

Nuclear Science and Instrumentation Newsletter

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Editorial

The Nuclear Science and Instrumentation Laboratory (NSIL) is pleased to bring to your attention the second issue of its Newsletter!

Since the release of <u>Issue Nr 1</u>, a significant progress has been made in the development of online resources to strengthen the information exchange with laboratories of IAEA Member States, including the creation of online databases of different types of nuclear instrumentation facilities, the increase of training materials (introductory courses, video demonstrations and lectures), all available from the *Nuclear Science and Instrumentation Portal* (<u>nucleus.iaea.org/sites/nuclear-instrumentation</u>).

In-house adaptive research in the last six months includes the assessment of capabilities of Grazing Emission XRF for the study of layered materials and the finalisation of a cooperation project for Rapid Environmental Mapping in support of the Fukushima Prefecture.

A significant progress towards the commissioning of the Neutron Science Facility in Seibersdorf has been made, including the optimization of the shielding for the D-D generator, the evaluation of capabilities for Dual Neutron/X-ray radiography and the commissioning of a Bonner Sphere spectrometer.

This Issue also includes a summary of the improvements brought to the Proficiency Testing scheme and the results of the last proficiency test.

Contributions from Austria, Czech Republic, Hungary and Morocco are presented in the section of Reports from Member States. LAREA Provide Anno Design Anno Anno Design Anno Design

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NSIL staff (from left to right): Mr Danas Ridikas (Section Head), Ms Ragdaa Attia (Team Assistant), Mr Jacopo Orsilli (Consultant), Mr Marco Magrin (Intern), Mr Natko Skukan (Associate NI Specialist). Mr Mladen Bogovac (Senior Laboratory Technician), Ms Pingping Gan (Intern), Mr Petr Sladek and Ms Haifa Ben Abdelouahed (NI Specialists), Mr Farhad Memarzia (Electronics Technician), Mr Alessandro Migliori (Senior Laboratory Technician), Mr Roman Padilla (NI Physicist), Mr Steven Mellard (Ass. Project Officer), and Mr Milan Matos (Senior Laboratory Technician, Nuclear Science).

NSIL looks forward to receiving both contributions and feedback from different Member State counterparts and stakeholders to this Newsletter, to help the laboratory to best continue supporting projects, fellowships, scientific and technical visits, and addressing research & development needs by national facilities worldwide.

1. Laboratory Projects and Activities

1.1 X-ray Fluorescence

Development of a Worldwide Database of XRF Laboratories

Currently X-Ray Florescence (XRF) facilities are present in almost every country, but for many their capabilities are yet not well-known by the wide community of end-users that might benefit from the advantages of this powerful analytical technique. Although several national and regional societies and networks exchange information to strengthen the cooperation among the XRF communities, no comprehensive repository of information on facilities available worldwide is yet available, that might increase their visibility and provide end-users necessary information to identify a potential partner for interdisciplinary research projects or a provider of analytical services.

In the Physics Section, we have created and are curating several databases and interactive maps depicting different nuclear research and instrumentation facilities operating worldwide. The youngest of these databases is the <u>worldwide Interactive Map of XRF Laboratories</u>, which offers information on more than 890 XRF facilities located in 111 countries. This database provides a broad overview of laboratories employing XRF techniques for fundamental or applied research, analytical services as well as for education & training purposes. The collected information is compiled either directly from the organizations operating these facilities by using an <u>online registration</u> form, or it is gathered from different publicly accessible sources.



Figure 1: Worldwide database (interactive map) of XRF laboratories

The database allows visitors to make customized searches by country, main activities, type of analysed samples and other key categories. For more information, please visit our <u>Nuclear Science and Instrumentation Portal</u>.

Angular Scanning XRF for the Analysis of Layered Samples

NSIL, in collaboration of the University of Milano-Bicocca (Italy), has upgraded its micro-beam multipurpose XRF spectrometer to perform the analysis of layered Cultural Heritage (CH) samples. This has been achieved following the approach suggested by Fiorini et al. [1], which is close to the one employed for Grazing Emission (GE)-XRF analysis. The sample is irradiated perpendicularly to its surface, and the fluorescence radiation is collected at small observation angles relative to the surface. The detector must be well collimated, down to angles of collection limited to tenths of milliradians, in order to obtain surface layer information.. As the detector is rotated around the grazing angle relative to the sample surface (or the sample is rotated with respect to the detector), the fluorescence radiation emitted from different layers composing the sample is collected.

Typically, GE-XRF is better suited for the analysis of thin layers (in the few nanometre range), whereas thicker layers in CH samples are often of interest, on the order of a few micrometres to a few millimetres. Furthermore, CH samples often have a complex composition, with many elements in low concentration or at trace level. Therefore, to enhance the collection of the fluorescence signal for such samples, the setup was designed to allow for larger angles for detection, which are preferred over strong collimation and small angular range used in standard GE-XRF geometry.

The spectrometer is equipped with a Mo-anode source with a power of 3kW (whose beam is focused by a monolithic glass polycapillary lens) and two Silicon Drift Detectors (SDDs). One detector is equipped with a polycapillary conic half-lens and is used to perform Confocal-XRF (SDD-1), whereas the second one (SDD-2) is simply collimated and is employed to perform standard micro-XRF analysis. As the spectrometer does not allow to move the detectors (except for fine adjustments for the correct positioning), an angular scanning can be carried out only by rotating the sample. To perform the analysis of the layered samples, SDD-2 was further collimated with a vertical slit of stainless steel with an opening of nearly 60µm and a thickness of 600 µm (Figure 2). The slit is aligned with the axis of rotation of the sample to maximise the intensity of the fluorescence signal without losing angular resolution. The distance between the sample and the slit is17mm and the angular resolution is of 4.5mrad.





Figure 2: Set-up of the multipurpose micro X-ray spectrometer.

We have completed initial test of the system by measuring laboratory-prepared metal samples (Table 1): five copper samples with a titanium surface layer of varying thickness (100 μ m of copper covered with 6, 12, 18, 25 and 30 μ m Ti) glued onto the copper substrate with a thin layer of low density, low-Z glue.

Sample	Description	Ti Mass Thickness [mg/cm3]		
Т0	100 μm of copper and 6 μm of titanium	27.24		
T1	100 μm of copper and 12 μm of titanium	54.58		
T2	100 μm of copper and 18 μm of titanium	81.72		
Т3	100 μm of copper and 25 μm of titanium	113.5		
T4	100 μm of copper and 30 μm of titanium	136.2		

Table 1: Description of the analyzed samples

The surface layer mass thickness was determined by leastsquares fits of the experimental angular fluorescence profiles to theoretical profiles, correcting for the absorption of titanium and gold layers of different thicknesses, calculated using the Fundamental Parameters (FP) method [2], showing that the experimentally determined mass thicknesses from the angular fluorescence analysis are in good agreement with the real thicknesses of the samples, as illustrated in Figure 3.



Figure 3: Fluorescence angular profile of copper for the titanium-copper samples, the curves represent the best fit of the data, obtained using the measured values in the legend.

References

- C. Fiorini, A. Gianoncelli, A. Longoni, F. Zaraga, Determination of the thickness of coatings by means of a new XRF spectrometer, X-Ray Spectrometry. 31 (2002) 92–99. <u>https://doi.org/10.1002/xrs.550</u>.
- [2] B. Beckhoff, B. Kanngießer, N. Langhoff, R. Wedell, H. Wolff eds, Handbook of Practical X-Ray Fluorescence Analysis, 2007 Springer Science & Business Media.

1.2 In-situ Radiation Monitoring

NSIL has long-standing experience in development and application of instrumentation techniques for in-situ measurements, especially mobile monitoring such as field portable gamma spectrometers mounted on backpacks or unmanned aerial vehicles (UAVs).

Radiological mapping is a challenge for many situations such as environmental monitoring, site characterization in the case of remediation activities and radiation protection as well as nuclear security. Modern in-situ techniques can offer tailored solutions for all those situations.

Support to NORM Project

Radionuclides from the U-238 and Th-232 decay chain and K-40 occur naturally in the environment in different matrices. These materials belong to a class of radionuclides referred to as Naturally Occurring Radioactive Material (NORM). Different industrial activities have the potential to increase the activity concentrations of these radionuclides in residues generated in the operational process, leading to a potential increase in human exposure to ionizing radiation. The number of industries that generate NORM residues is large, including mining and mineral processing, other resource extraction activities, coal combustion, oil and gas operations, and geothermal energy and water treatment plants, among others. The IAEA NORM Conference in October 2020 gave visibility to the efforts and progress of many countries towards the safe management of NORM. Previously, IAEA launched the Environet NORM Project, well-aligned with the needs of the Member States, giving emphasis to sustainable solutions to the management of NORM residues in the context of the circular economy.

One of the more compelling challenges identified by this project is how to efficiently characterize different NORM residues. Such characterization is needed not only to evaluate the radiation hazards, but also to determine the inventory of these materials that in turn will guide management strategies. Of great relevance is how to develop, implement, and harmonize procedures for sampling and analysis of these materials.

NSIL supports the work of a dedicated Working Group created to compile and share good practices and technologies in the form of knowledge transfer, obtaining practical experience, and implementing best practices related to representative sampling and characterization of NORM products.

Project on "Rapid Environmental Mapping with Unmanned Aerial Vehicle (UAV)" in support of Fukushima Prefecture

The cooperative project between NSIL and Fukushima Prefecture (FP) focused on development and utilization of modern Unmanned Aerial Vehicles (UAV) technology for radiological mapping and was conducted in 2012-2020. Several activities were carried in the framework of two consecutive projects NA9/2 and NA9/3 on "Rapid Environmental Mapping with Unmanned Aerial Vehicles (UAV)".

An NSIL-developed instrumentation and methodology for UAVs equipped with radiation detectors, camera, and GPS has been tested and validated under real conditions in the Fukushima Prefecture in Japan (Figure 4).



Figure 4: Illustration of exponential dependence of the dose equivalent rates measured by the UAV-based system and represented as a function of altitude. Picture credit: the Fukushima Prefecture.

The basic lightweight detection module equipped with GM detectors, as well as gamma spectroscopy module coupled with high resolution scintillation LaBr3(Ce) detector, were tested in real conditions of contamination for dose rate measurement and potential spectroscopy radionuclide identification.

Nine areas of public importance or use as temporary storage sites (TSS) for contaminated soil were assessed by UAV monitoring. These areas with different levels of radiation contamination were located 4 to 68 km away from the TEPCO Fukushima Daiichi NPP, as reported in Newsletter No. 1 (February 2021).

The excellent cooperation on this project led not only to the development and optimization of the UAV based radiation monitoring system but also to other related activities associated with this technology. For example, NSIL has designed and produced its own digital pulse processing electronics with Multichannel analyser and fast signal processing using FPGA. The use of modern scintillation SiPM detectors was continuously explored and tested. The photogrammetry was incorporated and verified for the possibility of creating orthophoto maps of areas or 3D models of objects for data presentation over actual situation. Also, a parallel use of UAV and backpack technology within the project led to better verification of results and the possibility of comparing ground and airborne results. The optimization and verification of developed methodology for measuring and evaluating the results became a very important part of this project. To this end, full-scale Monte Carlo modelling was used for the recalculation of dose rate at the ground surface, which was verified and later implemented into the methodology of measurements.

The UAV-based radiation monitoring technology is now available for practical use in routine or emergency situations. In many cases, this technology has shown its advantages over conventional monitoring approaches in areas that are not accessible on foot or where high radiation levels might exist. The encouraging results of the project and response of the Member States to the possibilities of using UAV technology for radiological mapping commit NSIL to follow current trends and continue with development in this area. NSIL is in communication with several counterparts in China, Malaysia, and Singapore, to mention a few, and to address their current needs and recommend technical solutions for the use of UAV technology in a number of specific areas of radiation monitoring. Another example of NSIL's support was the procurement of the UAV-based gamma spectrometer under the IAEA TC Project GEO9016 "Improving Regulatory Oversight and Response Capabilities".

1.3 Accelerator Technology and Applications

IAEA Conducts First Virtual Training on Using Ion Beam Techniques and Applications

Ion Beam Analysis (IBA) techniques are widely used as nondestructive nuclear analytical methods, which rely on beams of low energy ions (charged particles) generated by electrostatic accelerators. These beams bombard the samples to be analysed, where they interact with the electrons and the nuclei of the sample material and produce X- and gamma-rays, or even new particles.

Physics Section support the use of IBA techniques through organizing training workshops, facilitating access to the facilities operated by partner organizations, and offering advisory services and review missions. The lack of training opportunities during the pandemic is affecting the professional development of the next generation of nuclear scientists. This is particularly true for skills requiring hands-on training to excel in IBA techniques, which are impossible to acquire remotely.

The IAEA and Croatia's Ruđer Bošković Institute (RBI) conducted the very first live virtual training workshop on advances in IBA techniques and their applications, to minimize the impact of training shortages caused by the COVID-19 pandemic. The five-day course focused on IBA techniques and attracted post-graduate students and young professionals from 16 countries. The online training (Figure 6) was attended by 36 (17 female and 19 male) young professionals, which is almost four times the number of participants the IAEA can accommodate during in-person trainings on IBA.

The course included lectures and demonstration videos of typical ion beam experiments relevant to environmental studies, biology, cultural heritage, forensic science, and materials research. The data collected during these experiments was sent to participants for practice in data analysis and interpretation. The training course also included a virtual tour to the RBI accelerator facilities. All demonstration videos from the training course are available on this link: <u>https://nucleus.iaea.org/sites/accelerators/Pages/IBA-video-demonstrations.aspx</u>

The reactions of the trainees were very positive regarding the organization and topics of the training. All found it useful, even though is evident that the hands-on part of the training can be fully utilized only in physical (in-person) training courses.



The next workshop, again to be organized in cooperation with RBI, will be held virtually in November 2021 and will feature accelerator technology and associated instrumentation, including operation and maintenance aspects of ion beam accelerator facilities. More information on how to participate can be found under this link:

https://www.iaea.org/events/evt1904154

1.4 Neutron Science and Applications

NSIL is currently establishing a Neutron Science Facility (NSF) based on two compact neutron generators: Deuterium-Deuterium (DD), resulting in 2.45 MeV neutrons, and Deuterium-Tritium (DT), resulting in 14.1 MeV neutrons, with maximum source intensities up to $1x10^7$ n/s and $4 x10^8$ n/s over 4π , respectively.

Shielding Optimization for D-D Neutron Generator

The NSIL's D-D neutron generator is a portable system, designed for laboratory or field applications, comprised of the miniature accelerator assembly and the electronics enclosure. It requires low power consumption and may be powered from battery, vehicle power sources, or standard AC line voltages. It provides pulsed (from 250 Hz to 20 kHz) or continuous output.

Having no electrical charge, the generated fast neutrons are deeply penetrating. As a result, shielding against neutron radiation, when compared to photons and electrons, is more difficult to realise. Therefore, a design and construction of an adequate Operation Housing Module (OHM) for our DD generator is of key importance in ensuring safe working conditions for personnel as well as for NSF users. Due to neutron interactions with nuclei - via elastic and inelastic scattering, capture, charged particle production, and nuclear fission - neutrons reveal a complex absorption cross section pattern in terms of neutron energy and atomic number of the traversed material. Consequently, materials containing low atomic number elements (H, Li, C, B, Al) with high scattering or absorption cross sections are among the most suitable for attenuation and shielding of both low and fast neutrons.

Based on a series of optimization studies using full scale Monte Carlo simulations, the planned OHM of the NSIL DD neutron generator will be composed of a 30 cm thick highdensity polyethylene (HDPE) moderating-shielding material. A 10 cm diameter aperture, so-called beam port, can be opened on one side of the OHM. The centreline of the aperture is positioned in a way that it intersects the neutron source location. The OHM is placed on a supporting base, also made of HDPE.

Practical aspects of setup, ease of use, and safe handling, as well as flexibility and extendibility features are considered in designing the NSIL OHM. Thus, the entire module with its support consists of thirteen (13) interlocking HDPE blocks, all supplied with handles, and the weight of each piece not exceeding 58 kg – still portable by two persons when needed, as illustrated in Figure 5.



Figure 5: Technical drawing for the design and 3D assembly of the OHM, hosting neutron generator.

The Beam Stop (BS) is an inseparable part of the DD OHM and is composed of a 25 cm thick HDPE board, 64x55 cm² on the side facing the beam port and 10 cm thick walls on the four sides around the neutron beam. The centreline of the BS cavity is positioned in front of the beam port so that it faces the neutron source emission. The BS is placed on a supporting base with same height and made also of HDPE (Figure 6).



Figure 6: MCNP 3D geometrical model of the DD neutron generator shielding system.

Both OHM and BS modules were designed and optimized by the NSIL team, using the MCNP6.2 Monte Carlo code by simulating monoenergetic and isotropic point source of 2.45 MeV neutrons, representing the DD neutron generator. Combined neutron-photon transport calculations were performed using active "PHYS N P" card with a history of 10⁷ incident neutrons. Dose rates were calculated using energydependent quality factors from the ICRP 21 (1971), and which were applied using the "DF" card in the MCNP6.2 input file. The total equivalent ambient dose (TEAD) values then were calculated using the neutron-photon fluxes tallied by sixteen (16) point detectors and by multiplying these values by the energy dependent quality factors. The point detectors were positioned inside and around the neutron facility premises. Nine (9) detectors were placed to tally combined photon and neutron TEAD around the outside of the OHM, including the BS. The other seven (7) detectors were placed at locations of interest, i.e. where radiation doses rates are relevant (at the entrances and exits, windows, and walls opposite to the beam port). The results of calculated TEADs at different locations are overlaid their respective positions in Figure 7.



Figure 7: Illustration of TEAD rates around the DD OHM and BS (left and right) and around the facility building (right). All units are in μ Sv/h.

With the beam port of the OHM closed, all estimated TEAD rates inside the neutron hall are below 5 μ Sv/h. With the beam port of the OHM open, the estimated TEAD in the BS, at one meter from the OHM, approaches 100 μ Sv/h. All other estimated TEAD rates in the facility premises are again

below 5 μ Sv/h. TEAD rates outside the facility premises are all below 0.5 μ Sv/h. The presence of the BS module greatly reduces the contribution of neutrons and photons to the surrounding total radiation dose and, therefore, the TEAD rates fully comply with recommended radiation protection regulations.

Experimental values of the TEAD surrounding the DD neutron generator shielding system are planned at the commissioning stage, which will ensure that they are consistent with the values predicted by our MCNP calculations. This experimental validation will provide credibility to our MCNP model built for the specific purpose of TEAD calculation to optimise shielding design and ensure radiological safety. Later the same code will be used to model experiments and optimized utilization of the facility, including for training purposes.

Progress of a Dual Neutron/X-ray Radiography and Tomography System

The NSF facility will also be equipped with a dual X-ray/ neutron radiography and tomography system, which consists of a sample/object rotation platform, a scintillator, a CCD camera, and a computer. While the DD and DT generators are not yet commissioned, the first tests of the radiography and tomography system were performed using a 30 kV Xray generator.

In Newsletter No. 1 in Feb 2021, we reported the commissioning of the X-ray radiography system and obtained 2-dimensional projection images. Since then, we have worked on measurements of a spatial resolution of the X-ray radiography system as well as X-ray tomography experiments. Generally, there are two different test samples to determine the spatial resolution of a radiograph: test patterns that indicate the resolution directly and items to determine the sharpness of a high contrast edge.

In the framework of a dedicated IAEA Round Robin exercise, a set of samples has been prepared and made available to determine the spatial resolution of radiographs and tomographs, mainly for research reactor-based neutron radiography facilities. The sample for radiography resolution, called slanted edge, is made of a Gd sheet with two polished edges mounted in a frame for convenient positioning. The modulation transfer function (MTF) describes the magnitude response of an optical system to sinusoids of different spatial frequencies and can be used to evaluate the ability of the imaging system or its components in reproducing fine detail. By analyzing the radiography image of the slanted-edge sample, edge profile can be obtained. The derivative of the edge profile yields the line spread function (LSF), and after applying a smoothing Hamming window to the LSF, performing a discrete Fourier transform, and normalizing, the MTF over a range of horizontal spatial frequencies can be estimated. Radiographic image analysis provides an estimate of the resolution. Using the slanted edge sample (Figure 8), we measured the spatial resolution of our X-ray radiography system to be about 0.1 mm at contrast MTF of 20%.



Figure 8: The experimental setup of the X-ray radiography resolution measurement (left), the radiography image of the slanted-edge sample (middle), and the modulation transfer function (MTF) calculated from the edge region in the measured radiography image (right).

A "homemade" sample was used during the commissioning of the X-ray tomography system (Figure 9). The sample was composed of five one-EURO coins, one metallic cylinder, one screw and one cup filled with potato powder.



Figure 9: The X-ray tomography experiment setup (up left), the prepared sample cup (up right), the reconstructed 3D image (down left), the 3D image of the metal items inside the sample cup (down right).

We recorded 361 radiography projections over a full 360degree rotation, four dark current pictures, and four open beam pictures. The whole tomography experiment took a round 10 hours to collect. All these projections were processed with the reconstruction software Muhrec to get 2D tomographic imagesradiographs. Two preprocessing modules were used for prior to the reconstruction: FullLogNorm and ProjectionFilterSingle. FullLogNorm corrects the projection images with open beam and dark current images. ProjectionFilterSingle performs the filtered back-projection algorithm, which applies a ramp filter combined with a Hammingn apodizing window on the projection data. One processing module named MultiProjBP was used to reconstruct. MultiProjBP provides the filtered back projection algorithm for the reconstruction. In the final step, 2D projections tomographic images were imported to image processing software ImageJ to obtain a 3D image view. From the 3D image view, we can clearly see the screw thread and discern ridges on the sides of the stacked coins.

The X-ray radiography and tomography system showed good imaging capability and, after the commissioning of the DD and/or DT generators is complete, the radiography and tomography system will be implemented with neutrons.

Commissioning of Bonner Sphere Spectrometer

The NSIL purchased a Bonner sphere spectrometer to determine the energy spectrum of neutrons in a variable neutron field. The operational principle of Bonner sphere spectrometer is detection of thermal neutrons in the centre of several moderating spheres of different sizes (see Figure 10).



Figure 10: The Bonner spectrometer consists of twelve polyethylene spheres with diameters from 3 to 15 inches.

The spheres are manufactured from polyethylene (PE), rich with hydrogen. Each sphere, depending on its diameter, will most efficiently thermalize neutrons of a specific energy range. In the centre of the sphere a thermal neutron detector is placed, in this case He-3 counter, sensitive to low energy (thermal) neutrons with a good discrimination to other signal generating probes (gammas in this case). During the experiment over a defined period of time, a number of counts is obtained for each sphere. Through a series of iterative or deconvolution operations, the data is analysed to provide the most probable shape for the energy spectrum of the measured neutron field. The RUFUS software provided by ELSE, the manufacturer of the Bonner sphere spectrometer, is used for this.

NSIL staff commissioned and tested this spectrometer using a Californium (Cf-252) and an Americium-241-Lithium (Am-Li) neutron sources. The energy spectra of Cf-252 and Am-Li neutron sources are generally well known and can be found in the literature [3]. The measurements conducted during commissioning of the spectrometer yielded results close to the expected values are depicted in Figure 11.



Figure 11. Unfolded experimental spectra for Cf-252 neutron source.

NSIL will use the Bonner spectrometer for energy characterization of the output of the DD and DT neutron generators after their commissioning. It will then be an important building block in NSIL's continuous efforts to deliver high quality training to scientists and engineers from the IAEA Member States. For example, this new instrument will be used to develop different experimental exercises as a part of the training in the use of neutron generators and associated instrumentation.

References

[3] IAEA Technical Reports Series No. 403 Compendium of Neutron Spectra and Detector Responses for Radiation Protection Purposes (2001).

1.5 NSIL Capacity Building Activities

Due to restrictions posed by the COVID-19 pandemic, inperson NSIL training activities, from basic courses to advanced and specialized trainings, in nuclear instrumentation have not been possible since March 2020. NSIL staff have developed several online alternatives to counteract these difficulties, including e-learning modules, lectures, and video practical demonstrations.

Detailed information on the training topics offered by NSIL, as well as the online access to these materials is provided in the <u>Nuclear Science and Instrumentation Portal</u>.

E-learning Introductory Courses

Since March 2020, Physics <u>e-learning courses on Nuclear</u> <u>Techniques</u>, hosted in the IAEA CLP4NET system, were followed by more than 1000 users (Figure 12).



Figure 12: Summary of attendance in 2020 to the e-learning courses

Currently, the e-learning modules available include:

- Introduction to Electrostatic Accelerators: from basic principles to operation and maintenance
- Introduction to In-situ Techniques for Radiological Characterization of Sites
- Introduction to X-ray Emission Spectrometry
- Quality Assurance of X-ray Fluorescence Analysis of Airborne Particulate Matter
- Introduction to Total Reflection X-ray Fluorescence
- Portable X-ray Spectrometry Techniques for Characterization of Valuable Archaeological/Art Objects
- Neutron Activation Analysis
- Neutron Imaging
- Nuclear Analytical Techniques for Forensic Science.

The last course on the list is now available both in English and Spanish. Three new additions were also incorporated into the existing modules: on XRF, on XRD and on Sample preparation for PIXE analysis.

Online Video Recordings and Lectures

The Nuclear Science and Instrumentation Portal has a section on <u>Training</u> that serves as a repository of various online materials. Recently, five lectures were added on the use of portable radiation detectors and in-situ techniques:

- Radiation monitoring using portable detectors
- In situ measurements and mapping to support radiological characterization of sites
- Utilization of HPGe detectors for Nuclear Security tasks (Introduction)
- Characterization of objects contamination using portable devices
- Recommendations for radiological characterization of different objects and cases



Figure 13: Example of one of the lectures available as MP4 movies recorded from MS PowerPoint presentations

Four practical demonstrations on XRF methods, recorded as videos, are also available:

- Preparation of solid samples for XRF analysis
- Handheld XRF analysis
- Total Reflection XRF
- XRF analysis of soil and sediment samples
- WDXRF analysis of Organic samples



Figure 14: The practical demonstrations on XRF methods are available as MP4 movies with the possibility of activating captions in English.

In the forthcoming months, a series of lectures on XRF topics will be released, as well as another four practical demonstrations on both the use of portable detectors and in-situ techniques and advanced XRF techniques for assessing the elemental spatial distribution in samples.

1.6 Proficiency Tests Organized by NSIL

The number of laboratories interested in joining the NSIL organized proficiency tests (PTs) continues to increase, since the first exercise offered in 2002. Figure 15 depicts the number of participating laboratories, including information on the region of participating laboratories for every NSIL PT. .



Figure 15: Number of participants to the proficiency test organized by NSIL, including the information on the region where the laboratory is located.

The type of analytical techniques involved in PT exercises varies according to the purpose of the test. Some PTs were open only to X-Ray Fluorescence spectrometry (XRF) or Neutron Activation Analysis (NAA) laboratories; conversely, most PT exercises had no restriction to the type of nuclear and related analytical techniques is applied, in order to allow maximum number of interested laboratories to participate.

Upgrade of the NSIL Proficiency Test Scheme

Compliance with ISO 17043

Since 2015 NSIL PTs comply with ISO 17043:2008 [4] concerning the organization and terminology adopted when running an interlaboratory exercise.

Starting in 2018 organization of NSIL PTs uses a dedicated website (<u>www.pt-nsil.com</u>), which simplified all operations related to implementation of the exercises for both the coordinator and the participating laboratories.

Compliance with ISO 13528

Since the last exercise PTNATIAEA18 in 2020, the data of the NSIL PT are processed in compliance with ISO 13528:2015 [5]. Until PTNAAIAEA17, evaluation of laboratory performance was based on determination of their *z*and *u*-scores, which were reported with fully anonymity for all the laboratories in the final report, along with criteria for satisfactory, questionable, or unsatisfactory performance results. In this case, consensus values were determined as arithmetical averages of data, after outlier rejection, whereas standard deviations were determined using a modified Horwitz function.

Since 2020, the statistical approach has been upgraded according to ISO 13528. The main changes implemented can be summarized in the list below:

- The consensus value of the participants' results and relative standard deviation is determined with robust statistics (Algorithm A)
- Depending on the uncertainty of the assigned value, *z*-scores or *z*'-scores are considered
- *u*-scores are replaced by *Zeta*-scores
- The definition of "outliers" is different; also, the term "blunder" is used to identify extreme outliers
- The three times standard deviation of the assigned value is no longer applied as a level of fitness for purpose.

Other functionalities were added in the NSIL PT database, including the possibility of easily extracting different graphs and statistical information, including, among others, performances of laboratories depending on analytical technique and/or on time and geographical distributions of participants.

Results of the last exercise PTNATIAEA18

The last exercise PTNATIAEA18 complied with ISO 13528. This PT was impacted by the critical situation related to the COVID-19 pandemic; especially challenging were the distribution of samples and limited access of some of the participants to their laboratories. The PT was announced on 5 February 2020. The samples were prepared for 103 participating laboratories by August 2020. The deadline for submission of the results was 28 February 2021, after postponements related to COVID issues. The <u>final report</u> was uploaded to the NSIL-PT dedicated website on 14 May 2021.

The test sample was a sandy soil prepared and tested by an external provider through an independent inter-laboratory survey. The powdered, homogenized, and dried material was prepared in plastic bags, each containing around 40 g of material. The test samples were distributed to 97 laboratories. Problems in delivering the parcel to the final consignee occurred in the remaining six laboratories out of those 103 registered. Of the 97 laboratories that received samples, 70 from 44 Member States managed to submit 1455 individual results for quantification of 65 chemical elements before the deadline.

Figure 16 represents the proportion of results submitted according to the analytical techniques used. Most of the analyses were carried out either by NAA techniques (about 48%, red/orange and salmon pink in Fig. 19) or by XRF spectrometry (about 46%, shades of blue). Particle Induced X-ray Emission (PIXE) results accounted for 1.3% of the total number of results, Atomic Absorption Spectrometry (AAS) techniques for 1.4% (shades of green), and Inductively Coupled Plasma Spectrometry (ICPMS) techniques for 3% (shades of brown).



Figure 16: Analytical techniques, number of associated submitted results out of 1455 in total, and their proportion in PTNATIAEA18. The percent values relate to the total number of 1455 submitted results.

The correlation between the values from the external provider and the consensus values is shown in Figure 17.



Figure 17: Correlation between the values from the external provider and the consensus values. The error bars are the standard deviations from the external provider and the participant standard deviations.

Figure 18 illustrates, as an example, the distributions of the PT results for the element rubidium, Rb. The individual results are marked with coloured circles (blue XRF, red NAA, and grey for other analytical techniques. Blunders are not shown.). The density distribution curve for all results (excluding the blunders) is shown by the solid black line. The vertical dotted black lines show the range of non-outlier results. The assigned value is show as vertical solid green line, whereas the range ± 3 standard deviations is shown by vertical dotted green lines.



Figure 18: Density distribution function for Rb. See text for details.

Figure 19 represents the results in a bar chart form, sorted in ascending order versus participant (bottom) and technique (top) code. Three times the standard deviation is indicated by uncertainty bars.



Figure 19: Bar chart for Rb results in Fig. 21. See test for details.

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2. Support to Technical Cooperation Projects

NSIL staff provide technical backstopping and support for more than 40 Technical Cooperation (TC) projects, particularly in the area of "Capacity building, human resource development and knowledge management", "Marine, terrestrial and coastal environments", "Radioisotopes and radiation technology for industrial, health-care and environmental applications", "Accelerators" and "Nuclear Instrumentation".

For the development of concepts and designs for IAEA TC projects, support is provided informally to individual organizations considering proposing projects to their National Liaison Officers (NLOs). More substantial assistance is available for Member State endorsed projects in the conceptual design and design review phases.

The restrictions imposed by COVID-19 pandemic have forced to find solutions to implement some of the activities in virtual mode. Some examples of these activities are provided below.

RAS1025: Enhancing the Capabilities of Radiocarbon Dating in Archaeological Applications (ARASIA)

Seminar on the Treatment Methods Applied to Sample Preparations for Carbon-14 determinations Virtual event, 3 September 2020

Arab States in Asia are very rich in archaeological patrimony, and numerous findings are still being collected from excavations. Often artefacts are preserved by different institutions without having reliable dates that define the period or era to which they belong. These need to be studied, in order to reconstruct the ancient human history. The main part of these artefacts are organic remains such as bones, charcoal, seeds and similar, which are conducive to carbon dating The ARASIA (Cooperative Agreement for Arab States in Asia for Research, Development and Training related to Nuclear Science and Technology) project RAS1025 "Enhancing the Capabilities of Radiocarbon Dating in Archaeological Applications" aims to enable participating countries to perform dating of their artefacts through collaboration among various Member States and through capacity building and training. Within RAS1025 a virtual "Seminar on the Treatment Methods Applied to Sample Preparations for Carbon-14 determinations" was held on 3 Sept 2020, attended by 21 participants representing 10 ARASIA countries. The seminar included the following topics:

- Radiocarbon dating (principles, basics, importance, QC),
- Sample contamination,
- Sample treatment methods (especially for bones and charcoals),
- Burial environment effects on sample preservation for radiocarbon dating,
- Characterization of collagen (bone samples suitable for dating, preservation of collagen-quality and quantity),
- Applied techniques to characterize collagen,
- A brief overview of data treatment.

As an outcome of this seminar, a document "Pre-Treatment Protocols and Characterization of Samples for Radiocarbon Dating" was prepared and shared within the counterparts of the project.

RER7012: Determining Long Term Time Trends of Air Pollution Source Tracers by Nuclear Techniques

Task Force Meeting to Compile Results on Identification of Air Pollution Sources, Virtual event, 20-30 October 2020

To address the high level of air pollution in Europe, several cycles of successful IAEA regional projects focused on studying air particulate matter pollution in Europe have been conducted. The ongoing regional project RER7012 has the objective to continue monitoring the concentration of atmospheric fine fraction of particles (PM2.5), to improve the accuracy of determining their elemental and chemical profiles, and enhance the usefulness of the available data. Local and regional air pollution sources in each study area will be identified using receptor modelling, specifically factor analysis via positive matrix factorisation (PMF) and comparison to available source profiles established across the region.

A virtual Task Force Meeting was organized to discuss the results obtained after monitoring the concentration of PM2.5 particles during 2016-2019, as well as identification of sources. The meeting was attended by 24 participants representing 19 countries. At the meeting, results of PM2.5 concentrations were compared for working days and weekends across the region as illustrated in Figure 20.



Figure 20: Box and whiskers plot of the reported concentrations of PM2.5 (mg/m3) and exceedances of the EC yearly average limit (red dashed line).

Further, participants extensively discussed the observed seasonal variations and the estimated inventory of source contributions (see e.g. results reported by one country in Figures 21 and 22).

Benefits from the monitoring and task force meeting were exchange between project partners having less experience with those having extensive experience in the field, offering an opportunity to harmonize the interpretation of results across the region.



Figure 21: Seasonal variations in PM2.5 concentrations reported by Croatia.



Figure 22: Main PM2.5 sources identified and their relative contribution (reported by Croatia)

3. Reports from NSIL Events

Consultancy Visit on In-situ Application of Portable HPGe Detectors

The consultancy visit of Mr Branislav Stribrnsky was focused on preparation of material for the NSNS-NAPC joint Webinar on Estimating Activity in a Package Using an HPGe Detector.

The work included preparation of slides and videos on introduction of the hardware and software, system preparation, including energy and efficiency calibration. Measurements in the form of case studies included scenarios with box, drum, disc and point sources (see Figure 23), and were followed by results presentation and interpretation.



Figure 23: Gamma spectrometry measurements in different scenarios.

The developed training materials will be published and used in future trainings.

Webinar on Overview of Approach for Estimating Activity in a Package using a Portable High Purity Germanium (HPGe) Detector

Virtual event, 15 June 2021

As part of cooperative activities between NSNS and NAPC, a joint webinar on Overview of Approach for Estimating Activity in a Package Using a Portable High Purity Germanium (HPGe) Detector took place on 15 June 2021. The event was attended by more than 250 participants from 70 countries and focused on characterization of different objects, like containers, packages, as well as point sources typical for screening processes in nuclear security and other fields.

The primary objective of this event was to share information and provide guidance on applications of portable High Purity Germanium (HPGe) instruments in the field of nuclear security. The webinar also served to strengthen awareness and contribute to the capacity building within Member States to effectively utilize HPGe instruments and train others through Nuclear Security Support Centres (NSSCs) and other regional centres.

In brief, when a package or shipment container emitting radiation is discovered as part of a nuclear security surveyance activity (see Figure 24), specific data is needed to determine an appropriate course of action, including isotope identification and an estimation of the respective activities. In the field, identification of isotopes is most accurately performed using high purity germanium (HPGe) detectors. However, using a HPGe detector to estimate the total activity adds complexity to the use and analysis of the experimental data and may require additional software and hardware.



Figure 24: Measurement of different packages / containers.

The webinar provided an overview of the general approach to estimate activity in a package or container containing radioactive material, discuss the uncertainty in the estimation, and demonstrate the process using a typical HPGe detector and software. The event was aimed at users familiar with operation and use of portable HPGe detectors from expert and front-line organizations involved in the measurement and estimation of activity in a package that contains radioactive material. The experimental methodology and detection techniques were demonstrated in practical examples in video sequences, photographs, and diagrams suitable for online attendees. Webinar presentations are available through the following link: <u>https://nucleus.iaea.org/sites/nuclear-instrumenta-tion/Pages/Portable-detectors_videos.aspx</u>

Joint ICTP-IAEA School on FPGA-based SoC and its Applications for Nuclear and Related Instrumentation (smr3562)

Virtual event, 25 January – 19 February 2021

A (virtual) joint IAEA-ICTP school took place at the beginning of the year, covering key aspects of fully programmable Systems-On-Chip (SoC) technology and its applications to nuclear and related instrumentation. The main aim of the event was to familiarize participants with underlying software design tools and hardware platforms through tutorials and project examples in the field of nuclear applications.

Design tools and hardware platforms required for the practical exercises for the School's virtual format were prepared and installed on both the ICTP computers in Trieste and in the NSIL computers at Seibersdorf (also called virtual training rooms). The computers in Seibersdorf were also equipped with cameras.



25 participants from 20 countries and 12 time zones could login remotely onto the computers at both sites and perform laboratory exercises individually, according to written instructions. To facilitate the work, participants were divided into five virtual rooms and one tutor was assigned to each room. This allowed easier interaction between fellows and tutors as well as among the fellows themselves.

During the School's first two weeks, participants were familiarized with Xilinx Vivado FPGA design software tools and two FPGA hardware development platforms through practical tutorials. The last week was devoted to two practical projects implemented in parallel. The first project on particle detectors was carried out on ICTP computers with 15 participants divided into three virtual rooms. The second project on gamma radiation detectors was carried out on NSIL computers with 10 participants divided into two virtual rooms. The participants learned how to develop a FPGA firmware for processing pulses from radiation detectors. Upon successful completion of the projects, the trainees could remotely measure radiation spectra from related detectors and detector emulators available at MLAB and NSIL.

The training materials used for this School can be accessed via the ICTP site <u>http://indico.ictp.it/event/9443/</u> and via Gitlab link: <u>https://gitlab.com/smr3562.</u>

Joint ICTP-IAEA School on Citizen Science with Application to Nuclear, Seismic and Air Quality Monitoring (smr 3565)

Virtual event, 8 – 19 March 2021

Open Science aims to make scientific research, data, and evidence freely open and available to society. It also aims to increase scientific collaborations and sharing of information for the benefits of everyone. Within this framework, citizen science is a key element, which enables collaboration between societal stakeholders and the scientific community by opening up practices and tools that are part of the research cycle.

A joint IAEA-ICTP School held online in March focused on how to balance low cost scientific tools and academic rigor in their usage/application to citizen science in specific application areas. The School included an introductory week highlighting the scientific benefits of citizen science, its societal impact, and the latest tools and instruments. The second week was devoted to the development of low-cost nuclear radiation, seismic, and air quality sensors/detectors.



The event was attended by 92 participants (30 females) from 46 countries. More information is available from <u>https://www.ictp.it/about-ictp/media-cen-</u>tre/news/2021/5/citizen-science-workshop.aspx

4. Reports from Member States

Austria

Laboratory Portrait:

Atominstitut X-ray Lab

The X-ray lab is part of the research area Radiation Physics at the Atominstitut, faculty of physics at TU Wien, Vienna. Our focus is on the development of tailored EDXRF spectrometers for targeted applications using special techniques. We are not an application lab, but a development lab. Our expertise is TXRF, μ -XRF, GIXRF, confocal XRF, XRF of low Z elements and Synchrotron radiation induced XRF.

WOBISTRAX, a tabletop TXRF spectrometer in a vacuum chamber using dual band excitation for simultaneous determination of low Z and high Z elements (Na-U)

The WOBISTRAX TXRF spectrometer [1] is equipped with a 12-position sample changer, a 10-mm² silicon drift detector (SDD) with an 8 μ m Be entrance window and electrical cooling by Peltier effect (so no LN2 is required). The chamber was designed to be attached to a diffraction tube housing. WOBISTRAX can be operated with a 3 kW long fine focus Mo-X-ray tube and uses a Mo/Si multilayer for monochromatization. The software is performing the motion control between sample changer and MCA features. The performance is expressed in terms of detection limits which are 700 fg Rb for Mo Ka excitation with 50 kV, 40 mA excitation conditions, 1000 s live time. Using a 35 W air-cooled low power X-ray tube with Rh anode and a 20-mm2 SDD with ultra-thin window, 90 pg detection limit for Rh can be achieved [2].

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This more convenient and low-cost setup provides detection limits of a factor 100 higher than the high power setup.



Figure 1: Vacuum chamber with 12 sample tray and loading port [1]

An adapted version uses dual energy-band for excitation allowing simultaneous efficient excitation of low, medium, and high Z elements [3]. The spectrometer is equipped with an air-cooled 35 W low power Rh X-ray tube and a 17 mm2 silicon drift detector with a thin 8 µm beryllium window. A Pd/B4C multilayer monochromator is used at the same time as a Bragg reflector for Rh-Ka radiation and as a highenergy cut-off reflector above 5 keV, where the characteristic Rh-L radiation is totally reflected and present in the spectrum of the exciting radiation. This leaves one broad low energy band below 5 keV and one high energy band around the energy of Rh-Ka. As Rh-L radiation would be absorbed on its path through air, a new beam entrance system was designed in order to guide the Rh-L photons into the vacuum chamber for efficient excitation of low Z elements.



Figure 2: The NIST1640 SRM measured in air and under vacuum conditions (1 mbar). The right spectrum displays the low-energy section, showing a dramatic enhancement of the fluorescence inten- sities of low Z elements due to additional excitation from the Rh-L lines. The applied solution contains 1 ppm Y as the internal standard. [3]

With this setup, elements down to sodium (Z = 11, E = 1.04 keV) could be detected. First results are presented, and spectra obtained in air as well as in vacuum are compared and discussed. Detection limits in the range of 1000 µg/kg for Na and around 140 µg/kg for Mg were achieved using the NIST SRM 1640 (trace elements in water)

JGIXA, a software for nanolayer characterization

Grazing incidence XRF (GIXRF) is a very surface sensitive, nondestructive analytical tool making use of the phenomenon of total external reflection of X-rays on smooth polished surfaces. In recent years the method experienced a revival and became a powerful tool for process analysis and control in the fabrication of semiconductor-based devices. Due to the downscaling of the process size for semiconductor devices, junction depths as well as layer thicknesses are reduced to a few nanometers, i.e. the length scale where GIXRF is highly sensitive. GIXRF measures the Xray fluorescence induced by an X-ray beam incident under varying grazing angles and results in angle dependent intensity curves. These curves are correlated to the layer thickness, depth distribution and mass density of the elements in the sample. On the other hand, the evaluation of these measurements is ambiguous with regard to the exact distribution function for the implants as well as for the thickness and density of nanometer-thin layers. In order to overcome this ambiguity, GIXRF can be combined with Xray reflectometry (XRR). This is straightforward, as both techniques use similar measurement procedures, and the same fundamental physical principles can be used for combined data evaluation. Such a combined analysis removes ambiguities in the determined physical properties of the studied sample and, being a correlative spectroscopic method, also significantly reduces experimental uncertainties of the individual techniques.

Here we report on the approach for a correlative data analysis, based on a concurrent calculation and fitting of simultaneously recorded GIXRF and XRR data. In brief, we developed JGIXA (Java Grazing Incidence X-ray Analysis), a multi-platform software package equipped with a user-friendly graphic user interface (GUI) and offering various optimization algorithms [4]. The software window is illustrated in Figure 3.



Figure 3: 18 nm Ti layer characterized by JGIXA

Both software and data evaluation methodology were benchmarked by characterizing metal and metal oxide layers on Silicon as well as Arsenic implants in Silicon.

Adaption of the WOBImodule to a compact module

The WOBI-module [5] developed in 1986 by P. Wobrauschek was used as TXRF attachment to existing EDX spectrometers, also distributed via the IAEA to a number of countries. As the development in X-ray sources and ED detectors offers great advantages in compactness compared with the 2 kW water-cooled X-ray tubes they can be replaced now by air-cooled low power tubes, liquid nitrogen cooled Si(Li) detectors can be replaced by small light weighted Peltier-cooled Silicon drift detectors.

Using these new sources and detectors the setup of the WOBI-module can be reduced drastically in length leading to a compact TXRF spectrometer with comparable detection limits as with the high-power equipment (see Figure 4).

	LLD		(pg/m3)	(1000 s, 1 m3)
Screen for			1000	S
adjustment and				
beamstopper	z		Old 50 kV 30 mA 30 mm2	New 50 kV 1 mA 30 mm2
	16	S	6811	8553
	17	CI	4699	5298
	19	Κ	1844	1744
	20	Ca	1522	1613
	22	Ti	973	1186
CDD T	23	v	718	911
SDD	24	Cr	568	701
	25	Mn	417	516
Radiation	26	Fe	312	388
protection	28	Ni	7992	7767
shielding	29	Cu	121	161
	30	Zn	100	134
	33	As	61	83
Low power X-	34	Se	65	87
ray tube	35	Br	76	95
Tay tube	37	Rb	131	128
	38	Sr	170	170
	56	Ва	2583	4708
	82	Pb	160	178

Figure 4: Compact TXRF module and detection limits for air particulate matter directly sampled on Silicon reflectors.

These ideas were realized in the KFKI lab in Budapest using a 50 W Mo tube from Petrick operated by a Matsusada high voltage generator. The controls software was developed by Atominstitut. Directly on the tube housing close to the exit window the easy adjustable monochromator unit with the multilayer monochromator is attached giving a monochromatic intensive beam for excitation. These components are now connected with the part of the WOBImodule allowing the insertion of the sample reflector and adjustment of the sample reflector to grazing incidence of the exciting X-rays. The detector used is a 7 mm² KETEK SDD.

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Czech Republic

Laboratory Portrait:

Czech Technical University, VR-1 Reactor Laboratory

The laboratory denoted as VR-1 reactor lab is the training and research centre in nuclear engineering, reactor physics, and radioanalytical techniques, oriented but not limited to those related to neutrons. It is operated by the Department of Nuclear Reactors, Faculty of Nuclear Engineering and Physical Sciences of the Czech Technical University (CTU) in Prague.

VR-1 reactor

The VR-1 reactor is the light water zero power reactor operated since 1990. It's used mainly for E&T but also for R&D. The main achievements in the past 30 years are summarized in Ref. [1].

The E&T includes mainly courses on experimental reactor physics for students and professionals in the field of nuclear engineering, but also tailored courses for nuclear newcomers (e.g. Research Reactor Group Fellowship Training Course organized within EERRI coalition in cooperation with IAEA), courses for nuclear policy students (e.g. Nuclear Research Reactor Practicum for students of Middlebury Institute of International Studies at Monterey), or dedicated training for Czech emergency response teams.



Figure 1: Training of emergency response teams in contamination identification utilizing short-lived Mn-56 isotope produced at VR-1 reactor.

To further increase the training capability of the reactor for remote users, the Internet Reactor Lab (IRL) has been recently developed. The system enables remote training in two arrangements: from reactor to classes utilizing commercial AVAYA videoconference system and from reactor to homes utilizing various videoconference software like ZOOM. Both arrangements enable to broadcast signal from one of six available cameras, reactor operator's screen or teacher's screens to keep the close contact on activities happening on-site. Dedicates SWIRL software for on-line experimental data flow to students and trainees has been developed as well. Several training courses have already run in this arrangement for local students, as well as for students from Slovakia, UK, and USA. Dedicated remote course through IRL supported by IAEA for students in Belarus and Tunisia will be launched in Autumn 2021.

The R&D at the VR-1 reactor includes validations of calculation codes, study on dynamic properties of multiplicative systems and transient investigations (incl. the role of human operator), development of control systems, detector characterization, testing and applications, and use of neutrons for various applications, mostly for Neutron Activation Analysis (NAA). The effective utilization of the facility for R&D benefits from the "open access" policy of the VR-1 training reactor supported by Ministry of Education, Youth and Sports of the Czech Republic. Recently, the facility closely cooperated with the Centre of competence on Advanced Detection Systems of Ionizing Radiation developed at the Faculty of Nuclear Engineering and Physical Sciences of the CTU in Prague, now it participates in the Centre of Advanced Applied Sciences - CAAS, a cross-CTU project integrating basic and applied research in various fields.



Figure 2: MONTE-1 device used for remote measurement of gamma spectra of unirradiated and slightly irradiated nuclear material developed in cooperation with National Radiation Protection Institute.

VR-2 subcritical reactor project

Currently, the department is developing a new subcritical reactor VR-2 which should further expand education and experimental capacity of the laboratory. The design will reuse the fuel donated by the Aalto University in Finland, where it was part of a subcritical reactor operated in the 1970's. Two types of fuel rods will be used: EK-10 fuel consisting of a dispersion of UO₂ enriched to 10 % of ²³⁵U with Mg in an aluminium cladding and rods with natural uranium. The design enables variations in core design with respect to fuel grid and arrangement of the two fuel types. The reactor will be moderated by light water and driven by source, in this case a portable D-D neutron generator. Other isotopic or generator sources are considered for the future as well. Around the reactor vessel additional layer of shielding concrete will be provided.



Figure 3: Cross-cut of the VR-2 reactor showing core barrel, reactor vessel with radial channel and shielding.

The VR-2 reactor will be used both for E&T and R&D in the fields related to reactor physics, but also to support development and testing of neutron detection systems and their applications. The VR-2 subcritical reactor already got site permit and the application for construction is now evaluated by the State Office for Nuclear Safety. Start of operation is expected at the end of 2022. The VR-1 facility includes several dedicated labs:

- Neutron activation analysis (NAA) lab used for gamma spectrometric activities.
- Radiation detection lab used for basic training with radiation detectors.
- Neutron lab used for experiments with non-reactor neutron sources.
- Electronic lab used for testing of reactor control system or neutron detection systems on artificial signals.
- Physical protection lab, with a set of technical measures for protection of nuclear installations allowing for demonstrations, modelling, simulations, or testing in the field

Use of other neutron sources

Beside the VR-1 reactor, the facility operates few radionuclide (Am/Be, Cf-252) and portable generator neutron sources (D-D and D-T) with intensities ranged from 10^5 to 10^8 neutrons per second. The sources are used both for E&T and R&D.



Figure 4: D-D neutron generator in polyethylene set-up used for experiments related to neutron activation and delayed neutron counting.

The activities include studies on neutron transport in various environments, characterization of neutron sources, neutron detection and spectroscopy, characterization of neutron detectors and their testing for applications (e.g., dosimetry in the mixed field of neutrons and gamma rays) or testing of innovative shielding materials.

Applications of radioanalytical methods

Several radioanalytical methods are used both for research and training activities: NAA at VR-1 reactor has been utilized in many studies including cultural heritage (e.g. Tibetan coins and medicinal pills, or Czech historical artefacts), natural samples (e.g. mammoth remains, meteorites), environmental samples (e.g. mosses, alluvial soils), or health (e.g. composition of dietary supplements). Moesbauer spectroscopy have been recently used for analysis of steels, metallic glasses or Fe-based nanoparticles. Delayed neutron counting (DNC) is established both at VR-1 reactors and with portable neutron generators. The latter option is used for research of methods for nuclear material detection. Recently, PIXE capability was included in the portfolio of used radiation methods. Under development is the digital neutron radiography at the radial channel of VR-1 reactor.

Summary

The VR-1 reactor facility is an internationally established training centre in the field of experimental neutron and reactor physics. Gradually, its training and research capabilities has been increased to the fields of nuclear analytical techniques and neutron measurements and applications. New isotopic and generator-based neutron sources have been acquired to support these activities and new neutron lab has been developed. Currently, the development is concentrated on the development and utilization of the VR-2 subcritical assembly. High quality E&T together with own R&D and open access policy thus create a hub with high potential to support the development in the above-mentioned field both locally and internationally.

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Hungary

Environmental Physics Department - Centre for Energy Research

Our department has two laboratories related to X-ray and neutron research. The research activity of the department mainly focuses at radioactive waste management, atmospheric particulate matter and energy storage systems.

The X-ray fluorescence (XRF) laboratory was recently improved using air-cooled low-power X-ray tube based systems. It offers TXRF analysis of liquid samples and aerosol particles directly collected onto reflectors and 2D micro-XRF mapping of solid samples with a 20 μ m resolution.

Neutron powder diffraction is a standard technique for studying atomic and magnetic structures. We host the PSD - neutron diffractometer (located at Budapest Neutron Centre, based on a research reactor) with a position sensitive detector system used for structural studies of amorphous materials. Materials can have varying degrees of disorder. The neutron diffraction technique can be used to measure the structure of atom clusters and molecules in disordered materials. Glasses lack long-range order but may have medium range ordering over several atomic lengths. The measured diffraction pattern is related to the positions of the atoms relative to each other in the glass.

The diffractometer is suitable for structural studies of powder, liquids and amorphous materials in the range of momentum transfer Q=0.45-9.8 Å⁻¹. The wavelength selected from the primary neutron spectrum is around 1 Å (now is λ =1.068 Å), for which, in the case of Cu(111) monochromator single crystal a neutron flux density of about 106 n·cm⁻ ²·s⁻¹ can be achieved at the sample position.

Application of TXRF to elemental analysis of aerosol particles

Analytical capabilities of the combination of Mayimpactor sampling and subsequent TXRF of the collected size-fractionated particulate matter were tested. Using a system comprising of a low power X-ray tube (50 kV, 1 mA), Mo-K α excitation with a multilayer monochromator and a 7-mm² SDD, a 100 pg/m³ detection limit could be reached on each stage for metals with time resolution of 1-4 hours [1].

In May-type impactor aerosol particles are deposited on 7 silicon wafers correspondent to $0.07-10 \mu m$ size range, size distribution of chemical elements could be determined accordingly. Figure 1 shows results for a sample set collected for 1 hour during the central fireworks on the Hungarian national day (20 August 2019). The difference in the distribution of Sr (fireworks origin, fine mode) and Ca (soil resuspension origin, coarse mode) is clearly visible [1].

Particles collected on Si wafers are available for further complementary investigations such as SEM/EDX, Ramanspectroscopy or even synchrotron radiation X-ray methods. Parallel monitoring of black carbon and number size distribution offer a more complete information on particulate pollutants present in the air.



Figure 1: Sampling and results of elemental size distributions during central fireworks in Budapest on 20 August 2019

Application of neutron diffraction in nuclear waste management

Structural investigation was performed on a multi-component glassy system as a potential matrix material for immobilization of high-level radioactive wastes. The structure factor S(Q) for the 70/90wt% Matrix glass $[55SiO_2 \cdot 10B_2O_3 \cdot 25Na_2O \cdot 5BaO \cdot 5ZrO_2 (mol\%)] + 30/10$ wt% of the Ln oxide [CeO₂, Nd₂O₃, Eu₂O₃] sample was measured at the PSD neutron diffractometer, the datasets were calculated using the program existing at the facility. We obtained typical amorphous spectra with broad distributions, specific to the amorphous state as illustrated in Figure 2 [2]. In order get deeper insight into the network structure, the neutron diffraction study was combined with reverse Monte Carlo (RMC) modeling.



Figure 2: Total structure factors derived from neutron diffraction on Matrix-Ce10 (blue), Matrix-Nd10 (green), Matrix-Eu10 (red) glasses (colour symbols) and RMC fits (black solid lines). For better visibility, the curves were shifted vertically.

The RMC simulation in accordance with experimental neutron diffraction data show the basic network structure of the Ln-oxide-doped borosilicate glasses to consists of mixed ^[3]B-O-^[4]Si and ^[4]B-O-^[4]Si chain segments, but the fractions of BO₃ and BO₄ units strongly depend on the Ln-

oxide content in such a way that formation of BO_4 is promoted by increasing the Ln-oxide content. The second nearest neighbour atomic pair correlations established between cerium, neodymium, europium and the network forming (Si, B) atoms accentuate that the Ln-doped glasses exhibit a stable basic network structure [2].

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Morocco

Use of TXRF in Characterization of Airborne Particulate Matter in Moroccan Urban Area (Sale City)

The chemical characterization of particulate matter can be carried out using many multi-elemental techniques. Total reflection X-ray fluorescence spectrometry (TXRF) is one of the widely used techniques for multielement analysis of different types of materials. The TXRF is a powerful analysis technique, which is very suitable for small amounts of material, such as that collected from aerosol samples. Because of its high sensitivity and the small quantity of material required for TXRF analysis, the technique was used in many studies to characterize and quantify air particulate matter pollution [1, 2]. Particulate matter (PM) is a complex mixture of extremely small particles and liquid droplets. In 2000, the World Bank estimated the cost of environmental degradation (COED) in Morocco at 3.7 percent of GDP, of which air pollution was the second most important component. Despite a number of significant positive changes, such as the introduction of cleaner fuels unleaded and 10 ppm of SO2, the establishment of norms on PM10, Pb, Cd, gaseous pollutants and benzene, air quality, in particular in urban areas, remains a challenge.

In this context, the National Centre of Energy, Sciences and Nuclear Techniques (CNESTEN) in Morocco has carried out some studies related to the evaluation of air quality in some Moroccan cities (Meknes, Kenitra, Sale, Tetouan) and quantification of emissions sources in the concerned areas [3-5]. We are also assessing the air pollution impact on human health and cultural heritage in Sale and Kenitra cities. These studies were performed with the support of the IAEA and under bilateral collaboration with France, Spain and Italy.

This short paper presents an example of TXRF application in monitoring trace metals in particulate matter collected in Sale City and in the framework of the regional IAEA TC project RAF7016 entitled: "Establishing and Improving Air Pollution Monitoring".

Presentation of the area under study

Sale, a city with 890 403 inhabitants (2014's estimate), is the third largest city in Morocco. The Bouregreg river separates Sale from Rabat (Morocco's capital city) (Figure 1).

The city is characterized by high demographic density with the main air pollution sources such as traffic, ocean, potteries, fishing, domestic heating and artisanal activities.



Figure 1: Localization of Sale and Tetouan Cities in Morocco.

Sampling was conducted in an urban area (34.043806, -6.788583) in Sale City using a Dichotomous sampler. It consisted of simultaneous collection of PM2.5-10 and PM2.5 fractions respectively called coarse and fine particles, at a total flow rate of 16 l/min.

Samples were collected over 1 year (from July 2018 to July 2019), based on one filter during a weekday and another on the weekend, with 24-h sampling time on 37 mm diameter

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PTFE membrane disc filters. In total, 212 filters were collected (106 for each fraction). The filters were manipulated in a clean area and handled with gloved hands and Teflon tweezers and stored in Petri dishes.

Methods

PM2.5 and PM2.5–10 mass concentrations were determined by gravimetric analysis. This method consisted of weighing the filters before and after sampling. Weighing was carried out on a Microbalance with 1 μ g sensitivity. Filter weighing was achieved after 24 h equilibrium at room temperature with atmospheric relative humidity maintained below 30%. The uncertainty for each weighting was in the order of 20 μ g. Each filter was weighed in duplicate for getting the average weight of exposed and unexposed filters.

Trace metals were analysed using a benchtop S2-PICO-FOX TXRF-spectrometer. The instrument is equipped with a Mo anode metal-ceramic X-Ray tube, a multilayer monochromator and a Si drift detector with 30 mm² area and 160 eV energy resolution for Mn K α -line. Energy excitation was 17.5 keV and the instrument was operated at 750 μ A and 50 kV. Treatment of X-ray spectra, analysis of elemental concentration and deconvolution of spectra fluorescence peaks were performed using the SPECTRA software provided with the instrument, after manual identification of the elements.

TXRF technique analysis of filters requires to bring the samples into solution. In this work, the loaded filters were decomposed with 8 mL of conc. HNO₃ and 2 mL of H₂O₂ in a household microwave oven (Milestone MAXI14) at 200°C for 45 minutes. The digested samples were kept in a refrigerator at 4°C until analysis. Ga was added for internal standardization. 10 µL of the resulting solution was transferred to a quartz glass sample carrier after homogenization step and dried under infrared lamp. The resulting solutions were measured for 1000 seconds. A blank filter was analyzed after each 10 samples analysis to determine the eventual contaminations. The blank filters were analyzed using the same procedure as the samples. Element intensities in the blank allowed to correct contamination effect in the unknown samples. Accuracy of the analysis was evaluated using a standard reference material (SRM, 2783 Air particulate on Filter Media - NIST). Relative deviation of the obtained concentrations from the certified values varies from 2 to 14%, in the absolute value.

Concentrations of PM2.5 and PM10 in Sale City

Regardless of particle size, the highest concentrations were recorded during the winter season, and attributed to stagnant atmosphere. In fact, winter season, during the sampling period, was characterized by low wind speed and high humidity compared to other seasons which favorite the accumulation of pollution during this season (Figure 2).



Elemental composition of PM in Sale City

The concentrations of soil-related elements (K, Ca, Ti, Fe, Mn and Sr) were higher in the coarse fraction. However, the elements from anthropogenic sources (V, Ni, Cu, Zn, and Pb) were more present in the fine fraction (see Figure 3).

The estimation of the emission sources and their contribution to ambient fine and coarse fractions will be performed using the positive matrix factorization receptor model developed by US EPA.



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Back Scatter

Selected recent Web stories

The IAEA regularly informs the media and public of its work through press releases, impact stories, videos and podcasts, as well as photo essays and statements of the IAEA Director General and senior staff. It also gives access to the many events it organizes, from conferences to technical meetings. The latest stories on NSIL results can be accessed from https://www.iaea.org/news and include:

- <u>Now Available: New Drone Technology for Radiological Monitoring in Emergency Situations, Feb 1, 2021</u>
- New IAEA Online Platform Offers E-Materials in Nuclear Instrumentation, Feb 26, 2021
- IAEA Conducts First Virtual Training on Using Ion Beam Techniques and Applications, Apr 7. 2021

Selected upcoming Events

- Virtual Technical Meeting on Advances in Neutron Detectors for Neutron Scattering and Imaging Applications, 30 Aug –3 Sep
- Virtual Training Workshop: on Advanced X-ray techniques for Characterization of Valuable Objects, 6-10 Sep
- Virtual Training Workshop on Advanced Use of Neutron Imaging for Research and Applications (AUNIRA), 6-10 Sep
- Technical Meeting on Developments in Non-Radiocarbon Accelerator Mass Spectrometry Techniques and Relevant Applications, 11 14 Oct, Vienna, Austria
- Virtual Technical Meeting on Artificial Intelligence for Nuclear Technology and Applications, 25 29, Oct
- Virtual Joint IAEA/ICTP School on Synchrotron Light Sources and their Applications Synchrotron Light Sources and their Applications, 6-17 Dec
- Virtual Training Workshop: on Expanding the Research Reactor Stakeholder Base through Strategic and Business Plans, 1 – 5 Nov.
- 4th Technical Meeting on Fusion Data Processing, Validation and Analysis, 30 Nov 3 Dec
- 16th Vienna Conference on Instrumentation VCI 2022 (organized in cooperation with the International Atomic Energy Agency), Vienna, 21-25 Feb, 2022.
- CN-301 the International Conference on Accelerators for Research and Sustainable Development: from good practices towards socioeconomic impact, 23 to 27 May 2022, Vienna, Austria, <u>https://www.iaea.org/events/AccConf22</u>

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The IAEA is organizing the First International Conference on Accelerators for Research and Development: from good practices towards socioeconomics impact. Such a Conference was long awaited by the Member States to address important needs in our high-tech oriented society, where particle accelerators have become indispensable.

The Conference aims primarily to present an international stage for discussing accelerator applications in research and industry, foster exchange of information on best practices in accelerator facility utilization and management, and to provide a showcase how achievements and experience attained with accelerator technologies contribute to a sustainable development. Special emphasis will also be given in accelerator applications of large societal impact such as human health, environmental monitoring, cultural heritage, food quality, energy sector, forensics, nuclear security, and others promoting economic development.

Three major themes will be addressed by the Conference: a) Cutting-edge scientific results and innovation in applications, b) Success stories and case studies demonstrating socioeconomic impact, and c) Best practices in effective management, safe operation, and sustainability of accelerator facilities, including establishment of new facilities.

Updated information on the Events organized by the NSIL is available on the IAEA <u>Nuclear Science and Instrumentation</u> <u>Portal</u> and also through the <u>IAEA Meetings webpage</u>.

Contributions to our Newsletter

Publication of materials for consideration in our Newsletter should be submitted in MS Word only.

Short article submissions should be no more than 800 words with up to 2 high quality figures/pictures (including captions) and, if needed, a maximum of 3 most relevant references.

Submissions for "Laboratory Portrait" articles, a new rubric in the Newsletter, should be no more than 1500 words with up to 4 figures/pictures and, if needed, a maximum of 6 most relevant references.

Impressum

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