of the investigations will be a basis for defining painting techniques during the past four centuries.

Fig. 9. Portable XRF analysis of the icons in the monastery of Krusedol, Serbia.

Philippines

Establishing the link between health effects and tire pyrolysis emissions using XRF spectrometry of $PM_{2.5}$

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In July 2009, a substandard tire pyrolysis facility for the production of fuel oil started operations in the rural municipality of Angat in the province of Bulacan, Philippines. Very soon afterwards, nearby residents noted adverse effects on the environment: oily effluents contaminating a nearby creek and adjacent rice fields; sacks of pyrolysis char dumped in empty lots, dark layers of soot on the ground, foliage and rooftops.

Adverse health effects were also noted. Symptoms observed were respiratory problems, such as chest congestion, difficulty in breathing, excessive phlegm, coughs, and colds, to skin allergies and eye irritation, as well as dizziness and stomach pains.

Fig.1. Pollutive tire pyrolysis facility in the rural municipality of Angat, Bulacan, Philippines. It is located right beside a poultry barn (left) and in the midst of rice fields. Black Smoke is emitted from several stacks and from the flare at the right.

It was necessary to link the observed health effects with tire pyrolysis emissions. Being a rural area, there were no monitoring stations in the area. Assistance was sought from the Philippine Nuclear Research Institute (PNRI) for the conduct of air particulate monitoring, in order to generate some information on the nature of emissions that were causing serious health conditions. $PM_{2.5}$ and $PM_{2.5\text{-}10}$ monitoring were undertaken with the Gent sampler and subsequent chemical speciation by XRF spectrometry was done, in an attempt to identify possible signature elements.

Sampling was done from January 31 to February 6, 2010 when the facility was operating and then on February 13 and 14, 2010 after the facility was shut down with the imposition of a cease and desist order. Multielement analysis of the filters was done at the Philippine Nuclear Research Institute (PNRI) using the PANALYTIC Epsilon 5 Energy Dispersive XRF spectrometer. Micromatter standards were used for the calibration of the XRF spectrometer. Analysis of the filters was performed using three secondary targets: CaF2, Ge and Zr secondary targets to yield concentrations of the following elements: Na, Mg, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Br, Pb. The analysis time was 200 seconds for each secondary target. Spectrum fitting and quantitation was by the method of elemental sensitivities using the IAEA software package QXAS (Quantitative X ray Analysis System). For quality assurance of the XRF results, NIST SRM2783, a multi-element reference material on filter medium was analysed.

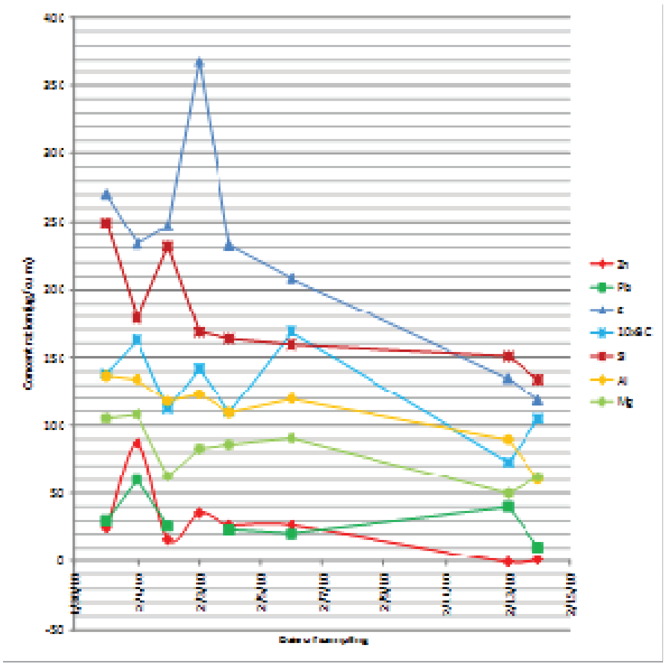

Fig. 2. Concentration of PM2.5 constituents associated with tire pyrolysis. Zn, Pb, S, Si, Mg and Al were analysed by XRF spectrometry and black C by reflectometry. The levels of all the elements went down after the tire pyrolysis facility was closed under a CDO. Zn declined down to almost zero.

$PM_{2.5}$ and PM_{10} levels were much below the WHO guideline values. Elements present above one per cent in $PM_{2.5}$ were: Black C, Na, S, Si, K, Ca, Al and Mg. Those present below one per cent in PM2.5 were: Fe, Zn, Pb, P and Ni. Ti, V, Cr, Mn and Cu were below detection limits. A drop in concentration was noted for elements present in tires (Zn, S, Pb, Al, Si, Mg and Black C) when the facility stopped operations after the implementation of the cease and desist order (CDO). Zn concentration dropped to zero, showing that all of the Zn comes from tire pyrolysis. Residual amounts of other elements (S, Pb, Al, and Mg) and black C could have come from other sources. Health effects ceased after facility stopped operations.

The data generated provided the technical basis linking health effects to tire pyrolysis emissions. This is now being invoked as the regulatory process takes its course and proponents of the facility claim that their emissions do not reach the affected communities.

Acknowledgements

This is to acknowledge the invaluable help of Ms. Gloria R. Jimenez in the preparation and weighing of air filters and Mr. Danilo A. Cuyco in the setting up and maintenance of the Gent air sampler.

Secrets of scalar energy infused products as revealed by XRF spectrometry

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The Nuclear Analytical Techniques Applications (NATA) Section (formerly the Analytical Measurements Research Section), as part of its function continues to provide analytical services. It has been offering qualitative or semi-quantitative analysis of solid samples using the autoquantify method in the Panalytical Epsilon 5 EDXRF spectrometer.

A recent health fad introduced in the market so called scalar energy infused products. Among these are ‘Quantum’ pendants and so-called energy bracelet/ballers. Claims of their ability to enhance energy levels and balance, detoxify, and prevent cancer have been made. In a demonstration class on the application of nuclear analytical techniques, particularly on the facility of XRF for rapid, non-destructive testing, one participant volunteered to have her ‘Quantum’ pendant tested. The results of the analysis made her decide to part with her pendant. What did XRF reveal that prompted her to that decision? Analysis of some samples of similar pendants by XRF revealed the secrets of these products.

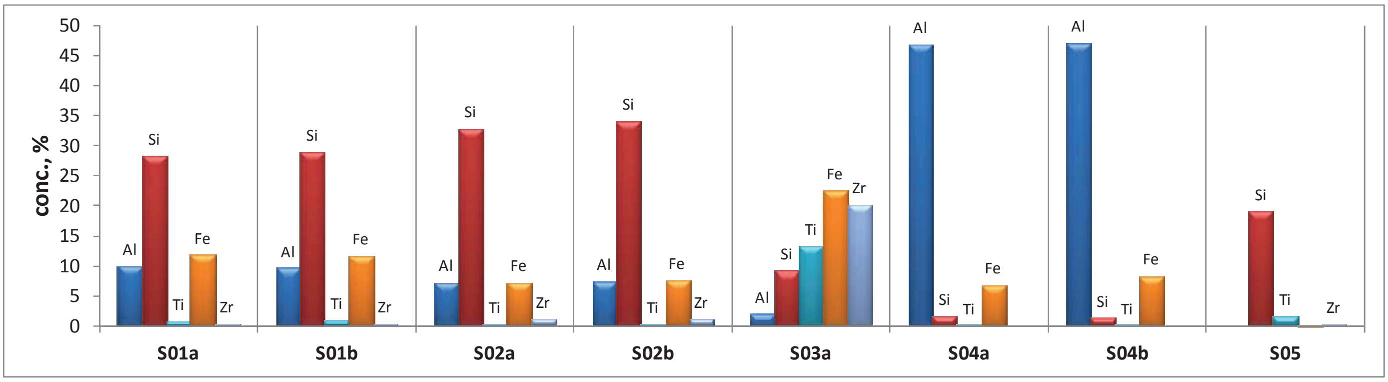

Fig. 1. Material composition of the scalar energy infused products. Samples S01 to S04 are Quantum pendants while sample S05 is an energy bracelet/baller. S01 and S02 samples show similar composition, primarily made of Si, Al and Fe; the S03 sample has a predominance of Fe, Zr, Ti and Si; the S04 sample is primarily made of Al while S05 is made of silicone rubber. (a, front of pendant while b, the reverse side)

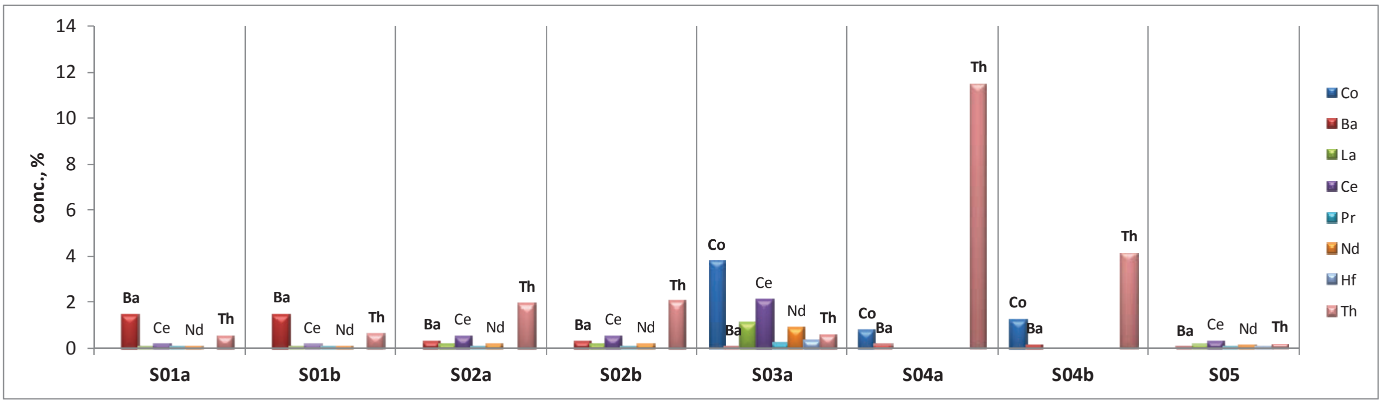

Fig. 2. Levels of NORMs and REEs in scalar energy infused products (I). Thorium (Th) is found in all the samples but is highest in S04 sample. Only the S03 and S04 samples contain Cobalt (Co) with higher levels in the former.

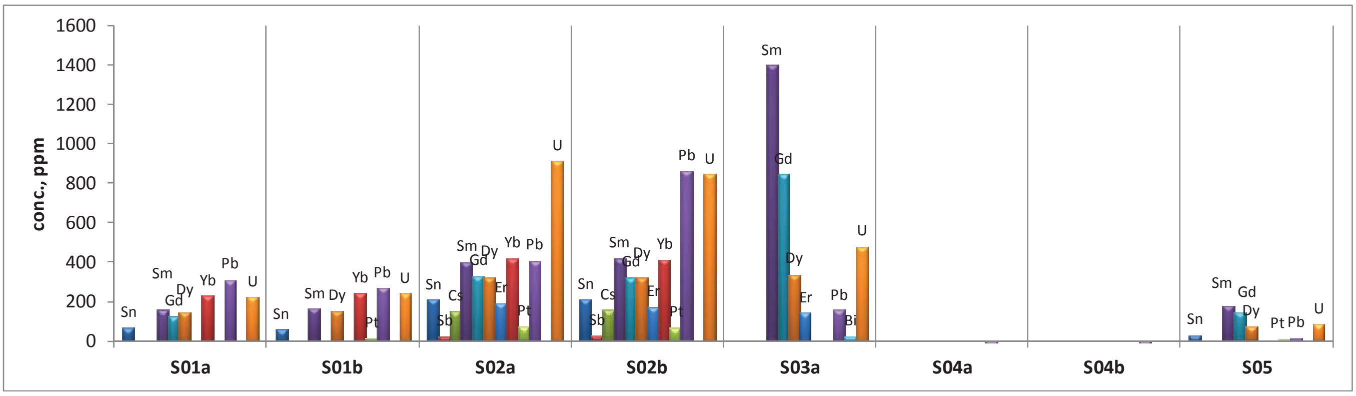

Fig. 3. Levels of NORMs and REEs in scalar energy infused products (II). Uranium (U) is found in all the samples except in S04. A plethora of REEs are contained in all the samples except in S04.

Do claims of power from these scalar infused products emanate from the NORMs these contain, similarly to Spider Man getting his superhuman power from an accidental bite from a radioactive spider? Is a little bit of the NORMs beneficial? XRF revealed the secrets of scalar energy infused products. As to the beneficial effects, this is another matter which might be worth exploring.

Publications of potential interest to the XRF community

1. Nuclear Techniques for Cultural Heritage Research, IAEA Radiation Technology Series, No. 2, IAEA, 2011
2. Proceedings of the National Conference on X ray Fluorescence, PRAMANA Journal of Physics, Vol. 76, No. 2, 2011, Indian Academy of Sciences – Springer.

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