

15th International Conference on Particle Induced X-ray Emission



PIXE: reaching new frontiers

April 2 – 7, 2017, Split, Croatia

CONFERENCE PROGRAM AND ABSTRACTS

Organised by

Ruđer Bošković Institute (RBI), Zagreb
University of Rijeka, Rijeka

In cooperation with

International Atomic Energy Agency (IAEA), Vienna
Center of Excellence for Advanced Materials and Sensing Devices (CEMS)

CONFERENCE PROGRAM AND ABSTRACTS: 15th International
Conference on Particle Induced X-ray Emission

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April 2 - 7, 2017 - Split, Croatia

Editors: Stjepko Fazinić, Ivana Zamboni

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Welcome

On behalf of the local organizing committee, it is our pleasure to welcome all participants to the 15th International Conference on Particle Induced X-ray Emission (PIXE2017). This fifteenth PIXE conference follows a series that began 41 years ago with the first one which took place in the city of Lund (Sweden) in 1976, followed by the second one again in Lund (1980) and continuing with conferences in Heidelberg (1983), Tallahassee (1986), Amsterdam (1989), Tokyo (1992), Padua (1995), Lund (1998), Guelph (2001), Portorož (2004), Puebla (2007), Guildford (2010), Gramado (2013) and Cape Town (2015). With such long tradition, the PIXE conferences have always been the right place to discuss recent developments and future prospects in particle induced X-ray spectrometry and its applications. As such, the conference brings together physicists, engineers and scientists from different fields, including biology and medicine, environmental and earth sciences, cultural heritage and materials research.

PIXE2017 is being organized by the Laboratory for Ion Beam Interactions of the Division of Experimental Physics, Ruđer Bošković Institute, Zagreb, together with the Department of Physics, University of Rijeka. We are grateful to our sponsors, exhibitors and all the people that helped us in the organisation of this event.

The conference will be held in Split, the second largest city in Croatia situated in the very heart of the Adriatic eastern coast. The Historic Complex of Split together with the enticing Palace of Diocletian, spanning across 38500 square meters, is recognized as UNESCO World Cultural Heritage Site. The cathedral was built in the Middle Ages, reusing materials from the ancient mausoleum. Twelfth and 13th century Romanesque churches, medieval fortifications, 15th century Gothic palaces and other palaces in renaissance and baroque style make up the rest of the protected area. The social program will include conference outing to the National park Krka and visit to the charming little town of Trogir included in the UNESCO list of World Heritage Sites, providing an excellent opportunity to spend enjoyable moments with colleagues and friends.

Finally, we hope that you will enjoy the conference and all that Split can offer.

Stjepko Fazinić (RBI), Ivo Orlić (University of Rijeka), Tonči Tadić (RBI)

Committees

International Advisory Committee

- M.A. Reis - Portugal (Chair)
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- D.D. Cohen - Australia
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- J.F. Dias - Brazil
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- Tonči Tadić, Ruđer Bošković Institute, Zagreb (co-chair)
- Iva Bogdanović Radović, Ruđer Bošković Institute, Zagreb
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- Milko Jakšić, Ruđer Bošković Institute, Zagreb
- Zdravko Siketić, Ruđer Bošković Institute, Zagreb
- Ivan Sudić, Ruđer Bošković Institute, Zagreb
- Ivana Zamboni, Ruđer Bošković Institute, Zagreb
- Marija Čargonja, University of Rijeka, Rijeka
- Tatjana Ivošević, University of Rijeka, Rijeka
- Darko Mekterović, University of Rijeka, Rijeka
- Vladan Desnica, University of Zagreb, Zagreb
- Domagoj Mudronja, Croatian Conservation Institute, Zagreb
- Domagoj Šatović, University of Zagreb, Zagreb
- Ian Swainson, IAEA, Vienna

Conference Manager

- Ana Vidoš, Ruđer Bošković Institute, Zagreb

Locations

The conference will be held at **Radisson Blu Resort**, situated about three kilometres from Split's city centre. The hotel address is:

Radisson Blu Resort
Put Trstenika 19, Split
tel: +385 (0) 21 303 030.



The conference dinner will be held at **Restaurant Kadena**, Ivana pl. Zajca 4, Split.



Exhibitors and Sponsors

- National Electrostatics Corp. (www.pelletron.com)
- RaySpec Ltd. (www.rayspec.co.uk)
- High Voltage Engineering Europa B.V. (www.highvoltage.com)
- Oxford Microbeams Ltd. (www.microbeams.co.uk)



Organizers

- Ruđer Bošković Institute (www.irb.hr)
- University of Rijeka (www.uniri.hr)



Collaborators

- International Atomic Energy Agency (www.iaea.org)
- Center of Excellence for Advanced Materials and Sensing Devices (cems.irb.hr)



Pelletron Systems

MATERIALS ANALYSIS

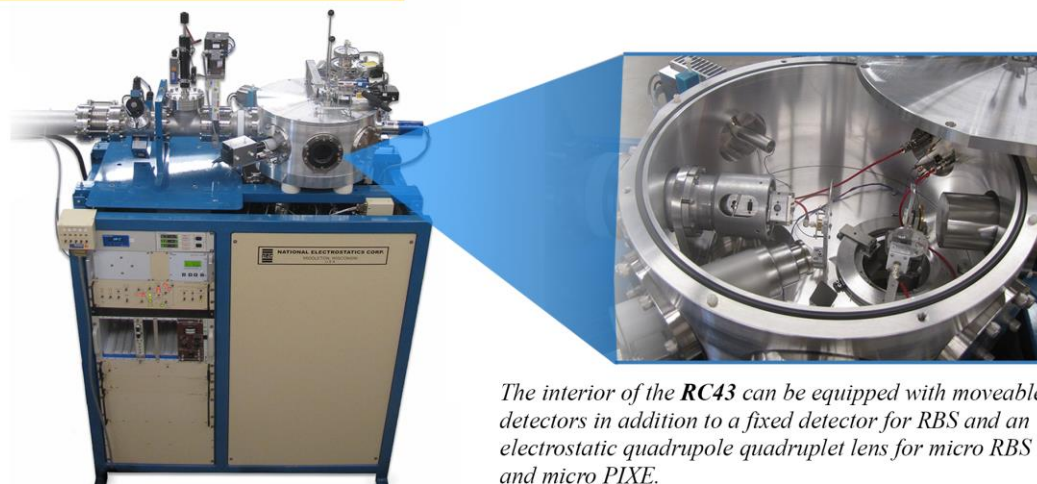
National Electrostatics Corp. offers tandem and single ended Pelletron systems with all hardware and software for **RBS, channeling, PIXE, ERD and NRA** data collection. Software is included for RBS and PIXE data analysis



Pelletron Model 3SDH equipped with NEC RF Charge Exchange Ion Source (Alphatross) and the NEC RC43 Analysis Endstation

Techniques available: RBS, Channeling, ERD, ToF ERD, PIXE, PIGE, NRA, IBIL

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The NEC **RC43 Analysis Endstation** combines several complementary materials analysis ion beam techniques with automation software to yield a fast, complete, materials analysis. The software allows several techniques to be simultaneously “live”. Also, the RC43 generates mappings of surface elemental concentrations and crystal structure as images yielding a true 3D analysis.

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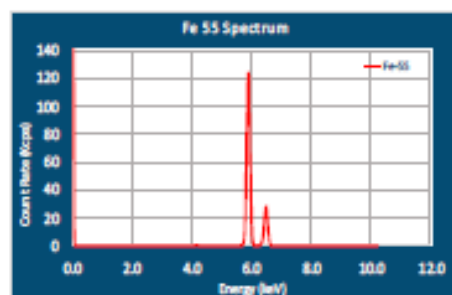
Contact NEC for more information

Phone: (608) 831-7600 | Email: nec@pelletron.com
7540 Graber Rd. P.O. Pox 620310 Middleton, WI 53562 USA

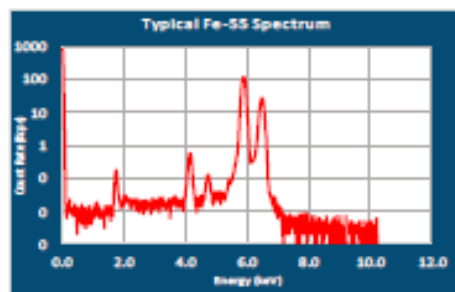
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Systems for Ion Beam Analysis

- Rutherford Backscattering Spectroscopy (RBS)
- Particle Induced X-ray Emission (PIXE)
- Nuclear Reaction Analysis (NRA)
- Elastic Recoil Detection (ERD)
- Medium Energy Ionscattering Spectroscopy (MEIS)

Accelerator Mass Spectrometers

^3H , ^7Be , ^{10}Be , ^{14}C , ^{26}Al , ^{32}Si , ^{36}Cl , ^{41}Ca , ^{53}Mn , ^{79}Se , ^{129}I , ^{236}U etc.
analysis for use in

- Archeology
- Oceanography
- Geosciences
- Material sciences
- Biomedicine
- Etc.

Systems for Micro-beam applications

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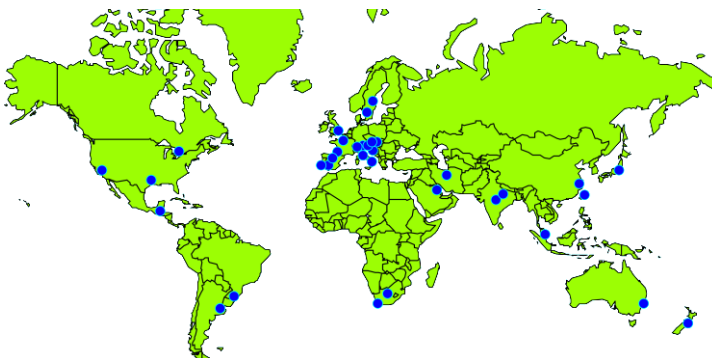


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- Heavy ion microbeams (to 150 MeV.amu/q²)
- Ionoluminescence microscopy
- Elastic Recoil Detection Analysis
- Ion induced secondary electron imaging
- Direct-write Ion Beam implantation and patterning of materials
- Channeling Contrast microscopy
- Ion Beam Induced Charge microscopy

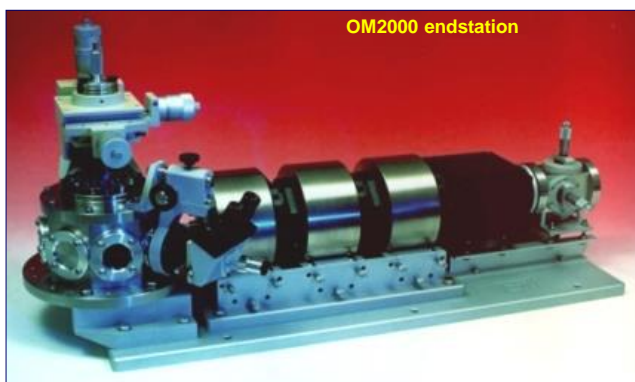
Performance of OM systems:

OM2000 Oxford triplet endstation:

300 x 450 nm at 50pA (2MeV protons).
900 x 900 nm at 1 nA (2.5MeV protons)

Oxford triplet configuration of OM-52 lenses:

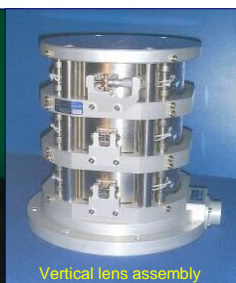
Spot sizes of **20 x 25nm** have been achieved for low current applications, and **20nm** high aspect ratio structures have been written using proton beam writing.



OM2000 endstation



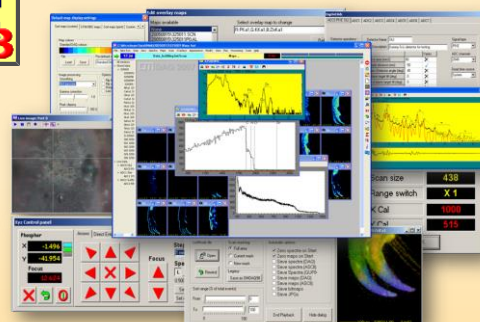
OM-52 Miniature triplet



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General Information

Oral Presentations

Invited talks are 30 minutes long (25 minutes presentation and 5 minutes discussion). Contributed talks are 20 minutes long (15-17 minutes presentation and 3-5 minutes discussion). All the talks should be accompanied by PowerPoint or PDF slides.

Lecture Hall will be equipped with a PC and LCD projection system. The PC will accept USB drives and will have Microsoft Windows 10, Microsoft PowerPoint 2016 and Adobe Reader. Speakers are kindly requested to check the time allotted for their talk in the Conference Program. Speakers are asked to load their presentations (MS Power Point or PDF) before the start of their sessions.

Poster Displays

The size allocated for posters is 90 cm (width) by 120 cm (height). There will be one poster session, that will be held on Tuesday 4th April, from 15:30 to 17:00. Poster presenters should be available at their display during this time. Posters should be set up on Monday 3rd April during Lunch break. The posters will be on display until Thursday 6th April 18:00. Please display your poster in the slot assigned with your number.

Conference Proceedings

The PIXE2017 conference proceedings will be published as a special volume of the journal "Nuclear Instruments and Methods B: Beam Interactions with Materials and Atoms". All submitted papers must be clearly written in excellent English and contain only original work, which has not been published by or is currently under review for any other journal or conference. All manuscripts and any supplementary material should be submitted through the Elsevier Editorial System (EES). Papers will be reviewed to the same standards as for the regular NIMB papers. Please contact Stjepko Fazinić (sfazinic@irb.hr) for other publication related questions.

Internet Access

Free internet access is available in the hotel and congress center.

Registration and Welcome Reception

Conference delegates and accompanying persons are invited to attend a Welcome reception at the conference hotel on Sunday, 2nd April, where food and drinks will be offered from 19:00 to 21:00. Registration will be open on Monday from 8:00.

Lunches

Lunches will be provided on Monday, Tuesday and Thursday at the conference hotel. On Wednesday, lunch will be provided during the conference outing. Lunches are included in the conference fee.

Conference Outing

The Conference outing will be held on Wednesday, 5th April. We will meet at the lobby of the Radisson Blu hotel at 9:45 and depart to Krka National Park by busses. Expected arrival at about 11:30 and visit with licensed guide for up to 2 hours. In a small village near the Krka National Park the participants will have guided tour through the etno collection and have traditional lunch.

At about 16:30 we will depart to Trogir, a picturesque town recognized as UNESCO World Cultural Heritage Site in 1997 with justification that "Trogir is an excellent example of a medieval town built on and conforming with the layout of a Hellenistic and Roman city that has conserved its urban fabric to an exceptional degree and with the minimum of modern interventions, in which the trajectory of social and cultural development is clearly visible in every aspect of the townscape." The arrival to Trogir is estimated to 17:30. The participants will have one hour free time in Trogir, and at 18:30 bus transfer will start to the hotel, with stop in the old town of Split for those who wish to spend the evening in downtown.

Outing is included in the conference fee.

Conference Banquet

The conference banquet will be held in the Restaurant Kadena on Thursday, 6th April with the start at 19:30. The restaurant is located at the walking distance from the conference hotel. Banquet is included in the conference fee.

Other Details

We kindly ask participants to stick closely to the allocated duration of oral presentation and to the scheduling for posters. This is particularly important as we have a relatively densely packed program. Please wear your conference badge at all times. We have made special arrangements regarding lunch and refreshments, and your badge needs to be visible at these venues. The Local organizing committee will be pleased to help and advise on travel, touring and other arrangements, or on any problem.

Meeting Questions

If you have any questions, please contact Ana Vidoš or anyone from the local organizing committee. Here are some cell phone numbers that may be useful:

Ana Vidoš avidos@irb.hr, +385 98 480 671

Stjepko Fazinić, sfazinic@irb.hr, +385 99 317 4866

Tonči Tadić, ttadic@irb.hr, +385 95 910 7758

Ivo Orlić, ivo.orlic@uniri.hr, +385 91 507 4066



Scientific Program

15th International Conference on Particle Induced X-ray Emission (PIXE2017) program

Radisson Blu Resort, Split

Sunday, April 2, 2017	
19:00 – 21:00	PIXE2017 Welcome reception

Monday, April 3, 2017		
8:00 – 9:00	Registration	
9:00 – 9:20	Opening remarks and welcome address by: Stjepko Fazinić – PIXE2017 chair Tome Antičić – RBI Director General Miguel A. Reis – chair of the PIXE International Advisory Committee	
Session 1 Chair: Miguel A. Reis		
9:20 – 9:50	John L. Campbell <i>Martian Chronicles</i>	I-1
9:50 – 10:10	Wojciech Przybyłowicz <i>Elemental mapping using XFM with the Maia detector array and the nuclear microprobe with a single-detector: exploiting their individual merits</i>	O-1
10:10 – 10:30	Andreas G. Karydas <i>A review of potentialities and perspectives of proton induced quasi-monochromatic X-rays in fundamental studies and analytical applications</i>	O-2
10:30 – 11:00	Coffee/Tea break	
Session 2 Chair: Keizo Ishii		
11:00 – 11:30	Marek Pajek <i>Emission of M-X-rays induced by light and heavy ions</i>	I-2
11:30 – 11:50	David D. Cohen <i>Comparison of proton and carbon PIXE at the same ion velocity</i>	O-3
11:50 – 12:10	Liangdeng Yu <i>Investigation on MeV C-ion PIXE cross sections</i>	O-4
12:10 – 12:30	Johnny Dias <i>Projectile and target X-rays induced by heavy ions</i>	O-5
12:30 – 14:00	Lunch	
	Posters mounting	

Session 3 Chair: Žiga Šmit		
14:00 – 14:30	Javier Miranda <i>Anisotropy and alignment in L-shell X-ray emission induced by ion impact</i>	I-3
14:30 – 14:50	Keizo Ishii <i>Theoretical detection limit of PIXE analysis using 20 MeV proton beams</i>	O-6
14:50 – 15:10	Mostafa Hazim <i>High energy PIXE: new K-shell ionization cross sections and comparison with theoretical values calculated using ECPSSR</i>	O-7
15:10 – 15:30	Alexandre Subercaze <i>High Energy PIXE: a powerful tool to characterize multi-layers thick samples</i>	O-8
15:30 – 16:00	Coffee/Tea break	
Session 4 Chair: Geoffrey W. Grime		
16:00 – 16:20	Paula Cristina Chaves <i>Study of geological samples from Azores ocean floor by High Resolution and High Energy PIXE</i>	O-9
16:20 – 16:40	Erin L. Flannigan <i>Mineral phase effects in PIXE analysis of igneous rocks</i>	O-10
16:45 – 18:00	Round table: Detectors Discussion moderator: Geoffrey W. Grime	

Tuesday, April 4, 2017		
Session 5 Chair: Milko Jakšić		
9:00 – 9:30	Francesco Paolo Romano <i>Full field PIXE by using an X-ray camera with high-energy and high-spatial resolution</i>	I-4
9:30 – 9:50	Josef Buchriegler <i>Full-field PIXE imaging: Multi-frame super-resolution to overcome optics pattern and imaging-based resolution limitations</i>	O-11
9:50 – 10:10	Thomas Calligaro <i>Development of a 3D positioner for the PIXE mapping of non-flat objects</i>	O-12
10:10 – 10:30	Laurent Pichon <i>Surface microtopography by stereo-PIXE for enhanced map processing</i>	O-13
10:30 – 11:00	Coffee/Tea break	

Session 6		
Chair: Johnny Dias		
11:00 – 11:30	Matjaž Kavčič <i>Chemical speciation using WDS in the tender X-ray range</i>	I-5
11:30 – 11:50	Iva Božičević Mihalić <i>High resolution K X-ray spectra of selected silicates induced by MeV proton and carbon micro-beams</i>	O-14
11:50 – 12:10	Wataru Kada <i>Continuous IBIL analysis and imaging for visualization of chemical composition and modification occurred in microscopic scale targets</i>	O-15
12:10 – 12:30	Group photo	
12:30 – 14:00	Lunch	
Session 7		
Chair: Ivo Orlić		
14:00 – 14:30	Alfonso Mantero <i>PIXE simulation with Geant4</i>	I-6
14:30 – 14:50	John L. Campbell <i>Analysis of surface layers on Martian rocks with the APX-Yield code</i>	O-16
14:50 – 15:10	Miguel A. Reis <i>Simulation and fit of Ta L and M shells PIXE spectra obtained at the C2TN HRHE-PIXE facility using proton and oxygen ion beams</i>	O-17
15:10 – 15:30	Silvina Limandri <i>Standardless method for PIXE analysis</i>	O-18
15:30 – 17:00	Posters, Coffee/Tea	
17:00 – 18:15	Round table: Software Discussion moderator: Ivo Orlić	
18:15 – 19:15	IHC-IAC Meeting	

Wednesday, April 5, 2017	
9:45 – 19:30	Conference outing

Thursday, April 6, 2017		
Session 8:		
Chair: Thomas Calligaro		
9:00 – 9:30	Zita Szikszai <i>Safe boundaries for analysis of heritage materials</i>	I-7
9:30 – 9:50	Žiga Šmit <i>Prehistoric glass beads from Slovenia</i>	O-19
9:50 – 10:10	Zoltán Szőkefalvi-Nagy <i>PIXE and PGAA - complementary methods for studies on ancient glass artefacts (from Byzantine, late medieval to modern Murano glass)</i>	O-20
10:10 – 10:30	Domagoj Mudronja <i>Comparative analysis of textile metal threads from liturgical vestments and folk costumes in Croatia</i>	O-21
10:30 – 11:00	Coffee/Tea break	
Session 9		
Chair: Andrea Denker		
11:00 – 11:20	Mohamad Roumie <i>PIXE contribution for a database of Phoenician Pottery</i>	O-22
11:20 – 11:40	Žiga Šmit <i>Analysis of Greek small coinage from the classic period</i>	O-23
11:40 – 12:00	Hanan M. Sa'adeh <i>PIXE-RBS beamline in the University of Jordan Van de Graaff Accelerator (JUVAC): Development and Applications</i>	O-24
12:00 – 12:20	Shivcharan Verma <i>Standardisation of the ion beam facility at Chandigarh cyclotron for simultaneous PIXE and PESA analysis</i>	O-25
12:20 – 14:20	Lunch	
	IAC Meeting during Lunch	
Session 10		
Chair: Wojciech Przybyłowicz		
14:20 – 14:50	Melanie J. Bailey <i>DAPNe-IBA: A new tool for the IBA community?</i>	I-8
14:50 – 15:10	Karen J. Cloete <i>In-air PIXE and MeV SIMS as analytical techniques for detecting xenobiotics and pharmaceutical compounds in human scalp hair</i>	O-26
15:10 – 15:30	Dragos A. Mirea <i>Particle Induced X-Ray Emission and Raman Spectroscopy in the study of patrimony art forgery</i>	O-27
15:30 – 15:50	Carla dos Santos <i>Elemental signature of Brazilian foodstuff by PIXE</i>	O-28
15:50 – 16:20	Coffee/Tea break	

Session 11 Chair: Zoltán Szőkefalvi-Nagy		
16:20 – 16:50	Kenichiro Mizohata <i>Heavy ion induced X-ray production cross sections and simultaneous heavy ion PIXE analysis with other IBA methods</i>	I-9
16:50 – 17:10	Mandla Msimanga <i>X-ray production cross-sections in metallic films due to C, O and Si ion beams at MeV SIMS energies</i>	O-29
17:10 – 17:30	Tonči Tadić <i>Ion microbeam NRA and PIXE analyses of individual dust particles from JET tokamak</i>	O-30
17:30 – 17:50	Vincent Métivier <i>The Cyclotron Arronax for fundamental and applied research with light ion beams <70 MeV</i>	O-31
19:30 –	Conference banquet – Restaurant Kadena, Split	

Friday, April 7, 2017		
Session 12		
Chair: David D. Cohen		
9:00 – 9:30	Franco Lucarelli <i>The application of PIXE and complementary nuclear techniques to aerosol studies: the LABEC experience</i>	I-10
9:30 – 9:50	Willy Maenhaut <i>Source apportionment revisited for long-term measurements of fine aerosol trace elements at two locations in southern Norway</i>	O-32
9:50 – 10:10	Zsófia Kertész <i>Composition and sources of particulate matter pollution in five Hungarian cities in 2015</i>	O-33
10:10 – 10:30	Mohamad Roumie <i>Characterization of fine and coarse atmospheric particulate matter from Beirut suburb using PIXE technique</i>	O-34
10:30 – 11:00	Coffee/Tea break	
Session 13		
Chair: Willy Maenhaut		
11:00 – 11:20	Marija Čargonja <i>Atmospheric impact of ship emission on fine particulate matter in the Port of Rijeka</i>	O-35
11:20 – 11:40	Alessandra Gianoncelli <i>Elemental distribution in feathers of the Common Swift (Apus apus), a PIXE, hard X-ray and soft X-ray fluorescence study</i>	O-36
11:40 – 12:00	Conference overview – Closing remarks (by Miguel A. Reis)	

List of Posters

<u>M. Pajek</u> , D. Banaś, Ł. Jabłoński, T. Mukoyama	Electronic wave function and binding effects in M-subshell ionization of gold by light ions	P-1
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ABSTRACTS

INVITED TALKS

I-1: Martian Chronicles

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We will discuss some of the strong body of evidence for the existence of water bodies in the Martian geological past that has been assembled by the spectrometric instruments on the Mars Science Lab's Curiosity rover. With some of these bodies apparently able to have supported primitive life, the major goal of the MSL mission has been achieved. Curiosity's Canadian alpha particle X-ray spectrometer APXS continues to be a reliable workhorse for chemical analysis on Mars. We therefore continue to refine our GUPIX-based "fundamental parameters with standards" approach to fitting its highly complex PIXE+XRF spectra. Non-linearity observed by us at low energies in the channel-energy relationship appears to have two dominant contributors. The first is a real non-linearity in the detector-electronic chain and the second is the presence of significant multiple ionization satellites. The two effects may shift peaks in the same or opposite directions. Incorporation of these in our GUAPX spectrum fitting code results in significantly improved fits. We are pursuing these two effects further because of their implications for improved accuracy in terrestrial He ion PIXE analysis. This is an interesting example of an exotic application of PIXE in outer space benefiting everyday PIXE in the laboratory! This research thrust fits with the presently increased interest in PIXE with particles heavier than protons. We have previously reported the interesting dependence of APXS analytical results upon the type of igneous rock geochemical reference material. We have now verified these results through use of accelerator-based PIXE [1]. In addition we can now predict these effects quantitatively from first principles [2]; examples will be given.

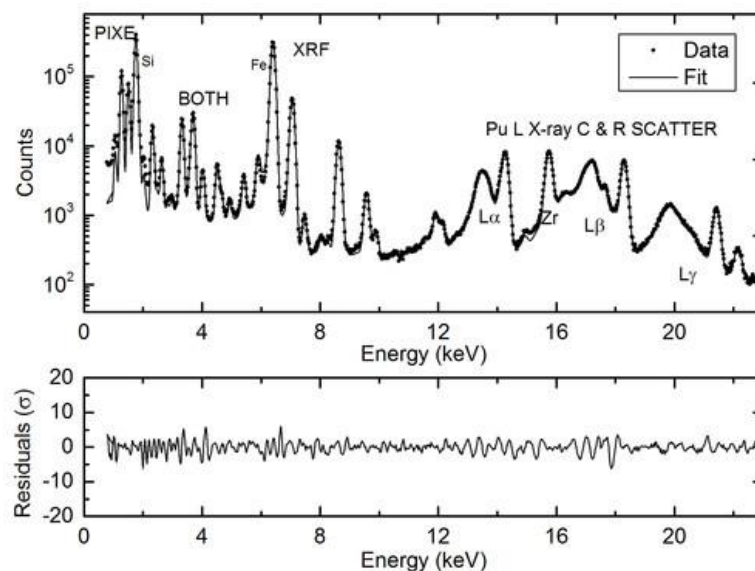


Fig. 1. APXS spectrum of Martian sample with GUAPX fit.

[1] E.L. Flannigan et al., this conference contribution O-10

[2] G.M. Perrett, J.A. Maxwell, J.L. Campbell, submitted to X-ray Spectrometry

I-2: Emission of M-X-rays induced by light and heavy ions

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M-X-rays excited in collisions of heavy charged particles with atoms are of great interest both for fundamental and applied aspects, in particular, for the particle induced X-ray emission (PIXE) technique. In the present paper the emission of M-X-rays induced by light ions, commonly used in PIXE, as well as heavy and highly charged ions will be discussed in context of basic physical processes, experimental aspects and theoretical interpretations. For light ion impact, such as hydrogen $^1\text{H}^+$ and helium $^3,4\text{He}^+$ ions [1] standardly used in PIXE, the direct single Coulomb ionization process dominates, while for fast heavy ions, namely O^{q+} ions [2,3] with moderate $q \leq 8$, respectively, both the electron capture and multiple ionization processes play important role, due to increased Coulomb field of the projectile. The multiple ionization effects modify the X-ray emission, causing the appearance of X-ray satellites, and affect the atomic decay rates by vacancy rearrangement processes. For heavy ions the intra-subshell coupling effects are also important. For slow highly charged ions (HCI), for instance Xe^{q+} with q up to 40, colliding with matter the fast process of neutralization of such ions on surfaces leads to formation of exotic “hollow” atoms. M-X-ray emission from hollow atoms gives access to study both the collision dynamics as well as the structure and relaxation processes of highly excited atoms. The experiments discussed here were performed using different accelerator facilities, namely Van de Graaff ($^1\text{H}^+$ and $^3,4\text{He}^+$ ions) generators [1], tandem [2] and cyclotron [3] (O^{q+} ions) accelerators and the electron beam ion source (EBIS) producing low energy highly charged Xe^{q+} ions. In these experiments both semiconductor Si(Li) or SDD detectors and reflection type von Hamos spectrometers were used with energy resolution of about 100 eV and below 1 eV, respectively, for the M-X-rays studied. In general, the direct Coulomb ionization of the M-shell electrons can be described within the plane wave Born approximation (PWBA), the semiclassical approximation (SCA) and the binary encounter approximation (BEA) while the electron capture process can be treated within the Oppenheimer-Brinkmann-Kramers (OBK) approximation. These approaches can be used to obtain the multiple ionization probabilities within the electron independent model. For the neutralization of highly charged ions at surfaces leading to emission of M-X-rays the classical over-the-barrier model (OBM) can be adopted. In these approaches, generally, the electronic wave functions, from nonrelativistic screened hydrogenic to relativistic self-consistent models, are discussed as well as the electron binding effect, which becomes crucial for low-energy heavy ion impact. Finally, the experimental M-X-ray emission cross sections are interpreted in details within the discussed theoretical approaches.

[1] M. Pajek et al., Phys. Rev. A 73 (2006) 012709.

[2] M. Czarnota et al., Phys. Rev. A 79 (2009) 032710.

[3] M. Czarnota et al., Phys. Rev. A 88 (2013) 052505.

I-3: Anisotropy and alignment in L-shell X-ray emission induced by ion impact

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Electron orbitals in atoms are spherically symmetric only for states with angular momentum quantum number equal to zero. Therefore, when transitions involving electrons from other asymmetrical wave-functions produce X-ray photons, it is expected for their average emission to present also some anisotropy. Moreover, the ion beam incoming trajectory defines a preferential direction, producing an alignment of the electronic orbitals, and as a consequence, of the emitted X-rays. In the present work, a shallow description of this phenomenon, with experimental examples of L X-rays emitted after impact of ^{12}C , ^{16}O , and ^{19}F ions on lanthanoid elements [1]. Additionally, the anisotropic emission must be taken into account when comparisons are done among different X-ray production cross section measurements for asymmetric lines, such as the L_I or the L_α . Following the procedure suggested by Kamiya et al. [2], the necessary corrections are illustrated with a compilation of L_α X-ray production cross sections by ^4He ions impact.

[1] O.G. de Lucio, J. Miranda, Nucl. Instrum. Meth. B 248 (2006) 47-53.

[2] M. Kamiya et al., Phys. Rev. A 20 (1982) 1820-1827.

I-4: Full field PIXE by using an X-ray camera with high-energy and high-spatial resolution

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Analytical applications often require the noninvasive and simultaneous elemental imaging of samples with spatial resolution and chemical sensitivity. Particle Induced X-ray Emission (PIXE) is a well-established technique that can be used for mapping the lateral distribution of chemical elements. Generally, elemental images are obtained in dedicated beam lines by scanning the samples with a primary beam (i.e., low energy protons) focused down to a dimension of few tens of microns or less. In recent times, a Full Field PIXE method has been developed as a novel alternative to the scanning approach [1,2]. A broad beam irradiates the sample at once; X-ray fluorescence induced by the primary radiation is detected through a polycapillary installed in front of a position- and energy-sensitive detector allowing the fast elemental imaging. This work discusses the possibility of performing the FF-PIXE with a commercial and low cost CCD detector coupled to a 1:1 straight shaped polycapillary [2]. This system, named Full Field X-ray Camera (FF-XRC), has been recently developed at the LANDIS laboratory of INFN-LNS and IBAM-CNR in Catania (Italy). The use of a special photon counting technique allowed the use of the device for the energy and laterally resolved X-ray fluorescence spectroscopy. The FF-XRC is operated with an in-house programmed software programmed in Matlab. It controls the device and allows performing on the fly the elemental imaging of samples with a region of interest (ROI) method. At the end of a measurement, it creates the spectra of each pixel allowing to analyze collected datasets with a least square fitting imaging procedure available in external software (for instance in PyMCA). The low cost of the system, compact dimensions and the easy-to-use electronics (only a USB cable and an office PC is necessary to control the instrument) make this device a valid tool for Full Field imaging applications. The FF-XRC camera has been installed in a FF-PIXE end station at the 80° beam-line of the INFN-LNS operated with a low energy proton beam of wide dimensions extracted in the air. The beamline is served by a 13 MV TANDEM accelerator that provides, among others ions, proton beams up to 25 MeV energies. Proton energy can be tuned down to about 5 MeV and a maximum current of 1 mA can be delivered to users for experiments. A sample holder equipped with a 4 axis travel system is positioned at about 14 cm from the Kapton window. This air path allows to degrade proton energy down to a value of about 3 MeV, better suited for performing PIXE measurements on samples. The beam dimension is about 30 mm diameter that fully cover the field of view of the detector. Results of the use of the novel FF-PIXE end station in applications of interest in Cultural Heritage and Geochemistry are presented and discussed.

[1] D. Hanf et al., Nucl. Instrum. Meth. B 377 (2016) 17-24.

[2] F.P. Romano et al., Anal. Chem. 88 (2016) 9873-9880.

I-5: Chemical speciation using WDS in the tender X-ray range

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Wavelength dispersive X-ray spectroscopy (WDS) employing Bragg crystal spectrometers can provide energy resolution on the level of core-hole lifetime broadening of the characteristic emission lines. While WDS has been traditionally used in combination with electron excitation for major and minor element analysis, it has been rarely considered in proton induced X-ray emission (PIXE) trace element analysis mainly due to low detection efficiency. Compared to the simplest flat crystal wavelength dispersive spectrometer the efficiency was enhanced significantly in modern spectrometers employing cylindrically or even spherically curved crystals in combination with position sensitive X-ray detectors. When such spectrometer is coupled to MeV proton excitation, chemical state analysis can be performed extending the analytical capabilities of PIXE technique also towards chemical speciation. In our laboratory the home-built X-ray emission spectrometer employing cylindrically curved-crystal in Johansson focusing geometry [1] is used to perform high energy resolution PIXE (HR-PIXE) in the tender X-ray range (~ 2 -5 keV). In this contribution the general properties, capabilities as well as limitations of HR-PIXE for chemical speciation in the tender X-ray range will be presented together with some typical results highlighting the analytical potential of such analysis in different research areas.

[1] M. Kavčič et al., Rev. Sci. Instr. 83 (2012) 033113.

I-6: PIXE simulation with Geant4

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Atomic Deexcitation simulation has been present since more than 15 years in Geant4. During the years it has been continuously developed, with the extension from photoelectric effect to continuous process, radioactive decay and PIXE.

Regarding PIXE, there are actually a good number of available models among which the user can choose the shells in which a vacancy appears upon interaction with protons or ions. The user can also choose the data used to simulate the deexcitation chain, according to his/her preferences.

Geant4 Atomic deexcitation has been applied and validated in various fields: from astrophysics to medicine and biology, proving to be a useful tool for predictive studies.

A brief overview will be presented on the working principle, the available options and their application and validation results. Foreseen upgrades and development options will be shared with the community for discussion.

I-7: Safe boundaries for analysis of heritage materials

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Certain analytical techniques considered as non-destructive, since no sampling is needed, may cause visible or non-visible, reversible or irreversible changes depending on the material and the experimental parameters. To monitor, understand and mitigate radiation-induced alterations which may be occurring during analyses of heritage materials is one of the Joint Research Activity tasks of the IPERION CH (*Integrated Platform for the European Research Infrastructure ON Culture Heritage*) EU H2020 project. Fourteen research groups are involved in the task, representing both large scale facilities (synchrotron, ion beam laboratories, research reactor) and smaller scale X-ray and laser instrumentations including mobile devices. The consortium members are open to collaborations with the wider analytical community, too, as we intend to develop a common approach to establish the safe operating boundaries for our techniques. In this talk, we will focus mainly on organic materials under ion beam irradiation. Organic materials, generally, are sensitive to high-energy ionizing radiations. The work has started with polymers which are used in the form of coatings, lacquers, and waxes in the cultural heritage field. The ion irradiation leads to the degradation of polymeric chains, chemical bond cleavage, forming of free radicals and release of gaseous degradation products. The type and degree of the degradation strongly depend on the chemical groups attached to the main chain of the polymer. Utilizing the knowledge of ion irradiation induced processes gathered from investigating polymers can provide useful information about how to proceed with other types of materials, as the next step envisaged is the systematic investigation of parchment samples.

[1] L. Bertrand et al., *Trend. Anal. Chem.* 66 (2015) 128-145.

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I-8: DAPNe-IBA : A new tool for the IBA community?

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With the emergence of MeV-SIMS and micro calorimeter X-ray detectors, our community has recently been presented with new opportunities for providing chemical speciation of samples, which will be highly complementary to PIXE. Whilst these techniques significantly increase the analytical power of IBA, their ability to provide trace molecular information is limited. Even for MeV-SIMS, the fragmentation of analytes precludes the detection of large molecules.

Direct Analyte Probed Nano Extraction [1] Mass Spectrometry (DAPNe-MS) is a method of selecting and removing a small quantity of material from a surface, using a capillary tip attached to a nanomanipulator. The extracted material is then introduced into a mass spectrometer via nanoelectrospray ionisation. The technique has achieved sensitivity at the attogram level and is particularly suited to picking up trace materials from surfaces. Unlike MeV-SIMS, there is minimal fragmentation, and this provides better selectivity.

The University of Surrey has been awarded a grant from the UK Electronics and Physical Sciences Research Council to install a system for Direct Analyte Probed Nano Extraction (DAPNe) to be used in conjunction with ion beam analysis. The basic concept is to provide a trace element or secondary ion image using IBA techniques and then select points of interest to be studied in greater detail using DAPNe. In this presentation, we will describe the technical set up of the recently installed system, show some preliminary results and discuss situations in which DAPNe-IBA can add significant value to analytical science.

[1] K. Clemons et al., J. Am. Soc. Mass Spectrom. 25 (2014) 705-711.

I-9: Heavy ion induced X-ray production cross sections and simultaneous heavy ion PIXE analysis with other IBA methods

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Particle induced X-ray emission (PIXE) with protons is routinely used for trace analysis. The emission of X-rays as a result of the atomic excitation with protons and alpha-particles has been widely studied and can be calculated from theoretical models with fair accuracy. When heavier ions are used as bombarding particles, the processes involved are more complex and their effects have not been fully described neither by theories nor experiments. However, there is an increasing interest in the application of heavy ions for analysis with particle induced X-ray emission, as they can be used simultaneously with other heavy ion based techniques like elastic recoil detection analysis (ERDA) and secondary ion mass spectrometry at MeV energies (MeV-SIMS). The development of the MeV-SIMS, promoted by IAEA's coordinated research project (F11019) "Development of the molecular concentration mapping techniques using MeV focused ion beams", combines MeV-SIMS with other ion beam techniques, especially with heavy ion PIXE. Lack of the experimental heavy ion PIXE cross section data limits effective use of this method for analysis and also hinders development of the theoretical X-ray emission cross section models. One objective of the CRP is to produce database of cross sections for PIXE analysis and provide means for development of theoretical models.

As targets for X-ray production cross section measurements, atomic layer deposited (ALD) thin oxide, nitride and pure element films on silicon or carbon substrate were selected. Thicknesses of the films were from 30 nm to 150 nm. Thicknesses of the targets were selected so, that they can be considered as thin targets in the cross section measurements, but still thick enough to give satisfactory count rate for the X-ray and ERDA detectors. Elemental compositions and areal densities of targets used in the experiments were measured by TOF-ERDA.

X-ray production cross sections for Ti, Cu, Zr, Nb, Ru, Ta and W induced by C, O, Ti, Cl, Br and I ion beams within the energy range of 15-55 MeV have been measured at the 5 MV tandem accelerator of University of Helsinki. The X-ray spectra were measured simultaneously with TOF-ERDA spectra, which allows accurate beam fluence determination. Determined ionization cross sections are compared with the predictions of the ECPSSR theory. As an example, heavy ion PIXE spectrometry combined with TOF-ERDA measurement is demonstrated.

I-10: The application of PIXE and complementary nuclear techniques to aerosol studies: the LABEC experience

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At the 3 MV Tandatron accelerator of the LABEC laboratory of INFN (Florence, Italy) an external beam facility is fully dedicated to PIXE-PIGE measurements of elemental composition of atmospheric aerosols. All the elements with $Z > 10$ are simultaneously detected by PIXE typically in one minute. This setup allows us an easy automatic positioning, changing and scanning of samples collected by different kinds of devices: long series of daily PM_{10} samples can be analyzed in short times, as well as size-segregated and high time-resolution aerosol samples.

Thanks to the capability of detecting all the crustal elements, PIXE-PIGE analyses are unrivalled in the study of mineral dust: as a consequence they are very effective in the study of natural aerosols, like, for example, Saharan dust intrusions. Among detectable elements there are also important markers of anthropogenic sources, which allow effective source apportionment studies in polluted urban environments by the use of multivariate method like Positive Matrix Factorization (PMF).

Examples regarding recent monitoring campaigns, performed in urban and remote areas, both on a daily basis and with high time resolution (hourly samples), as well as with size selection, will be presented. The importance of the combined use of Particle Induced Gamma Ray emission technique (PIGE) and of other complementary (nuclear or not) techniques will highlighted.

ORAL PRESENTATIONS

O-1: Elemental mapping using XFM with the Maia detector array and the nuclear microprobe with a single-detector: exploiting their individual merits

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The X-ray Fluorescence Microscopy (XFM) beamline at the Australian Synchrotron, equipped with the Maia large solid-angle detector array composed of 384 planar 1 mm² Si-PIN detector elements [1], is at present the most high-throughput X-ray fluorescence mapping system worldwide. The Kirkpatrick-Baez (KB) mirror end-station provides a monochromatic beam in the 4–20 keV energy range, focused to ~1 micrometer spot. Elemental maps can be acquired over areas of many square centimeters with the stage range of 150 mm x 100 mm. This capability enables capturing datasets that can exceed 108 pixels within hours.

The nuclear microprobe at iThemba LABS uses the classical OM150 triplet system manufactured by Oxford Microbeams. Ion beam is scanned using an OM40e dual scan amplifier. PIXE elemental mapping is typically performed using a 3 MeV proton beam and a single Si(Li) or HPGe detector, complemented by simultaneous registration of proton backscattering spectra by an annular Si surface barrier detector. Although the best attainable beam spot is 1.6 μm x 0.6 μm [2], routine elemental mapping is performed with beam sizes in the order of 3 x 3 μm². The maximum scan size for 3 MeV protons is ~2.7 mm x 2.7 mm, and the number of pixels is limited to 16384 (128 x 128). Scanning larger structures requires stitching of maps.

Both facilities use the GeoPIXE software for processing of the data streams and for generating elemental maps and spectra using the Dynamic Analysis (DA) method [3].

Recently, an extensive study with the aim of understanding nickel biopathways in the roots of hyperaccumulating and non-hyperaccumulating genotypes of the plant *Senecio coronatus* has been undertaken at the XFM beamline, employing 2D elemental mapping, micro-XRF tomography and confocal fluorescence XANES. Some of the dehydrated root sections were earlier analysed using the nuclear microprobe at iThemba LABS. In addition, 2D elemental maps were obtained for cross-sections of seeds of the Zn and Cd hyperaccumulating plant *Arabidopsis halleri* for which micro-PIXE maps existed. These two test cases present an excellent opportunity to compare the two techniques, and to explore their individual merits and limitations.

- [1] C.G. Ryan et al., X-Ray Nanoimaging: Instruments and Methods, Proc. SPIE 8851 (2013) 88510Q.
- [2] V.M. Prozesky et al., Nucl. Instrum. Meth. B 104 (1995) 36-42.
- [3] C.G. Ryan, Int. J. Imag. Syst. Tech. 11 (2000) 219-230.

O-2: A review of potentialities and perspectives of proton induced quasi-monochromatic X-rays in fundamental studies and analytical applications

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The use of ion induced quasi monochromatic X-rays has been envisaged since 1983 [1] in various analytical applications, including the selective XRF analysis of bulk samples but also for elemental imaging purposes through the formation of X-ray microbeams. The ideas presented in this pioneer work were motivated by the benefits offered by the various physical processes that govern the energetic ion-induced emission of X-rays by solid targets: the high inner-shell ionization cross sections and generally the very low emission of bremsstrahlung radiation following the ion-impact (two-three orders of magnitude lower compared to electron-impact). Various groups have mostly employed proton induced quasi monochromatic X-rays for improving detection limits of trace elements [2]. Proton induced X-ray microbeams have been generated utilizing different set-up configurations and X-ray focusing optics such as polycapillary X-ray half lens [3] and Fresnel zone plate [4] to achieve selective elemental imaging capabilities [3] and consistent dose delivery during irradiation of a single cell layer [4], respectively. On the other hand, Sokaras et al. [5,6], have demonstrated that the quasi monochromatic X-ray beams can be also reliably applied for fundamental studies, such as the measurement of absolute Resonant Raman Scattering cross section [5] and the L-shell cascade emission [6]. These potentialities, but also additional perspectives which can be triggered by optimizing features of the set-up configuration and experimental parameters will be presented and discussed.

- [1] L. Grodzins, P. Boisseau, IEEE Trans. Nucl. Sci. 30 (1983) 1271-1275.
- [2] D. Sokaras et al., Rev. Sci. Instr. 83, (2012) 123102.
- [3] K. Ploykrachang et al., Int. J. PIXE 23 (2013) 1-11.
- [4] D. Harken et al., Nucl. Instrum. Meth. B 269 (2011) 1992-1996.
- [5] D. Sokaras et al., Phys. Rev. A 81 (2010) 012703.
- [6] D. Sokaras et al., Phys. Rev. A 83 (2011) 052511.

O-3: Comparison of proton and carbon PIXE at the same ion velocity

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The Plane Wave Born Approximation (PWBA) ionisation theory for heavy ions predicts that the cross section for an inner shell vacancy should vary as (Z_1^2/Z_2^4) where Z_1 is the atomic number

of the ion and Z_2 is the atomic number of the target. For protons with $Z_1=1$ and carbon ions with $Z_1=6$ this should produce ionisation cross sections 36 times that of the proton cross sections at the same ion velocity (MeV/amu) for a given target (Z_2). This could be significant for carbon-PIXE versus proton-PIXE for elemental characterisation if the secondary electron and Compton scattered backgrounds for the two methods at the same velocity did not completely swamp this apparent increase. More recent ionisation theories like the ECPSSR theory make corrections to these cross sections for ion energy loss (E), Coulomb deflection of the ion (C), perturbed stationary states of the orbiting electrons (PSS) and electron relativistic effects (R). The ECPSSR theory is used extensively in PIXE analysis. It shows this (Z_1^2) increase with ion atomic number for carbon versus proton PIXE is not constant for the same target but decreases by around a factor of two for ion energies from 3 MeV/amu to 1 MeV/amu for the K and L shells typically used for PIXE. This paper will look in detail at the experimental differences between the proton-PIXE versus carbon-PIXE spectra for 0.5 MeV/amu to 3 MeV/amu ions for a range of K and L lines in standard targets. It will consider in detail contributions to the signal to noise (S/N) for the two situations, the multiple ionisation effects on fluorescence yields and X-ray line peak splits and shifts that are relevant for heavy ions. It will come up with a conclusion as to whether carbon-PIXE can be superior to proton PIXE for elemental characterisation for elements from aluminium to lead.

O-4: Investigation on MeV C-ion PIXE cross sections

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In development of heavy-ion particle induced X-ray emission (PIXE) technology which has been demonstrated to be superior to conventional light proton PIXE owing to larger cross sections, MeV carbon-ion PIXE was tested and studied for availability and cross sections. The relatively low ion energy range around 1 MeV for C-ion PIXE had never been investigated before. In the work, C-ion PIXE at ion energy around 1 MeV was firstly tested and demonstrated to be available at our 1.7-MV tandem accelerator and its beam line. In measurement of the cross sections for the MeV C-ion PIXE, 0.8, 1.0 and 1.2 MeV C-ion beams were applied to analyze materials of Si, Fe, Cu, Zn and Au and the spectra were compared with spectra of 1.0- and 2.0-MeV proton PIXE. Results showed that at the same low ion energy, 1-MeV C-ion PIXE yields were significantly higher by orders than those of proton PIXE which were actually negligible, demonstrating the former significantly more sensitive than the latter. With the 2-MeV proton PIXE cross sections used as the reference, the MeV C-ion PIXE cross sections were calculated. An interesting trend of the cross section against the atomic number Z showed that the cross sections of MeV C-ion PIXE compared with those of proton PIXE for the analyzed materials had a transition around Cu. For lower Z the former was higher than the latter, while for higher Z the former was lower than the latter, indicating MeV C-ion PIXE more sensitive in detecting lower- Z elements. Detailed experimental and calculating methods as well as discussions are reported in the presentation.

O-5: Projectile and target X-rays induced by heavy ions

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Traditional PIXE (Particle-Induced X-ray Emission) experiments are usually performed with MV electrostatic accelerators and a few MeV protons are routinely used worldwide. The choice for protons over alpha particles or even over heavier ions relies on two points: i) simplicity of the experimental setup; ii) availability of an extensive database for the X-ray production cross section induced by protons and, to a lesser extent, by alpha particles. In the past years, a new concept in the ion beam analysis (IBA) community has emerged, namely to use of different ion-based techniques simultaneously and perform a self-consistent analysis. This has been termed as “Total IBA”. In this scenario, the combination of techniques like MeV-SIMS (Secondary Ion Mass Spectrometry) and PIXE would enhance the overall analytical capability covering the simultaneous characterization of elements and compounds. However, PIXE experiments with ions heavier than protons require the knowledge of X-ray production cross sections. In this work we discuss the potentialities and pitfalls of experiments employing heavy ions in the analysis of a thin titanium film deposited over thick vitreous carbon. In particular, we focus our attention to the chlorine X-rays induced during ion impact on the target. The measurements were carried out at the Ion Implantation Laboratory of the Physics Institute (UFRGS) using a 3 MV Tandatron accelerator. Experiments were performed with 6 – 10 MeV Cl ions. A Si(Li) detector positioned at 135° with respect to the beam direction was used to detect X-rays generated in the target. The results of X-ray yields show an increasing trend as a function of the ion energy. Possible reaction mechanisms are discussed.

O-6: Theoretical detection limit of PIXE analysis using 20 MeV proton beams

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In the PIXE analysis, the beam of 2-3 MeV proton is usually used because the ratio of characteristic X-ray production cross section to continuous X-ray radiation cross section is large in this energy region [1]. However, the X-ray cross section decreases rapidly with decrease in proton energy particularly in the energy region less than 3 MeV and the energy loss through a target is not neglected in comparison to the incident energy, therefore, a very thin target is required for accurate quantitative analysis. In consideration to the benefit of low stopping power of protons with the energy up to 100 MeV, A. Denker et al suggested a high energy PIXE analysis and applied it to analyze art and archaeological objects [2].

Cyclotron accelerators in PET centers are used only to produce positron emission short life radioisotopes. We consider the application of Cyclotron proton beams to the PIXE analysis. Here, we theoretically estimate the detection limit of PIXE analysis using 20 MeV proton beams. The energy loss of this proton energy is 0.1-3 MeV for Japanese paper of thickness of 0.1 mm

and an accurate quantitative analysis can be performed for ancient document samples. For the high energy PIXE analysis, it is desired to use an X-ray detector of which the performance is low sensitive for gamma rays and neutrons produced in nuclear reactions, but high sensitive for X-ray detection. Recently, a silicon drift detector with a high energy resolution of 125 eV at Mn K-X-ray, the detection depth of 0.5mm which permits the detection of X-rays of 1-25 keV (Na K-X-ray – Ag K-X-ray and Cu L-X-ray – U L-X-ray), and the detection area of 17 mm² (AmpTek 123SDD) is commercially available. We predict to use this detector in our PIXE analysis. Main components of continuous backgrounds in 20 MeV proton PIXE spectrum are the quasi-electron bremsstrahlung [3] in the region of 0-10 keV and the secondary electron bremsstrahlung in the region of 10-40 keV. In such high energy proton case, we can estimate the inner shell ionization cross sections without considering the binding energy effect and the Coulomb deflection effect. In accordance to the knowledge of statistics, a signal peak on X-ray energy spectrum can be recognized when signal counts are larger than 3 times of root square of signal counts+background counts within the width of detector resolution (FWHM). Based on this idea, we can estimate the detection limits of PIXE analysis for each element. In the case of ancient document of thickness of 0.1 mm, the detection limit of iron element is estimated by 0.9 ppm (mg/kg) under the experimental condition of beam currents of 10 nA and measuring time of 1 minutes.

PET center is now in construction at Rokkasho village, Aomori prefecture in Japan. The 20 MeV cyclotron of this center will be used to PET diagnosis, neutron imaging in nondestructive testing and PIXE analysis.

[1] K. Ishii, S. Morita, Nucl. Instrum. Meth. B 34 (1988) 209-216.

[2] A. Denker et al., Nucl. Instrum. Meth. B 219-220 (2004) 130-135.

[3] T.C. Chu, K. Ishii et al., Phys. Rev. A 24 (1981) 1720-1725.

O-7: High energy PIXE: new K-shell ionization cross sections and comparison with theoretical values calculated using ECPSSR

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The inner shell ionization process induced by incident charged particles can be described by different theoretical models: Plan Wave Born Approximation (PWBA), Perturbed Stationary State theory with Energy loss, Coulomb correction and Relativistic effects (ECPSSR) [1]. Over the years, a lot of experimental data have been reported in the literature for K-shell ionization cross sections at low-energy (below 10 MeV) and are in good agreement with ECPSSR theory [2].

However, there is a lack of experimental data at higher energies (up to 100 MeV) which do not allow determining the accuracy of these models in this energy range. In addition, these information are necessary to perform quantitative HE-PIXE (High Energy Particle Induced X-ray Emission) [3].

At the Arronax facility, we have set an experiment to measure the K-shell ionization cross sections with high proton beams (up to 68 MeV). A particular attention has been paid on a good

control of the experimental parameters, particularly the characterization of the efficiency of our High-Purity Germanium X-ray detector in the energy range of 3-80 keV. A special study has been made to select the most accurate physical parameters from the literature data, like the K-shell fluorescence yields and the X-rays emission rates for K_{α} and K_{β} , necessary to convert the K- X-ray production yield to the K-shell ionization cross section.

The first experiment has been conducted to measure these K-shell ionization cross sections for gold with proton energy of 68 MeV. The outcomes of the experiment are in good agreement with the existing data (Fig. 1). Using the same set-up and techniques, a set of new data for K-shell ionization cross section for various elements (titanium, copper, silver and gold) at high proton beam energies (30, 42, 54 and 68 MeV) have been measured.

During this talk, I will present these new dataset and compare them with the theoretical calculations. A particular care will be made to make an overview of existing models in this energy range and of the description of our experiment (beam, detector and targets).

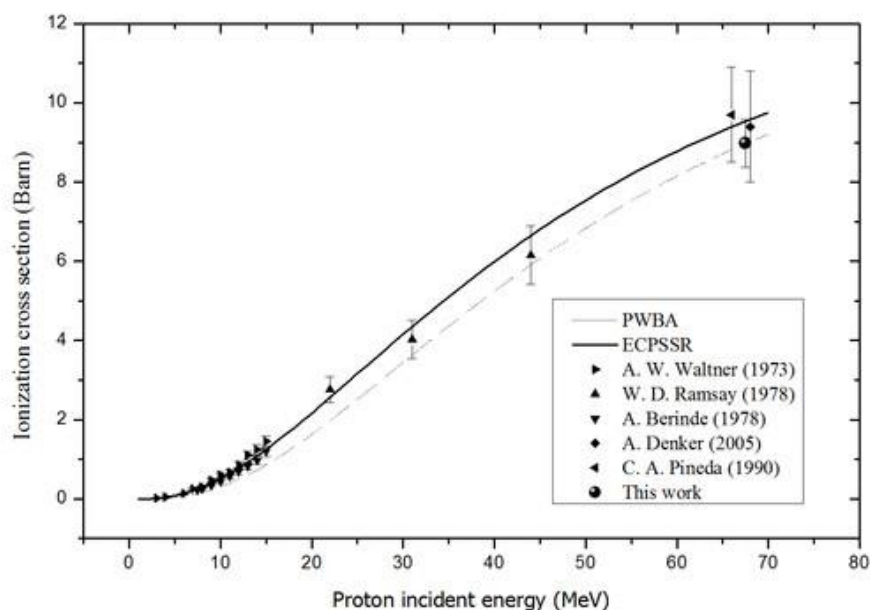


Fig. 1. K-shell ionization cross sections of gold by for 1-70 MeV protons (high energy) compared with theoretical values of PWBA and ECPSSR data from different reference.

- [1] W. Brandt et al., Phys. Rev. A 23 (1981) 1717-1729.
- [2] S.Ouziane et al., Nucl. Instrum. Meth. B 161-163 (2000) 141-144.
- [3] D. Ragheb et al., J. Radioanal. Nucl. Ch. 302 (2014) 895-901.

O-8: High Energy PIXE: a powerful tool to characterize multi-layers thick samples

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High-energy PIXE (HEPIXE) is suitable to analyze thick target placed in air due to the large penetration range of energetic ions in matter and their ability to excite the energetic K X-rays.

In addition, energetic ions get the advantage of having a low energy loss per length unit reducing radiation damages. All these points make this method interesting for application in the field of cultural heritage in general and especially for the characterization of ancient paintings with several superposed layers [1] and the analysis of metallic archaeological objects [2] which can then be investigated without removing the patina layer on the surface.

In our previous work on HEPIXE, we have demonstrated that it is possible to provide the thicknesses, the sequences of the layers and also qualitative results about the presence of several layers containing the different elements in some specific samples. Those results were obtained using the measured intensity ratios $K\alpha/K\beta$, $L\alpha/L\beta$ and $L\alpha/K\alpha$ [3] resulting from the excitation of the sample. However, in real applications of multi-layer analysis (paintings, coins) [1,2], hypothesis on the sample composition are required which are not always easy to infer. Moreover, there might be some layers composed of light elements, located at the surface or in the bulk that are invisible to the detection system due to the strong attenuation of the emitted low energy radiation in the air between the sample and the detection system or within the sample itself (self-attenuation). The presence of those hidden layers makes the analysis of such samples difficult.

We present in this work a new method that allows to get rid of these issues and to analyze samples containing hidden layers.

Our work has been done within the platform for high energy PIXE developed at the ARRONAX cyclotron (Nantes-France) with proton beam of 68 MeV. The experimental set-up includes a High Purity Germanium detector for X-ray detection and an automated sample holder that can be tilted with respect to the incident beam direction. Our newly developed method is based on the ratio variations of $K\alpha/K\beta$, $L\alpha/L\beta$ and $L\alpha/K\alpha$ with respect to the angle between the target and the detector. These ratios, when properly considered, allow the determination of the thicknesses and the sequences of the layers of a given sample even when hidden and/or repeated layers are present (complex multi-layer samples). In this presentation, we will present both the theoretical considerations and results coming from several experiments with known multi-layer samples (simple and more complex). This opens new applications for HEPIXE and constitute a step toward the determination of the concentration profile of an element as a function of the depth in thick samples.

[1] A. Denker et al., Nucl. Instrum. Meth. B 213 (2004) 677-682.

[2] A. Denker et al., Nucl. Instrum. Meth. B 226 (2004) 163-171.

[3] D. Ragheb et al., J. Radioanal. Nucl. Ch. 301 (2014) 895-901.

O-9: Study of geological samples from Azores ocean floor by High Resolution and High Energy PIXE

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Samples from the Atlantic ocean floor in the Azores region were irradiated at High Resolution High Energy (HRHE)-PIXE setup of C2TN [1] using proton and oxygen beam of 3 MeV and 6 MeV respectively. Simultaneous spectra were collected using both the CdTe and the X-ray microcalorimeter spectrometer (XMS) detectors of the facility. M, L and low energy K lines are visible on the XMS spectra while L and K lines from medium and high Z elements are present in the CdTe detector spectra. Spectra were analysed both qualitatively for major comparison. As shown in the figure below differences between samples are clearly seen in the 20 to 45 keV energy region. One of the samples showing a possible presence of Cd. Spectra were then analysed in detail using the DT2 code [2]. In this work the results obtained are discussed, and the advantages of the XMS and of using it simultaneous to the CdTe detector covering an energy window of more than 100 keV are shown.

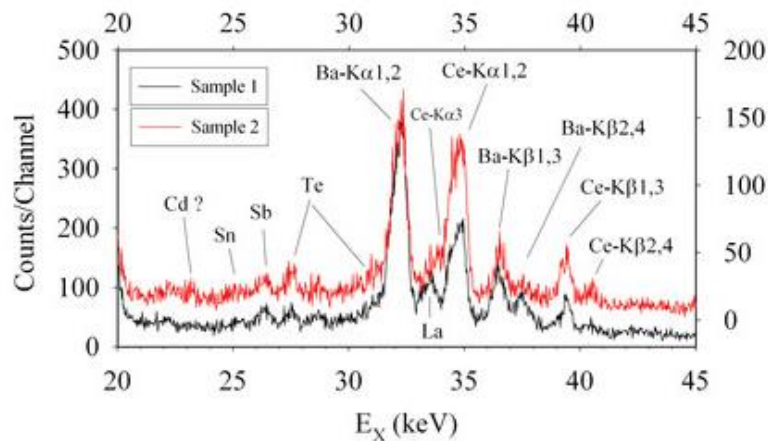


Fig. 1. Differences between two samples studied are clear in the medium Z elements K shell spectra. Sample 2 shows more elements than sample 1 including the possible presence of Cd.

[1] P.C. Chaves et al., Nucl. Instrum. Meth. B 318 (2014) 60-64.

[2] M.A. Reis et al., Nucl. Instrum. Meth. B 318 (2014) 65-69.

O-10: Mineral phase effects in PIXE analysis of igneous rocks

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The Alpha-Particle X-ray Spectrometer (APXS) on board the Mars Science Laboratory rover, Curiosity, performs non-destructive *in situ* measurements to determine elemental concentrations in rock and soil samples on Mars. Using a ^{244}Cm source and a silicon drift detector, the APXS combines XRF and He-ion PIXE. Calibration was achieved using a suite of terrestrial geochemical reference materials (GRMs), provided as powders by various suppliers. Campbell et al. [1] reported the ratio (R) between APXS-measured and supplier's concentrations in these GRMs. In mono-mineralic samples, which follow the assumption of matrix homogeneity, the R-values were close to 1.0. In multi-mineralic rocks deviations become apparent for the lightest elements Al, Mg and Na: as Fe concentrations increase, the Al and Na R-values systematically rise from unity; for Mg the opposite is the case. We refer to these

effects, which arise from the mineral structure's violation of the homogeneity assumption, as mineral phase effects (MPEs). Using the APXS, Si, Al, Mg and Na are excited exclusively by PIXE. The shallow PIXE interrogation depth (a few microns), suggests that single minerals produce the X-rays of these light elements instead of the "average rock matrix" assumed by homogeneity. We have used a broad-beam PIXE system to emulate the APXS, whose sampled area is 2.25cm^2 . Using our 2.5 MeV APXS emulation beam line, MPEs were determined for Na, Mg and Al in basalt, andesite, syenite and mugearite GRMs. The results agreed well with the APXS results. Systematically low values of Fe concentrations were additionally present in PIXE measurements. The necessary normalization of oxide concentrations to 100 wt% accounts for a portion of this decrease. It causes the overall excess concentration of the lightest elements to force a decrease in those of high Z elements such as Fe. Scanning Electron Microscopy (SEM) and laser diffraction performed on the basalt GRMs showed wide particle size ranges. SEM with EDX on basaltic materials determined that Fe and Mg within the sample are present in the mafic mineral grains which are several magnitudes smaller than the Na- and Al-bearing felsic minerals. In PIXE, 90% of the detected X-rays arise within a depth of around 25 microns. Since some of the Fe-bearing pyroxene grains are smaller than this depth, some protons will penetrate them, resulting in the remaining observed Fe concentration decrease.

[1] J.L. Campbell et al., Space Sci. Rev. 170 (2012) 319-340.

O-11: Full-field PIXE imaging: Multi-frame super-resolution to overcome optics pattern and imaging-based resolution limitations

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The combination of a pnCCD-based detector (264 x 264 pixels) with a polycapillary X-ray optics was installed and examined at HZDR [1]. The set-up is intended for PIXE imaging with protons (2-4 MeV) to survey large, flat/polished geological samples with respect to their (trace) elemental composition. In the standard configuration a 1:1 polycapillary X-ray optics (78 mm length, 20 μm capillary diameter) is used to guide the emitted photons towards the pnCCD-chip divided into nearly 70000 pixels. Their dimensions of $48 \times 48 \mu\text{m}^2$ cause a native lateral resolution of about 100 μm . By applying dedicated sub-pixel algorithms to recalculate the footprint of the photon's electron cloud in the chip [2], this limitation can be bypassed and the lateral resolution is then mainly determined by the capillary's diameter.

Nevertheless, all images gathered with this kind of set-up from a single measurement are superimposed by the optics pattern. The optics' capillaries are grouped in hexagonal bundles during the fabrication process and these bundles are grouped together again. This process results in a reduced transparency in the regions where the bundles are joint making the hexagonal pattern visible. This influence can be (largely) removed by combining several short measurements with slightly shifted positions. The optics pattern is averaged out and in addition the lateral information (shift-lengths) can be used to further improve the resolution limit beyond the pixels'/capillaries' dimensions. The total measurement time can be kept almost

similar by dividing the single measurement time by the number of “shots” without losing statistics/sensitivity.

Results from descriptive image-sets of first test-measurements (Fig. 1) will be shown to demonstrate the potential of this technique for full-field PIXE imaging.

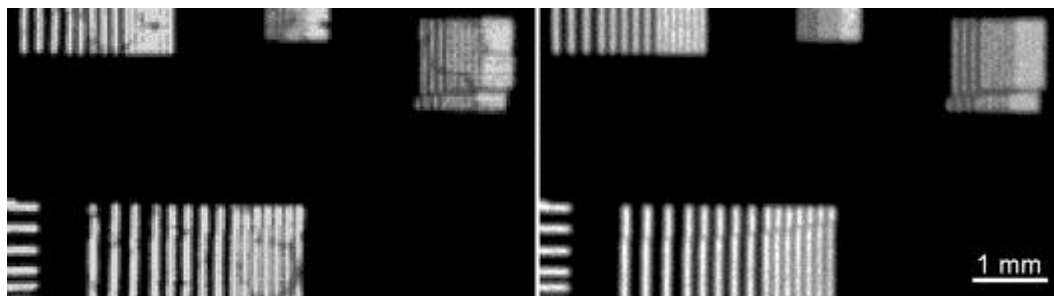


Fig. 1. Comparison of a standard image (left) of Cu-strips with a multi-frame image (right), composed of 26 images (equal total measurement time).

[1] D. Hanf et al., Nucl. Instrum. Meth. B 377 (2016) 17-24.

[2] S.H. Nowak et al., X-ray Spectrom. 44 (2015) 135-140.

O-12: Development of a 3D positioner for the PIXE mapping of non-flat objects

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PIXE mapping is routinely applied for the acquisition of elemental maps on flat targets, but it is generally restricted to the scanning of flat surfaces. The present communication reports the development of a new type of sample holder capable to carry out PIXE mapping on artefacts having a high relief which are common in heritage applications. The prototype is developed in the framework of the IPERION [1] European program and is primarily designed for the AGLAE external beam end-station. It will be eventually adapted to the X-ray beam line PUMA at the SOLEIL synchrotron and the neutron beam lines of the Budapest Neutron Centre.

The new positioner is based upon a machine vision scheme in three steps: 1) the target area to be mapped is digitized using a 3D optical scanner to produce a high resolution 3D mesh model of the curved surface. 2) The sample is moved to a motorized sample holder built upon a combination of X/Y/Z translations and a hexapod for rotations around the X and Y axes. During the scan, the system dynamically brings the local target surface at the focal point of the proton beam and tilts it to keep it perpendicular to the beam axis. The scanned surface is virtually divided in small flat-considered square tiles (less than 1 mm²) that are eventually stitched together to form the entire surface. PIXE data are collected in list mode while the beam is raster scanning the surface of each tile at high speed using magnetic coils. 3) Acquired PIXE maps will be visualised using a specially developed interactive interface projecting the acquired data on a 3D model. The positioner can map areas of 100 mm x 100 mm max on objects weighing a few kg with max curvature angles of 30° with a positioning resolution better than 10



μm . The mapping of larger areas is possible, with smaller angles. Small surface details presenting higher curvatures such as grooves or protrusions which cannot be compensated by the positioner will be taken into account off line using the surface orientation calculated from PIXE images recorded from different viewpoints using PIXE detectors in the four quadrants [2] (see contribution in these proceedings [3]).

Fig. 1. Positioner prototype in construction.

[1] www.iperion-ch.eu

[2] L. Pichon et al., Nucl. Instrum. Meth. B 318 (2014) 27-31.

[3] L. Pichon et al., this conference contribution O-13

O-13: Surface microtopography by stereo-PIXE for enhanced map processing

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The present contribution shows the combined use of the elemental maps obtained by PIXE detectors to determine each pixel orientation in order to correct the PIXE/BS data processing. Indeed, the recent development on the external beam end-station of the AGLAE facility enables to perform easily fast maps with PIXE, PIGE, RBS and IBIL methods on art objects [1]. Four EDX detectors placed at 90° from each other at an angle of 50° from the beam direction produce four elemental images. The pixel size can be set down to the beam diameter ($20\ \mu\text{m}$).

The mapping is realized by combining a fast magnetic deflection of the beam with the horizontal and vertical mechanical movement of the target. However, the present design is adapted to the mapping of flat targets. The mapping of large object with high curved shape is tackled by the development of a multi-scale 3D positioning system [2]. However, the mechanical positioning will only correct the global object orientation at a large scale. This contribution shows the use of the four PIXE images taken from different viewpoints to determine the sample surface microtopography at small scale ($< 1\text{mm}^2$). The four images are combined using the photometric stereo algorithm that produces a map containing the surface orientation of each pixel. This map will be used during the data processing of PIXE map by the TrauPIXE software or by DataFurnace for RBS analyses to take in account the geometrical parameter in the calculation of the elemental composition or layer thicknesses. In addition, the reconstruction algorithm can also provide the 3D topography of the object surface.

The usefulness of this development will be illustrated on the relief decoration of a gold artefact.

[1] L. Pichon et al., Nucl. Instrum. Meth. B 318 (2014) 27-31.

[2] T. Calligaro et al., this conference contribution O-12

O-14: High resolution K X-ray spectra of selected silicates induced by MeV proton and carbon micro-beams

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We developed a high resolution wavelength dispersive X-ray spectrometer based on the flat diffraction crystals for use with focused ion beams [1]. The spectrometer was incorporated within a dedicated vacuum chamber positioned at the ion-microprobe beam line. We have on disposal three diffraction crystals: (i) LiF(110) for the X-ray energy range between 4.7 and 8.4 keV, (ii) PET (polietilen tereftalat) for the X-ray energy range between 1.6 and 3 keV, and (iii) ADP (ammonium dihydrogen phosphate) for the X-ray energy range between 1.2 and 2 keV.

Recently we used the spectrometer to study ion beam induced chemical effects in silicon and its selected binary compounds [2]. In this work we extended the study to selected silicates. We measured high resolution Si K α and K β X-ray spectra of three silicates: feldspar, kyanite and tourmaline. The spectra have been measured with 2 MeV protons (K α and K β regions) and 20 MeV carbon ions (only K α region). The results obtained for peak relative intensities were analysed and compared with the related relative intensities obtained from Si, SiC and SiO₂.

[1] S. Fazinić et al., Nucl. Instrum. Meth. B 363 (2015) 61-65.

[2] I. Božičević Mihalić et al., J. Anal. Atom.Spectrom.31 (2016) 2293-2304.

O-15: Continuous IBIL analysis and imaging for visualization of chemical composition and modification occurred in microscopic scale targets

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With a beneath of confocal optics and sensitive detectors, ion beam induced luminescence (IBIL) analysis could be used as chemical composition analysis of organic and inorganic targets [1,2]. In previous studies, IBIL was combined with external micro-PIXE and used for the recognition of biological contaminant in microscopic particles [3]. Under 3 MeV proton microbeam exposure, significant degradation of IBIL signals was observed at typical wavelength range for nicotinamide adenine dinucleotide (NADH) and riboflavin. It was experimentally evaluated by using organic standards of NADH, riboflavin, and a polycyclic aromatic hydrocarbon (PAH), each chemical composition has different decay scheme in peak intensity of IBIL. At the same time there could be also observe the changes in chemical composition (modification) occurred during the beam irradiation through the generation of new peaks in IBIL spectra. Using these properties, IBIL can be used as in-situ monitoring tool for chemical modification driven by proton microbeam irradiation.

In this study, we demonstrated continuous analysis and imaging of IBIL from microscopic beam scanning region. 3MeV proton microbeam from Single-ended accelerator at QST/Takasaki was employed for the continuous IBIL measurement. Multi-channel photon-counting mode photomultiplier array was used for the multi-wavelength IBIL imaging with effective wavelength range from 300-750 nm and wavelength resolution of 14 nm. Fig. 1 show the result of IBIL imaging for pre- and post-irradiation of microbeam scan on uniformly distributed PAH powder sample. Simplified beam scanning with square pattern of $200 \times 200 \mu\text{m}$ was used to induce modification in target. By comparing the images of IBIL, the irradiated region emits different IBIL wavelength after the irradiation. These results suggest that continuous measurement of IBIL is capable to visualize the changes in chemical composition occurred during focused beam irradiation used in either analytical or lithographic applications.

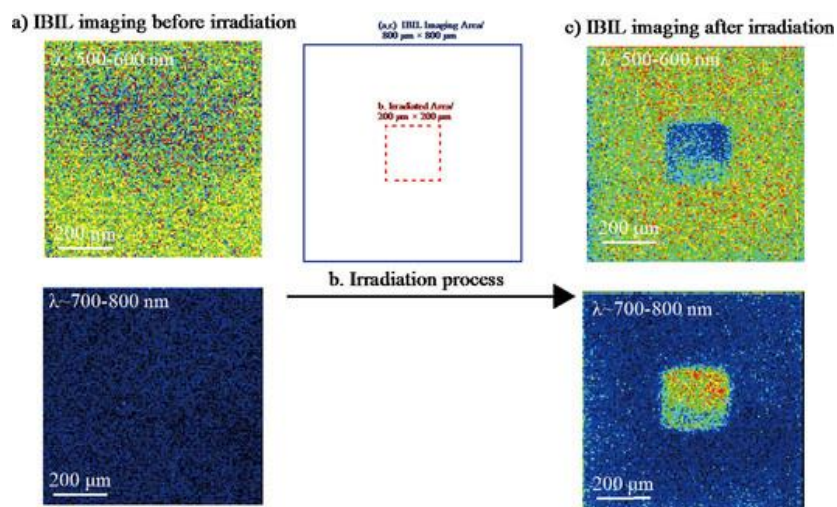


Fig. 1. Comparison of band-pass IBIL imaging of PAH target before and after intense irradiation.

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O-16: Analysis of surface layers on Martian rocks with the APX-Yield code

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The GUYLS utility supplied within the GUPIX package predicts the X-ray yields from a single or multi-layer sample where the element concentrations are known. This code was the starting point for our new code APX-Yield which performs similar predictions for the PIXE and XRF yields from the Mars alpha-particle X-ray spectrometers. APX-Yield was intended for use with Martian rocks where camera images suggest the presence of a surface coating. The example given here is the rock Mazatzal studied in the early work of the Spirit rover. In APXS analysis of such a coated rock, the lightest element (Na-Si) concentrations are significantly tilted towards those of the coating due to the small PIXE interrogation depth, while those of the heavier elements tend

to represent both substrate and coating. Several nearby rocks were abraded and analyzed to obtain an average substrate composition. This composition was then used in the two-layer mode of APX-Yield, together with a preliminary estimate of the coating thickness and composition, and these coating quantities were varied until a match was obtained between the predicted X-ray yields and the fitted peak areas in the spectrum of the “brushed” Mazatzal rock. The results enabled us to make quantitative estimates of the coating’s enrichment in elements mobilized by aqueous fluids: P, S, Cl, K, Ni, Cu, Zn. This in turn enabled efforts to interpret the geochemical history of the coating. The method now appears ready for application to many other instances of surface phenomena on rocks analysed by the Spirit, Opportunity and Curiosity rovers.

O-17: Simulation and fit of Ta L and M shells PIXE spectra obtained at the C2TN HRHE-PIXE facility using proton and oxygen ion beams

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X-rays having energies below 5 keV are more prone to important significant chemical effects than higher energy ones. Recently the DT2 code was developed bearing characteristics specifically designed to handle a large amount of uncertainty upon fundamental parameters. These characteristics provide DT2 with great versatility and allow the user to use it as a full computer aid spectra data handler rather than a simple spectra fitting code. In this work, the specific capacities of the DT2 code will be used to simulate the M-shell spectra obtained during the irradiation of a Ta₂O₅ thin film deposited upon a polished graphite substrate produced in the frame of the Heavy Ions PIXE workpackage of the IAEA Coordinated Research Project F11019 on uses of MeV focused ion beams. Proton and oxygen beams, both produced by a duoplasmatron ion source, were used as exciting radiation. Spectra were collected using both the CdTe and the X-ray Microcalorimeter Spectrometer detectors of the High Resolution High Energy PIXE facility of C2TN. Problems, solutions and results associated and extracted from both the simulation processes and the fitting of the spectra will be presented and discussed.

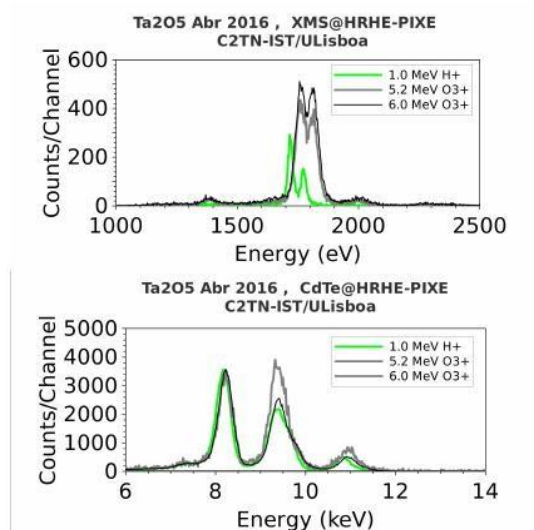


Fig. 1. L-shell normalised to H⁺ beam irradiation Lalpha group peak height (bottom) and raw M-shell (top) spectra from Ta₂O₅ CRP F11019 standard sample irradiated by a H⁺ beam at 1.0 MeV and O³⁺ beams at 5.2 and 6.0 MeV. Spectra were collected simultaneously using the CdTe for L-lines (bottom) and the POLARIS XMS for M-lines (top). The differences in the ratios of the Lalpha and Malpha groups to their corresponding beta groups are impressive and unexpected.

O-18: Standardless method for PIXE analysis

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This work introduces a method to fit the experimental PIXE spectrum by means of an analytical function whose fitting parameters can be chosen by the user. The whole set of optimizable magnitudes involves instrumental parameters, scale factors, atomic parameters and mass concentrations. The method was implemented in a software called "Parameter Assessment Method for Pixe Analysis" (PAMPA), which is available on internet [1]. The spectral processing carried out by PAMPA is based on a parameter optimization method, which consists in minimizing the function χ^2 . This function represents the squared differences between an experimental X-ray spectrum and an analytical function proposed to describe it, which depends on atomic and experimental parameters taking into account the continuous background, the characteristic peaks from the elements present in the sample, and the spurious peaks [2], such as sum peaks, escape peaks and the Si internal fluorescence peak (see, e.g., Fig. 1a). PAMPA allows for different tasks depending on which parameters are set as known and which ones are released for optimization. For instance, if the sample is perfectly known and the atomic parameters involved are known with sufficient precision, instrumental parameters, such as those associated with detection efficiency or peak asymmetry can be assessed. On the other hand, once the detection system has been properly characterized, using monoelemental or well-known samples, different atomic parameters can be obtained, such as characteristic energies or relative transition probabilities. Finally, if everything else is known with sufficient accuracy, the mass concentrations can be set as the refinable parameters of the fitting process. In the latter case, the software becomes a quantification tool or, at least, allows for semiquantitative standardless assessments. In order to test the performance of the present software when used as a quantification routine, mass concentrations obtained by PAMPA are compared to the ones obtained with GUPIX [3], one of the quantification software packages most versatile and used nowadays. The comparison was performed for 9 thick samples comprising 70 elements: Muscovite, Feldspar, Kaolinite, MgO, NbO₂, BCR, GSP, Fe₂O₃ 99%-Al₂O₃ 1% and CoO 5%-Cr₂O₃ 95%. The relative differences of concentrations obtained with both software packages with respect to the nominal values, $\Delta C/C$, are shown in the histograms of Fig. 1b. Both histograms show similar shapes. From the 70 elements considered, in 36 cases PAMPA give results closer to the nominal concentrations than GUPIX; in addition, 50% of the cases correspond to $|\Delta C/C|$ values lower than 13.1% for PAMPA and 13.3% for GUPIX. In conclusion: for quantification purposes both programs exhibit a similar performance.

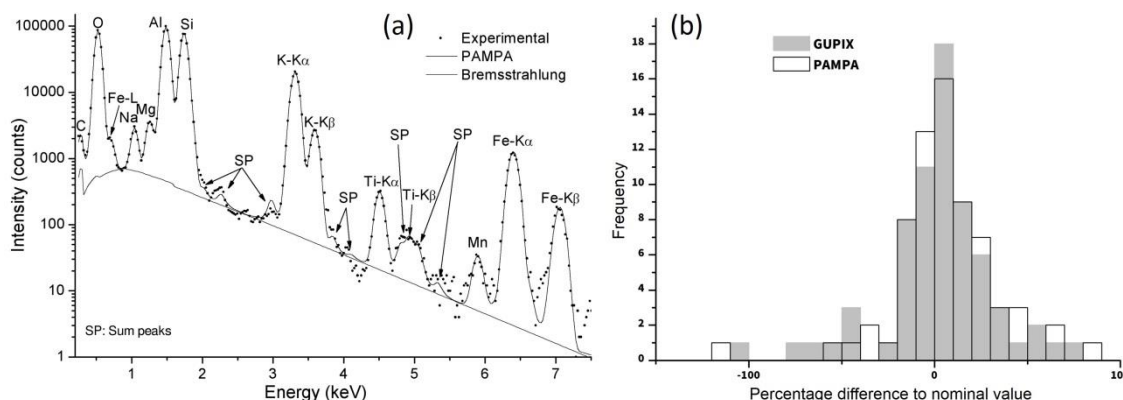


Fig. 1. (a) Experimental and fitted spectrum for a muscovite sample; (b) Histograms of $\Delta C/C$ obtained by means of PAMPA (black hollow bars) and GUPIX (gray full bars). Two cases below -150% were excluded for each software.

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[2] R.D. Bonetto et al., X-Ray Spectrom. 30 (2001) 313-319.

[3] J.A. Maxwell et al., Nucl. Instrum. Meth. B 43 (1989) 218-230.

O-19: Prehistoric glass beads from Slovenia

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In prehistoric sites of Slovenia, numerous glass beads were found that can be dated from the Late Bronze Age till the end of Iron Age. A set of beads was selected for the analysis that uniformly spans the 1st millennium BC. Analysis was performed by a combined PIXE-PIGE method using the in-air beam of the Tandetron accelerator of the Jožef Stefan Institute in Ljubljana. Several methodological improvements were introduced, such as a handy program for fitting the gamma spectra that employs automatic energy and detector resolution calibration according to the strong sodium lines in the spectra. The beads were found to be made of the glass using natron for the flux. This qualifies them as imported items, which is expectable for the types that are common in different archaeological sites, but natron glass was also found on the beads that were parts of the brooches.

O-20: PIXE and PGAA – complementary methods for studies on ancient glass artefacts (from Byzantine, late medieval to modern Murano glass)

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Typical glass composition is practically unchanged from antiquity to our days: formers - mainly SiO_2 , fluxes - soda (Na_2CO_3) or potash (K_2CO_3) and stabilizers – CaCO_3 . The colours are obtained by adding small amounts of minerals mainly oxides. Opacifiers, pigments, colouring and de-colouring agents can be used as “tracers” for the technology of production and area location of ancient glass artefacts. We determined the composition of paste, colorants and opacifiers from coloured painted Byzantine bracelets (discovered during the archaeological excavations from various 10th-12th Centuries AD Byzantine archaeological sites situated on Danube border as Isaccea - Noviodunum, a commercial town situated before Danube Delta), late medieval (17th-18th Centuries discovered during architectural restoration of two Churches in Bucharest, samples partially local produced and partially imported from Central Europe) and modern Murano glass pieces using external milli-beam Particle Induced X-ray Emission (PIXE) spectroscopy and Prompt Gamma Activation Analysis (PGAA). As fluxes, PGAA revealed all the samples are soda-lime glass, except four samples which have potash flux – two medieval vessel white shards and two dark Byzantine fragments of bracelets. PGAA also indicated the presence of low quantities of Cd, high levels of As and Pb (possibly lead arsenate) and ZnO in Murano glass, and of CoO traces (maximum 0.1%) in all blue-coloured samples, from Byzantine to Murano (the oldest preserved recipe). PIXE confirmed the use of small quantities of CoO for blue colour, indicated Mn combined with Fe for dark glass, Cu for green, Pb for yellow and, only in modern Murano glass, the presence of Se together with Zn to obtain a reddish colour and Se together with Mn in two medieval transparent samples. Our study demonstrated an impressive continuity in glass recipes from 10th to 18th Century – fluxes, stabilizers, colourants and opacifiers as Fe, Mn, Co, Pb, Co, Se, As coupled to modern innovation as ZnO for Murano glass. To identify the minerals used as colourants more non-invasive methods as Optical Microscopy, SEM investigations coupled with energy dispersive X-ray spectrometry (EDS), LA-ICP-MS or Raman Spectrometry could be used. Our investigation proved that PIXE and PGAA as complementary methods can produce an efficient identification of glass elemental composition.

O-21: Comparative analysis of textile metal threads from liturgical vestments and folk costumes in Croatia

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Textile is essential for everyday life in all societies. It is used in clothes for protection and warmth but also to indicate class and position, show wealth and social status. Threads from precious metals have also been used in combination with fibres for decoration in order to create luxury fabrics for secular and religious elites. We performed elemental analysis of 19th and 20th century metal threads from various textile articles of liturgical vestments and festive folk costumes collected in the museums of northern, southern and central Croatian regions. In order to determine elemental concentrations in threads we performed comparative X-Ray Spectroscopy measurements using: (i) Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX) at the Faculty of Textile Technology, (ii) X-Ray Fluorescence Spectroscopy (XRF) at the Croatian Conservation Institute and (iii) Particle Induced X-Ray Spectroscopy (PIXE) at the Ruđer Bošković Institute Tandem Accelerator Facility using ion micro beam. SEM-EDX investigations of cross-sections along with the surfaces were also performed. In this work we report and discuss the results obtained by the three methods for major (gold, silver, copper) and minor elements on different threads like stripes, wires and srma (metal thread wrapped around textile yarn).

O-22: PIXE contribution for a database of Phoenician Pottery

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Pottery is the most abundant archaeological artifact and therefore it is the most studied by archaeologists nowadays, since it is characteristic of ancient civilizations and, thus, several related information could be revealed. In this project, it is proposed to study the Phoenician ancient cult place of Kharayeb, in the rural hinterland of Tyre, southern of Lebanon, dated to Persian and Hellenistic periods. Rural contexts, in fact, are particularly helpful in evaluating the complexity and variability of the so called “Hellenism” and of “Greek cultural influences” in the Phoenician world. The studied objects are mainly figurines and some artisanal objects with possible analysis clay sources in the area. PIXE technique will contribute to establish a first database of chemical composition of Phoenician pottery, which will broaden the existing database currently related to archeological pottery from coastal sites in Lebanon. Furthermore, the PIXE information about the composition of the clay will help to understand if the figurines were locally produced or imported from the coast and how was the process of production connected to the sanctuary. Finally, this work will be probably used for any future archeological study of Phoenician pottery in the Phoenician colonies in the Mediterranean basin.

O-23: Analysis of Greek small coinage from the classic period

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A set of Greek oboloi from the 6th-4th c. BC was analyzed by the in-air beam of the Tandatron accelerator in Ljubljana. The coins were characterized according to the admixture of copper, lead, gold and bismuth. It was found out that the coins from Athens differ from particular coins minted in other Greek cities of mainland Greece, Asia Minor and Southern Italy. This may be explained by the Athenian coinage using the silver ore from Laurion, which according to the literature contained very small admixtures of gold, but more lead. Two coins departed from this scheme: a coin from Lesbos was minted from the same type of silver as the Athenian coinage, and one Athenian coin dated shortly after 400 AD showed a rather high percentage of copper, about 9%. This may indicate a degradation process of Athenian coinage, which was urged by the economic circumstances following the defeat of Athenian fleet in the battle of Aegospotami 405 BC.

O-24: PIXE-RBS beamline in the University of Jordan Van de Graaff Accelerator (JUVAC): Development and Applications

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The University of Jordan Van de Graaff Accelerator (JUVAC) was devoted many years ago to promote scientific research through the installation of several beamlines based on nuclear techniques. It constitutes the first and unique, till now, ion beam analysis (IBA) facility in Jordan. Among these techniques, the JUVAC has installed, with the financial and technical aid of the International Atomic Energy Agency (IAEA), a combination of particle-induced X-ray emission (PIXE) and Rutherford backscattering spectrometry (RBS). By combining PIXE and RBS, the stoichiometry of different samples is determined. This talk is mainly a description of the combined PIXE-RBS beamline and its development and applications where various research projects were performed. In this regards, some examples will be discussed.

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O-25: Standardisation of the ion beam facility at Chandigarh cyclotron for simultaneous PIXE and PESA analysis

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The proton beam facility at variable energy cyclotron (VEC) Panjab University, Chandigarh, India [1] is being used for Particle induced X-ray emission (PIXE) analysis of different environmental, biological and industrial samples. The PIXE method, however, does not provide any information of low Z elements like hydrogen, nitrogen, oxygen and fluorine. Therefore, several nuclear analytic techniques are generally used in conjunction with PIXE, such as Proton Induced Gamma-ray Emission (PIGE) and Proton Elastic Scattering Analysis (PESA) to obtain the concentration of low Z elements. In addition to the X-ray detector, the scattering chamber at VEC facility can accommodate both gamma and charge particle detectors for simultaneous analysis of samples by PIXE, PIGE and PESA using the millimeter size proton beam. The

increased need for rapid and multi-elemental analysis of biological and environmental samples has made us upgrade and standardize the PIXE facility to facilitate simultaneous PIXE and PIGE experiments. For this purpose, 2.7 MeV proton beam from the VEC, which offers maximum stability and current, was used for the standardization of the setup. We used a set of thin films of known thickness covering a wide range of elements (both low Z and high Z) across the periodic table and standard reference materials (SRM) from NIST and IAEA. The X-rays were detected by Canberra HPGe detector positioned at 225° and the backscattered protons were detected by Canberra SSB detector positioned at an angle of 165° with respect to the beam. Both the spectra were recorded using a dual channel data acquisition system. The present detection system was optimized to provide a better detection limit over a wide range of elements. Collected PIXE data has been analyzed using GUPIX [2] software. For standardization, the instrumental constant H (solid angle and charge correction factor) was determined by completely relying on GUPIX database. Backscattered proton data was analyzed with SIMNRA [3] software. The detection limit of PIXE for thin targets varies from 4 ng/cm² (~ Z = 24) to 20 ng/cm² (~ Z = 40); and for thick targets, it varies from 1-10 ppm depending upon the total charge and the absorber thickness. For PESA analysis the limit of detection was ~ 30 ng/cm² for low Z elements like carbon. Aluminum X-ray absorbers of different thickness mainly 13 mg/cm² and 30 mg/cm² are used during the experiments. PIXE has already become a basic technique and PESA compliments it by enabling analysis of light elements, thus providing broader applications. The concurrent use of PESA with PIXE system has allowed us to analyze low Z elements in the biological and environmental samples. Because they operate simultaneously, the additional cost is quite less and the low Z elemental analysis can be used as a quality assurance check of individual calculations of fine mass.

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[2] John L. Campbell et al., Nucl. Instrum. Meth. B 170 (2000) 193-204.

[3] M. Mayer, Nucl. Instrum. Meth. B 332 (2014) 176-180.

O-26: In-air PIXE and MeV SIMS as analytical techniques for detecting xenobiotics and pharmaceutical compounds in human scalp hair

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There is a growing interest in screening human biopsy tissue or biomarkers to identify and quantify xenobiotic and medicinal or illegal drug residues in the body [1]. Of these biomarkers, human scalp hair has gained increased recognition for assessing exposure to xenobiotics in the workplace or environment and for the retrospective testing of drug use [2]. Hair testing is now routinely used in clinical medicine for therapeutic monitoring, to determine medicinal or chronic doping use, to confirm gestational drug use, or to assess exposure to toxins and pollutants such as heavy metals [1]. Hair testing has also become popular in forensic science to

assess drug use history, criminal liability of drug users, and intentional or unintentional poisoning in post-mortem toxicology [1]. A significant factor affecting the outcome of hair testing is the choice of analytical analysis technique [3]. Here, two ion beam methods for the detection of xenobiotics and pharmaceutical compounds in virgin human hair are presented. Firstly, we optimized and explored the analytical capability of the in-air PIXE setup at the Jožef Stefan Institute for elemental profiling in intact human hair fibres. Secondly, we explored the analytical capabilities of the linear time-of-flight mass spectrometer for MeV secondary ion mass spectrometry (MeV-SIMS) added to the nuclear microprobe at the Jožef Stefan Institute as a complementary technique for the chemical mapping of virgin human hair fibres. In-air PIXE proved to be a sensitive technique for the detection of toxic metals in an intact volume of hair. The results further showed that MeV-SIMS allowed the simultaneous detection and localized distribution of pharmaceutical as well as illegal drug residues in longitudinally sectioned and untreated human hair fibres. The significance of these findings and future applications of these versatile ion beam analysis techniques will be discussed.

[1] Joana Barbosa et al., *Bioanalysis*. 5 (2013) 895-914.

[2] Elena Viola Buononato et al., *Environ. Monit. Assess.* 188 (2016) 337.

[3] Tatiana Baciú et al., *Anal. Chim. Acta* 856 (2015) 1-26.

O-27: Particle Induced X-Ray Emission and Raman Spectroscopy in the study of patrimony art forgery

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Nowadays the forgery of valuable paintings has become a serious industry damaging the image and reputation of the museums displaying the artworks, but also the patrimony of the state owning that specific painting. To overcome this new threat, experts started using non-invasive physical methods to analyze the artworks while preserving them. The authenticity of paintings can be established through many methods, of which the physical analysis of pigments used is very popular considering its non-invasive profile [1, 2]. Over the years, technological advances allowed the development of pigments, resulting in modern types characterized by completely different chemical compositions. Thus the difference between classical and new or authentic and forged can be established through the chemical elements identified in the pigment's composition [3].

In the present paper we coupled Particle Induced X-Ray Emission (PIXE) with Raman Spectroscopy to analyze six of the commonly used pigments in paintings: white, black, blue, green, orange and brown. Modern and classic pigments were deposited onto white linen to mimic most of the paintings and analyzed using PIXE and Raman Spectroscopy. The goal was to identify the principal components in each pigment in order to obtain a view over the painting's authenticity without damaging it. The use of the two chosen analysis techniques was justified also by their non-invasive profile, a clear requirement in the field of cultural heritage.

The results obtained through the PIXE analysis were comparable with the ones obtained through Raman Spectroscopy analysis. Both methods successfully identified the components of each modern and classical pigment, enabling the differentiation between titanium white and flake white or marble dust, Prussian blue and azurite or ultramarine. This result encourages not only to identify as a new technological center aimed at backing up art experts in their offensive against forgery, but also as a new opportunity to develop the applications of these two physical methods in our institute.

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[2] Ji-Yeon Nam et al., Vib. Spectrosc. 85 (2016) 181-184.

[3] W. Berrie, B. Hepburn, Artists' Pigments, A Handbook of Their History and Characteristics, Vol. IV, Oxford University Press, New York, 2008

O-28: Elemental signature of Brazilian foodstuff by PIXE

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The information about food provenance is an essential aspect related to safety and authenticity. The knowledge about food provenance means quality control and higher market value, which hampers illegal production and commercialization practices. In this context, isotopes, macro and trace elements or compounds may be used as geochemical markers to indicate origin, provenance and authenticity of foodstuff. The aim of this work is to apply Particle Induced X-ray Emission (PIXE) to determine the elemental composition of food products and beverages from Brazil, thus looking for an elemental signature through statistical analysis. Initially, coffee and *Ilex paraguariensis* samples have been analyzed using the PIXE beamline at the Ion Implantation Laboratory (Porto Alegre, Brazil). These products were selected for study because of the following reasons: 1) Brazil is an important producer and exporter of coffee. Some restricted regions or coffee farms like Alta Mogiana (Sao Paulo, Brazil) are recognized by the geographical indication (IP) and are engaged in obtaining the denomination of origin (DO). 2) Brazil presents several regional products, like *Ilex paraguariensis* tea, used to make a hot beverage called *chimarrao*, which is typical and very appreciated in the south states of Brazil and South America countries. However, for both cases, there is few scientific data for provenance and authenticity determination. For this study, toasted and green beans of Arabica coffee are collected directly from Alta Mogiana's farms during harvest time, while toasted bean coffee from other Brazilian regions are acquired in the local market to comparison. *Ilex paraguariensis* tea packs from different regions of Brazil, Argentina and Uruguay are also acquired in the market. Five and fifteen samples of coffee and tea from each farm or production region, respectively, have been analyzed by PIXE. Qualitative results obtained for *Ilex paraguariensis* tea showed differences for Ti, Br, Mn, Sr and Zn, for three studied regions. The next step is to quantify all PIXE spectra and perform the multivariate statistical analysis in order to verify if those elements may be used as geochemical markers to provenance determination.

O-29: X-ray production cross-sections in metallic films due to C, O and Si ion beams at MeV SIMS energies

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The development of theoretical models to describe various ion beam-matter interaction phenomena for ion beam analysis (IBA) applications is strongly dependent on the availability of experimental data to test these models. IBA using heavy ion beams at MeV energies is now commonplace in many small accelerator labs. Secondary Ion Mass Spectrometry using heavy ion beams in the MeV energy regime (MeV-SIMS) for instance promises to be an effective technique in molecular imaging studies. Heavy Ion Elastic Recoil Detection (HI-ERD) analysis has been found to be very effective in depth profiling of thin film structures without using any standards. In the spirit of ‘total IBA’, heavy ion PIXE can be co-implemented with these and other techniques to get fuller descriptions of samples being analysed. An inhibiting factor in the implementation of heavy ion PIXE is that heavy ion X-ray production cross sections, among other important variables, are not well known for many materials. This contribution reports on measurements carried out to determine X-ray production cross sections in Ti, Zn, Ru and Ta induced by 0.25-1.0 MeV/u ^{12}C , ^{16}O and ^{28}Si ions. Experimental data is compared to predictions by the PWBA and ECPSSR theories calculated using the ISICSoo code.

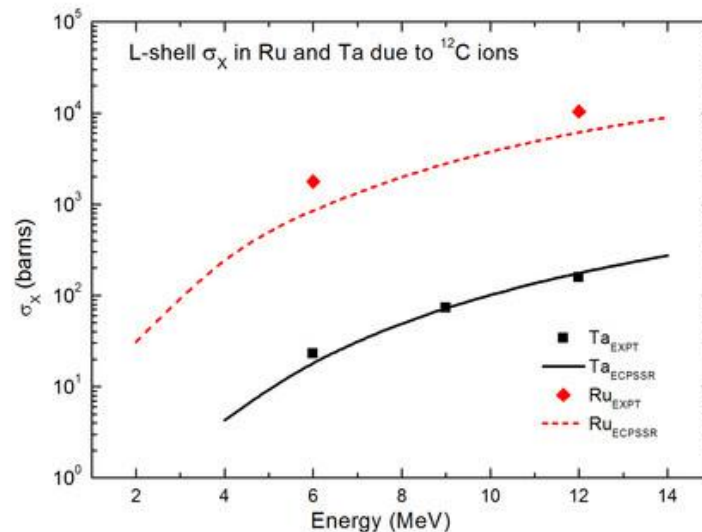


Fig. 1. Experimental and theoretical L-shell X-ray cross sections in Ru and Ta due to carbon ions

Acknowledgements: Authors would like to acknowledge the International Atomic Energy Agency (IAEA CRP F11019), Tshwane University of Technology and the National Research Foundation (iThemba LABS) for financial and material support.

O-30: Ion microbeam NRA and PIXE analyses of individual dust particles from JET tokamak

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The mission of the JET tokamak is to demonstrate efficient operation with metal walls: tungsten in the divertor and beryllium in the main chamber wall. This material configuration is planned in the International Experimental Reactor (ITER). Plasma-surface processes cause wall erosion followed by migration and re-deposition of eroded species. This results in the formation of deposited layers and may also lead to the generation of dust. Ion beam analysis together with microscopy methods belong to the most efficient tools in studies of first wall materials in controlled fusion devices. To our knowledge this is the first report on combined μ -IBA (PIXE, NRA, RBS) studies of dust particles from a tokamak. The study was concentrated on thick co-deposits loosely bound to the surface of tungsten-coated tiles from the JET inner divertor. The area was identified as the region with the thickest deposits in JET with the ITER-Like-Wall. The sample was retrieved using an adhesive carbon pad. Ion microbeam analyses of individual dust particles from JET tokamak, attached to sticky carbon pad, were performed at ion micro-beam line of the RBI Tandem Accelerator Facility. 4 MeV ^3He ion beam was used for simultaneous collection of PIXE and NRA spectra. Semi-quantitative/qualitative analysis of collected PIXE and NRA spectra were performed using GUPIX and SIMNRA software packages. Semi-quantitative analysis has revealed the presence of Be with some D present as thick “layer” (not only on surface), as identified by NRA. They occur in coincidence with high Ni, W concentrations and some presence of Cr, Fe, Cu, as detected by PIXE. In addition, some small particles containing Al, Si, S, P, K, Cl, K, Fe were identified by PIXE. These are most probably contaminants in the tape itself, not particles from JET (in agreement with NRA spectra which show only the presence of carbon). In general, microscopy combined with energy dispersive X-ray analyses has revealed: (a) particles of mixed columnar and stratified structure detached from the deposition zone: thick and cracked Be-based layers; (b) there are regions rich in tungsten and nickel. There are two strands of concluding remarks. From the point of view of material migration studies the results have shown not uniform distribution of species in loosely bound deposits/dust particulates. To perform the study technical developments in the IBA system were introduced. Both aspects will be presented and discussed.

O-31: The Cyclotron Arronax for fundamental and applied research with light ion beams <70 MeV

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The Arronax cyclotron, operational since 2011, deliver beams with wide characteristics in terms of energy, particle and intensity. Arronax fulfills several missions for the production of radionuclides and for the research. An experimental room is dedicated to experiments in physics, radiochemistry and radiolysis. It is open for both internal and external teams.

Wide expertise exists at Arronax, which has enabled experiments in several fields. For example, the measurement of the cross-sections of X-ray and gamma-ray production to complete the data bases, the high-energy PIXE / PIGE method for thick sample analysis, the dosimetry to conduct radiobiology experiments in good conditions.

During my presentation, I will show the different methodologies that have been putted in place, and the accompanied instrumental developments to meet the needs of several types of experiments.

O-32: Source apportionment revisited for long-term measurements of fine aerosol trace elements at two locations in southern Norway

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Five-year-long aerosol trace element data sets for the fine (PM₂) size fraction from the sites of Birkenes and Skreådalen in southern Norway were analysed by US EPA positive matrix factorization PMF₅ in order to assess the sources and their contribution to the PM₂ aerosol. The samples had been collected on Nuclepore polycarbonate filters with Gent stacked filter unit samplers. The data sets contained the concentrations of the PM mass, black carbon (BC) and 21 elements in over 700 samples for each of the two sites. The PM mass was obtained from weighing with a microbalance and BC was determined with a light reflectance technique. The data for the elements were obtained by a combination of PIXE and instrumental neutron activation analysis (INAA). Eight source factors were retained for each site, i.e., (i) secondary sulfate, which accounted for around 40% of the average measured PM₂ mass, (ii) wood burning, with BC, K and Zn, which accounted for about 17%, (iii) an iodine factor (with also Br and Se), which is probably related to a marine biogenic source and was responsible for about 7%, (iv) aged sea salt with Na, Mg, Cl and Ca, but heavily depleted in Cl; (v) a crustal factor containing Al, Si, Ca, Ti, and Fe; (vi) a heavy oil burning factor with V and Ni in a ratio of 3-4; (vii) a general pollution factor (with Cu, Zn, As, Se, Sb, and Pb), and (viii) an almost pure manganese factor, which is attributed to Mn and FeMn industries in southern Norway. Substantial seasonal variation was observed for some factors, e.g., for the wood burning factor, which was most

prominent in winter, followed by fall, spring, and summer, and for the secondary sulfate and iodine factors, which were most pronounced in spring and summer. The results were compared with those of an earlier PMF analysis, in which use was made of PMF2; strangely enough, no wood burning factor was identified in that earlier analysis, but rough agreement was observed for the other factors and their contributions. It is concluded that this new PMF5 analysis provided improved and more consistent results than the earlier PMF2 analysis. A comparison was also made with the results of aerosol chemical mass closure calculations. In the latter, around 40% of the average measured PM2 mass was left unaccounted; this unaccounted mass contains likely mainly organic matter and nitrate, which were not included in the chemical analysis. In the PMF5 analysis about 10% of the average measured PM2 mass was left unexplained, indicating that most of the non-measured components were contained in some of the factors, e.g., in the wood burning and iodine factors.

O-33: Composition and sources of particulate matter pollution in five Hungarian cities in 2015

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In this work concentration, composition and sources of atmospheric aerosol pollution was studied in five Hungarian cities. Daily PM2.5 and PM10 samples were collected in a synchronized way two times a week in three urban background (Debrecen, Budapest, Pécs) and in two traffic sites (Miskolc, Nyíregyháza) from January to August, 2015. At each site 62-65 PM2.5 and PM coarse samples were gathered using Gent-type stacked filter units. In the heating season another 12 PM10 samples per site were collected with high volume samplers for EC/OC and C-14 analysis. The PM mass was weighted on a microbalance, elemental composition for $Z > 11$ was determined by PIXE, the black carbon (BC) content was measured with a light reflectance technique, the EC/OC content was quantified by using a Sunset Laboratory EC/OC analyser and the radiocarbon characterization of the samples was done at the EnviroMicadas accelerator mass spectrometer of Atomki. Source apportionment was carried out using the EPA PMF5.0 receptor model. Seasonal variation, similarities and differences of concentration, composition, source profiles and the time pattern of source contributions were studied for the different geographical locations and site types. Several local, regional and long range transport emission episodes were also identified. E.g. in April, 2015 three different dust episodes could be distinguished by the different trace element composition and concentration ratios: one was a local dust storm which appeared only in East Hungary, the second one was a Saharan dust intrusion detected in all locations and the third was desert dust from middle Asia observed also in the whole country.

Acknowledgements: This work was prepared within the frame of the János Bolyai Research Fellowship of the Hungarian Academy of Sciences. The research was supported by the European Union and the State of Hungary, co-financed by the European Regional Development Fund in the project of GINOP-2.3.2.-15-2016-00009 'ICER'.

O-34: Characterization of fine and coarse atmospheric particulate matter from Beirut suburb using PIXE technique

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In this work, it is investigated the elemental composition of fine and coarse air particulate matter $PM_{2.5}$ and $PM_{10-2.5}$ collected during 2014-2016 in a suburb area of Beirut, using the ISAP® 1050e sampler having a combined inlet. The collection of fine particles was carried out on thin Teflon filters while the coarse ones were collected by impaction using a custom made polypropylene ring foil. The characterization of the elemental content of the two fraction mode, fine and coarse particles, were analyzed using proton induced X-ray emission technique PIXE. It will be more focused on the elemental composition of the fine fraction $PM_{2.5}$. Depending on the volume of pumping air in 24h, PIXE is used to determine the concentration of Na, Mg, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn and Pb as ng/m^3 of air. For each sample, there was a clear dependence of the elemental content of PM, as well as their total mass, on weather conditions and on the type of pollution sources, such as car traffic, that are affecting air quality.

O-35: Atmospheric impact of ship emission on fine particulate matter in the Port of Rijeka

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In the recent years the Port of Rijeka, Croatia, has recorded an increase of cargo ship traffic and a number of containers. The total cargo traffic has increased app. 20 % and the number of containers app. 15 % in 2015 compared to 2013. Impacts of ship emissions to air quality have negative effects on costal urban areas on both, human health and local climate. To investigate these impacts, daily $PM_{2.5}$ samples were collected from August 2013 to October 2015. The samples were analyzed by means of Particle Induced X-Ray Emission (PIXE) method to determine concentrations of 22 elements from Na to Pb, and also by Laser Integrated Plate Method (LIPM) to determine black carbon concentrations. The results were statistically evaluated by Positive Matrix Factorization method. The average daily contributions of ship emission to the total $PM_{2.5}$ fraction was 10 ± 8 % fluctuating from 0 to 43 %. As expected, contributions depend strongly on local meteorological conditions and on the intensity of the ship traffic.

O-36: Elemental distribution in feathers of the Common Swift (*Apus apus*), a PIXE, hard X-ray and soft X-ray fluorescence study

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Trace elements in bird feathers are scientifically relevant for several reasons: they are a monitor of environmental quality in the place where the bird feeds at the time of feather growth; they may provide information on the biology of feather growth; and they may have applications in determining the location of migratory birds at the time of feather growth. We have mapped the microscopic distribution of trace and other elements in bird feathers using three methods: PIXE (at the Ruđer Bošković Institute, Zagreb) [1]; hard X-rays (Australian Synchrotron, Melbourne) [2] and soft X-rays (Elettra Sincrotrone Trieste) [3]. Light elements are best mapped using soft X-rays and elements such as Na, Mg, Si and Al are observed. Hard X-ray fluorescence is best for heavier elements, such as Ca and transition metals such as Fe, Cu and Zn. PIXE constitutes an intermediate case with some light as well as heavy element sensitivity. Issues related to overlapping fluorescence lines, self-absorption and related matters will be discussed.

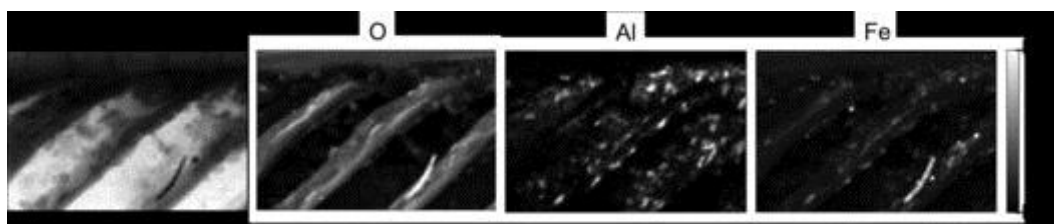


Fig. 1. Absorption (Abs) images of swift feathers, with corresponding XRF maps of O, Al, and Fe. Acquired at Twinmic, 1.7 keV photon energy; spot size, 1 micron diameter. The oxygen map correlates with the absorption image, indicating most absorption is due to the keratin (protein) of the feathers. It also correlates with the strongest feature in the Fe map (linear structure, bottom right), suggesting this is iron oxide. The aluminium and iron appear to be present as particles.

[1] M. Jakšić et al., Nucl. Instrum. Meth. B 260 (2007) 114-118.

[2] David Paterson et al., AIP Conf. Proc. 1365 (2011) 219-222.

[3] Alessandra Gianoncelli et al., J. Synchrotron Rad. 23 (2016) 1526-1537.

ROUND TABLES

ROUND TABLE 1: Detectors

Discussion moderator: **Geoffrey W. Grime** (University of Surrey)

Outline:

Two factors contributed to the development of PIXE in the 1970s. One was the move of experimental nuclear structure physics to higher energies which freed up beamtime on small accelerators and the other was the increasing availability of the lithium drifted silicon detector. Since this was also used in electron probe microanalysis it was intensely developed and high quality detectors became readily available in what was effectively a mass market. Si-Lis became virtually the only detector used for PIXE analysis and understanding their properties and imperfections was essential for high quality work.

In recent years this “mass market” source of Si-Lis has dried up. The Si-Li is virtually extinct in EPMA and XRF laboratories, having been displaced by the much more convenient (and cheaper) SDD.

The aim of this round table is to explore the questions raised by this change, namely whether the advantages of Si-Lis for PIXE really are so great that we should still try to seek them out at all costs or can we live with the disadvantages (real or mythical) of the cheaper SDDs. What are the alternatives?

The round table will be attended by representatives of RaySpec who will present the state of the art in SDD development, but the main interest is to share and pool our experience and fears of life in a post-Si-Li world.

ROUND TABLE 2: Software

Discussion moderator: **Ivo Orlić** (University of Rijeka)

Outline for GUPIX 3 presentation:

John L. Campbell

Guelph-Waterloo Physics, University of Guelph, Guelph, Ontario, N1G4B3, Canada

With over 170 GUPIX copies (mostly GUPIXWIN) now provided in 51 countries, work is starting on GUPIX 3. Some of the proposed changes derive from more elegant approaches devised for the recent GUPIX-based code GUAPX which fits PIXE+XRF spectra recorded by alpha particle X-ray spectrometers in the laboratory and on Mars. Changes for convenience will include a new folder structure, detailed csv output files, and user choice of concentration units. Various small and not-so-small inconveniences reported by users will be repaired. One example is the inability of the PAR file to recall full detail of invisible compounds. Another is the present inability to zoom final plots of the fits in the way that the initial spectrum plot can be zoomed. New detector windows and contacts will be added. Changes aimed at improved accuracy include a non-linearity correction in the channel-energy relationship, improved KLL radiative Auger intensities, improved Ge escape ratios, and a choice of three attenuation coefficient databases. The ability to fix chosen concentrations (e.g. pre-measured by PIGE or RBS) will be provided in iterative matrix mode. For alpha-PIXE a means of including multiple ionization satellites is ready for the energy region 3-5 MeV and experimental work is in progress to improve the relevant database.

POSTER PRESENTATIONS

P-1: Electronic wave function and binding effects in M-subshell ionization of gold by light ions

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Recent progress in applications of the particle induced X-ray emission (PIXE) needs accurate knowledge and understanding of the M-X-ray production cross sections by light ions. In the present paper we report on the measurements of the X-ray production cross sections for dominating M-X-ray lines and the M-subshell ionization cross sections for gold bombarded by hydrogen ^1H and helium ^3He ions in the energy range 0.2-4.0 MeV/amu. This work is a continuation of our earlier systematic studies of M-shell ionization by light ions [1], where only the total M-X-ray production and M-shell ionization cross sections were discussed. Generally, the available M-subshell X-ray production cross sections were found [1,2] to be systematically up to a factor of three higher at low energies than the predictions of the ECUSAR theory commonly used in PIXE applications. For this reason the present data on the M-subshell ionization by light ions were interpreted in terms of the basic theoretical approaches describing M-subshells ionization by charged particles, namely the plane-wave Born approximation (PWBA) which was further developed to the ECUSAR theory, the semiclassical approximation (SCA) and the binary encounter approximation (BEA), in order to investigate a role of the electronic wave function and the electron binding effects. In particular, to calculate the M-subshell ionization cross sections different electronic wave functions, from nonrelativistic hydrogenic to relativistic Dirac-Fock types, were used within the SCA while in the BEA both the nonrelativistic and relativistic hydrogenic and the Hartree-Fock-Roothan wave functions (see Ref. [3]) were used. Similarly, the electron binding effect was taken into account within the extreme separated-atom (SA) and united-atom (UA) limits as well as the proposed molecular (MO) model describing this effect. The experimental results for the M-shell X-rays excited by light ions are interpreted in terms of the discussed theoretical approaches in order to explain the observed discrepancies, in particular, for the lowest impact energies.

[1] M. Pajek et al., Phys. Rev. A 73 (2006) 012709.

[3] D.D. Cohen et al., Nucl. Instrum. Meth. B 318 (2014) 11-14.

[3] T. Mukoyama, Nucl. Instrum. Meth. B 354 (2015) 155-158.

P-2: Carbon ions induced M shell X-ray production cross sections in Au and Pb thin targets in the energy range 2.8 - 5 MeV

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In the present work, we present new experimental $M_{\alpha\beta}, M_{\gamma}, M_{M2-O4,5}$ and total M shell X-ray production cross sections measured on thin solid targets of gold and lead using $C^{4,3+}$ ions in the

energy range 2.8 MeV to 5 MeV. The obtained experimental X ray production cross sections are compared to the predictions of the Plane wave Born Approximation [1] and the ECPSSR [2] models calculated using the ISICS code [3]. Our experimental results are compared to the experimental data existing in literature.

[1] E. Merzbacher, H.W. Lewis, Encyclopaedia of Physics, Vol. 34, ed. S. Flügge, Springer, Berlin, 1958, p. 161

[2] W. Brandt, G. Lapicki, Phys. Rev. A 23 (1981) 1717-1729.

[3] S.J. Cipolla, Comp. Phys. Comm. 182 (2011) 2439-2440.

P-3: X-Ray Production Cross Section of Ti, Cr and Ni induced by Cl ions

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X-ray production cross sections induced by ions are the most important parameter as far as PIXE (Particle-Induced X-ray Emission) experiments are concerned. Protons have by far the most comprehensive database for X-ray production cross section, which makes them the ion of choice for quantitative X-ray spectrometry employing swift ions. Despite some research was dedicated to other light ions like alpha particles, lithium, carbon and oxygen, cross sections for heavier ions are practically non-existent. In recent years, the advent of SIMS (Secondary Ion Mass Spectrometry) experiments induced by heavy swift ions has opened up the possibility of measurements of PIXE and MeV-SIMS simultaneously. However, for typical IBA (Ion Beam Analysis) energies, i.e. few MeV, no X-ray production cross sections are available for heavy ions. In this work we measured X-ray production cross sections induced by Cl ions in thin Ti, Cr and Ni films. The measurements were carried out at the Ion Implantation Laboratory of the Physics Institute (UFRGS) using a 3 MV Tandetron accelerator. Experiments were performed with 4 – 10 MeV Cl ions. The targets were evaporated on glassy carbon. A Si (Li) detector with energy resolution of about 150 eV at 5.9 keV positioned at 135° with respect to the beam direction was used to detect X-rays generated in the targets. The results are compared with PWBA calculations as well as with the ECPSSR theory. In general, PWBA calculations overestimate the experimental cross sections while ECPSSR calculations underestimate the results.

P-4: M-X-rays from interaction of slow highly charged Xe^{q+} ions (q=26-40) with beryllium foil

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In interaction of slow highly charged ions (HCI) with matter the so-called “hollow atoms” are formed in the process of fast neutralization of HCI at the surface [1,2]. In these exotic atomic states a large part of the electrons are in high Rydberg levels while the inner shells remain empty. The X-rays and Auger electrons emitted in deexcitation of hollow atoms carry

information about formation and the structure of hollow atoms as well as their relaxation processes. In this work we report on the measurements of X-rays emitted in interaction of ~ 3 keV $\times q$ Xe^{q+} ions ($q=26-40$) with metallic Be foil. The ion beams were produced in the EBIS-A facility [3] consisting of the electron beam ion trap (EBIT), ion extraction and beam transport system, analyzing dipole magnet and UHV experimental chamber equipped with a 5-axis sample manipulator. The X-rays were measured with the XFlash silicon drift detector (SDD) having a resolution of about 80 eV in 1-2 keV photon energy range studied. The X-rays measured for different charge states of Xe^{q+} ions were interpreted as the M-X-ray $n\ell-3\ell$ transitions (where $n \geq 4$) corresponding to different multivacancy configurations, including both X-ray satellites and hypersatellites. The energies of these transitions were calculated using the multiconfiguration Dirac-Fock (MCDHF) method. In order to interpret the measured spectra, the experimental broadening of spectral lines have been taken into account. The observed intensities of $n\ell-3\ell$ transitions, including their satellite structures, yielded information about population of electrons in high n -states. In this way an initial population of electrons as appears in ion neutralization process, followed by the Auger and radiative deexcitation cascade was probed experimentally. Consequently, the predictions of the classical over-the-barrier model (OBM) describing the neutralization of HCl at surfaces were tested experimentally. For Xe^{26+} ions with no M-shell vacancies expected the observed M X-rays may indicate more complex electronic structure of these ions or the internal dielectronic excitation process.

[1] H. Winter, F. Aumayr, J. Phys. B: At. Mol. Opt. Phys. 32 (1999) 39-65.

[2] J.-P. Briand et al., Phys. Rev. Lett. 65 (1990) 159-162.

[3] D. Banaś et al., Nucl. Instrum. Meth. B 354 (2015) 125-128.

P-5: Physical model for the response function of a semiconductor X-ray detectors

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A response function of a general semiconductor detector is still missing in literature. Following the necessity to describe in detail the response function of the C2TN HRHE-PIXE CdTe detector, a physical model was established to describe the response function of semiconductor detectors more complex than the Si(Li). Reasoning started on observations in spectra collected with Si(Li) detectors, in particular the fact that the low energy tails in Si(Li) detectors drastically changes at the energy of Si-K absorption edge. After this, was noticed that the CdTe detector having two component elements in the crystal shows a more complex escape peaks structure and a more complex low energy tail. Adjustable parameters of the response function were obtained from 8 keV Cu-Kalpha up to 98 keV U-Kalpha by fitting spectra obtained by proton irradiation of pure oxides pallets. In this work the physical model is presented and its used for describing the response function of a general semiconductor detector is discussed.

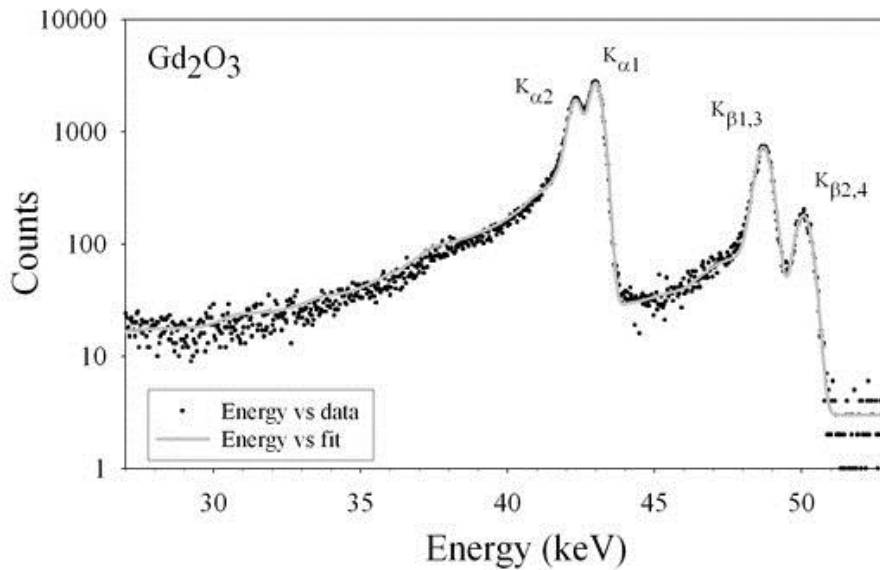


Fig. 1. Spectrum of a Gd_2O_3 pure thick sample irradiated with a H^+ beam of 4.0 MeV and collected using a CdTe detector placed at 35° relative to beam direction. The details in the peak tails can be seen. The final fit and the tail details can be seen to reproduce very well the spectrum.

P-6: Evaluation of the Si(Li) detector efficiency through the H value

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One of the key issues in experiments involving the detection of X-rays resides in the characterization of the spectrometer used to detect low energy photons. Despite different detectors have been used to date, Si(Li) detectors have an undisputable edge over the others due to their relatively good energy resolution and their constant efficiency between 4 and 15 keV, thus covering the most important energy range as far as PIXE (Particle-Induced X-ray Emission) is concerned. From the theoretical point of view, different parametrizations and Monte-Carlo calculations have been used to describe the detector efficiency. On the experimental side, the use of calibrated radioactive standards has been widely employed in the measurement of the detector efficiency. One of the most popular softwares used to analyze X-ray spectra is GUPIXWIN, which is based on the GUPIX engine developed at the University of Guelph during the eighties [1]. Despite GUPIX does not require the use of standards, the so-called H value embodied in the GUPIX methodology is based on standards measured under the same conditions as the PIXE experiments. In this work we show a simple way to extract the detector's efficiency from the measurement of the H value obtained as a function of the X-ray energy. The experimental results are in good agreement with Monte-Carlo simulations [2]. In particular, the role of the solid angle is discussed.

[1] John Maxwell et al., Nucl. Instrum. Meth. B 43 (1989) 218-230.

[2] Stephanie Taylor et al., X-Ray Spectrom. 39 (2010) 191-201.

P-7: Standardization of an SDD using PIXE

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For current work with both PIXE and Alpha Particle X-ray Spectrometer analysis of geological reference materials, an accurate and detailed calibration of silicon drift detectors (SDDs) is necessary. Heirwegh et al. [1] had previously determined the non-linearity of our SDD detector and determined tail parameters; the latter work has been extended. H was first conceived as an instrumental “constant”, incorporating the solid angle of detection together with any calibration constant inherent in the method of charge measurement. Deficiencies in the GUPIX database and the detector description may cause H to depart from constancy. For our system H was determined using 20 pure element and compound standards providing X-ray energies ranging from 0.5keV to 20keV. The compatibility of H-value results from K and L X-rays, as well as the choice between the NIST XCOM attenuation coefficients currently used in GUPIX and the newer NIST FFAST database, were examined. Heirwegh et al. [2] found that FFAST values provided better results for photons with energies below the light element K edges: Na in Na; Na and Mg in Mg; Na, Mg and Al in Al; Na, Mg, Al and Si in Si; and Na, Mg, Al, Si in O. XCOM was maintained by Heirwegh et al [2] for all other situations. The plot of H-value vs X-ray energy shows a steep fall at low energies. This is too great to be attributed to exclusively the difference between the real and nominal window thickness and is likely an electronic effect. Using XCOM, L X-rays and the P K X-ray from an apatite standard provided H values that sat too high on the curve. Replacing the attenuation coefficients of these elements with those of the FFAST database achieved consistency with the rest of the H curve, seen in Figure 1. This work lays the foundation for accurate studies of mineral phase effects in PIXE analysis of geologic materials.

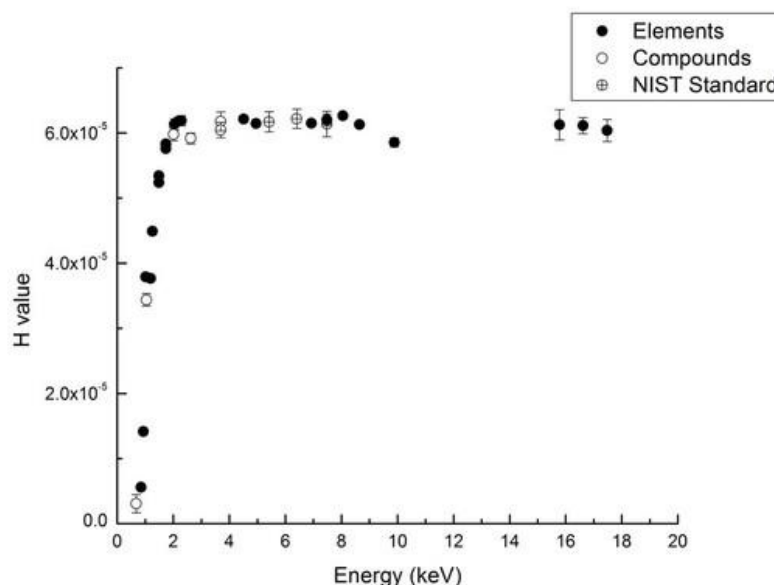


Fig. 1. H curve with an extension of the mixed FFAST and XCOM database.

[1] C.M. Heirwegh, I. Pradler, J.L. Campbell, J. Phys. B: At. Mol. Opt. Phys. 46 (2013) 1-9.

[2] C.M. Heirwegh, J.L. Campbell, G.K. Czamanske, Nucl. Instrum. Meth. B 366 (2016) 40-50.

P-8: Some effects of non-linearity in SDD-based PIXE systems

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We have discussed in detail elsewhere [1] our observation of non-linear behaviour in one SDD-based PIXE system. Peaks of the lightest elements (X-ray energies 1 - 5 keV) were found to undergo an “apparent energy shift” relative to the linear channel-energy calibration established using diagram lines having energies above 5 keV. A means has been developed for accurate quantification of this effect. As a result, a simple correction can be introduced into GUPIX to compensate for the non-linearity. More than ten SDD systems have now been examined, including the APXS units on Mars rovers [2], with non-linearity observed in more than half the cases. The “apparent energy shifts” can be either negative or positive. Non-linearity introduces distorting effects in the low-energy region of PIXE spectra which in turn impacts analytical accuracy. Some examples of these in one particular SDD-based system are given.

[1] C.H. Heirwegh et al., Nucl. Instrum. Meth. B 366 (2016) 40-50.

[2] J.L. Campbell et al., Nucl. Instrum. Meth. B 383 (2016) 143-151.

P-9: Silicon Drift Detector response function for PIXE spectra fitting

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The accurate determination of the X-ray peak areas in PIXE spectra by fitting with a computer program requires an adequately good parametrization of the detector response function. In the Guelph PIXE software package, GUPIXWIN, the most used PIXE spectra analysis code, the response of a semiconductor detector to monochromatic X-ray radiation is described by a linear combination of several analytical functions: Gaussian or Voigt for the X-ray line itself, and additional tail contributions (exponential tails and step functions) on the low-energy side of the X-ray line to simulate incomplete charge collection effects. The literature on the spectral response of silicon X-ray detectors for PIXE applications is rather scarce [1,2], in particular data for Silicon Drift Detectors (SDD) and for a large range of X-ray energies are missing.

In this work a series of thick ultra-pure (99.99+%) mono- or bi-elemental targets were irradiated with low intensity 0.85 MeV protons and corresponding X-ray spectra from 1 to 15 keV were measured using several SDD devices used for routine PIXE analysis at the INFN LABEC laboratory in Florence, differing for active area and thickness, and for preamplifier layout. The choice of low proton beam energy and thick targets resulted in a suppression of the continuous Bremsstrahlung background and in better line-to-background intensity ratios.

Using the analytical functions implemented in GUPIXWIN, the SDD response functions were satisfactorily reproduced for the X-ray energy range 1–15 keV. An attempt to describe with

simple polynomial functions the dependence of the parameters of the SDD response function on the X-ray energy, for easy implementation in GUPIXWIN, is also presented.

[1] Puc et al., Nucl. Instrum. Meth. B 189 (2002) 123-130.

[2] Eggert et al., Nucl. Instrum. Meth. A 568 (2006) 1-11.

P-10: Upgrade of the microprobe at Dresden-Rossendorf

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The nuclear microprobe that was in operation until 2104 at the Ion Beam Center of the Helmholtz-Zentrum Dresden-Rossendorf was installed in 1994 [1]. It has been in operation since then with only minor changes. This necessitated an upgrade to bring the setup up to current standards of technology and good working practice. This study presents the details of the upgrade and modernization process we have undertaken.

The major drawbacks of the old system were the poor resolution and low contrast and brightness of the optical microscope. However, a good optical image is essential to localise the areas of interest on, for example, large geological samples.

On the other hand, the main investigative tool is the focused beam of high-energy ions and the corresponding detectors. Any other system such as an optical microscope has to be designed around this equipment. A new custom-designed microscope has been installed for which the first light-collecting lens is mounted in the sample chamber at only a few centimetres from the sample. The light is then guided over large mirrors and focussed on a CCD camera outside the sample chamber. Also the illumination is fed in through the lenses instead of using a separate light source as in the old system. The lens system can also serve as a basis for a possible ionoluminescence detector.

Other improvements concern the beam deflection system, the control of the scanning system and the control and monitoring of all relevant parameters for the experiment. The control of the scanning system is done by custom-designed hardware to guarantee the real-time execution of the scanning without the need for a computer with a real-time operating system. This makes it possible to use a standard Windows based computer with commercial software for the data acquisition. A new channeltron has also been installed to detect secondary electrons that can be used to obtain a quick overview of a measurement area.

Technical details and first test measurements with the new system are presented.

Acknowledgements: The technical support of R. Aniol is gratefully acknowledged.

[1] F. Herrmann, D. Grambole, Nucl. Instrum. Meth. B 104 (1995) 26-30.

P-11: Setup and Features of a New External Micro-beam of the 5SDH Tandem Accelerator at LAEC

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A new external micro-beam is in ongoing preparation at the 1.7 MV tandem accelerator of the Lebanese Atomic Energy Commission. Despite the use of a RF ion source, it was possible to steer a measurable beam and extract it into air. The setup is performed using an assembly of object slits, collimating slits and two quadrupole magnets from “Oxford Microbeams”. Furthermore, an end stage from AGLAE, France was designed as “custome-made” to accommodate micro-PIXE analysis using 3 SDD detectors. A description of the beamline components as well as its features will be shown through some preliminary results and some case studies.

P-12: Development of tapered glass capillary microbeam PIXE mapping technique at Chiang Mai University

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Developing high technologies but in economic manners is necessary and also feasible for developing countries. At Chiang Mai University, Thailand, we have developed MeV-ion microbeam technology based on a 1.7-MV Tandetron tandem accelerator with our limited resources in a cost-effective manner. Instead of using expensive and technically complex electrostatic or magnetic quadrupole focusing lens systems, we have developed cheap MeV-ion microbeams using tapered glass micro-capillary technique for MeV-ion particle induced X-ray emission (PIXE) mapping. The capillary technique utilizes our home-fabricated tapered glass capillaries to realize microbeams. The paper reports technical details, installation, alignment and quality tests of the system. Fig. 1 shows this system. The system has been applied for MeV-ion PIXE mapping of biological and gemological samples. Some examples are presented.

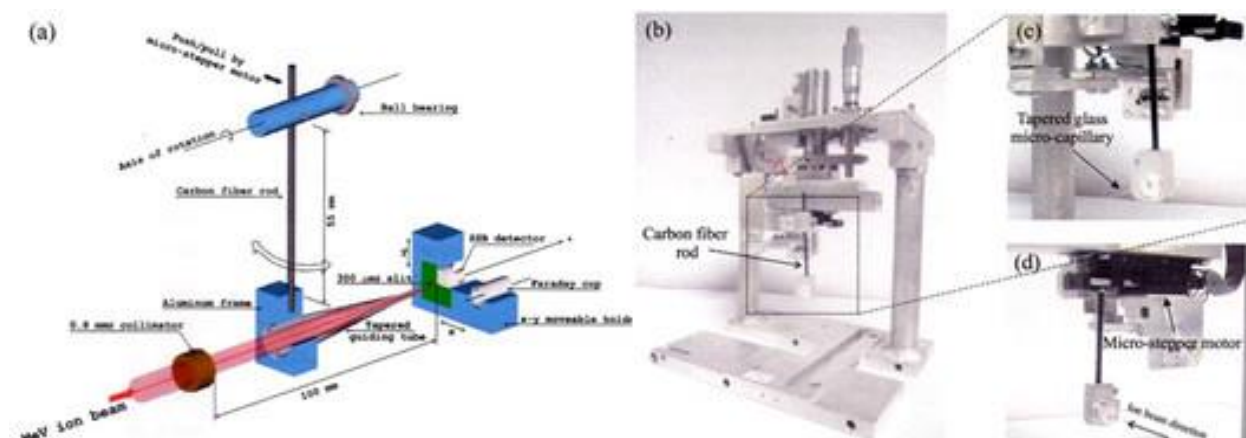


Fig. 1. Tapered glass capillary microbeam system: (a) Schematic diagram; (b) A photograph of the system; (c) and (d) Close-ups of the capillary holder.

P-13: New Parallel Beam Wavelength Dispersive Spectrometer (PB-WDS) at Ljubljana Ion Microprobe

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A new parallel beam wavelength dispersive (PB-WDS) X-ray emission spectrometer for high energy resolution PIXE analysis using microfocused proton beam has been constructed and installed at the microprobe of the J. Stefan Institute. Polycapillary X-ray optics is used to enhance the solid angle of X-ray collection and to transform collected proton-induced X-rays into quasi parallel beam which is analyzed using diffraction on a flat crystal. The use of optics enhances drastically the X-ray collection efficiency, while the energy resolution, defined by a very low divergence of the X-rays exiting the X-ray semi lens, is kept on the ~10 eV level. The whole spectrometer is installed in a vacuum chamber and operates in the 2-10 keV energy range. In this contribution, the design and construction of a new PB-WDS system coupled to our proton microprobe are described. The main operational characteristics are presented together with the results of characterization measurements used to determine the actual performance of the spectrometer. Finally, few selected experimental examples are given illustrating the capabilities of the new PB-WDS spectrometer in PIXE analysis and also fundamental research in atomic physics.

P-14: Chemical speciation of third-row elements by means of high energy resolution K β X-ray emission spectroscopy

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The partial induced X-ray emission (PIXE) is well known analytical technique for rapid multielemental detection. The technique is based on the detection of characteristic X-ray emission following inner shell ionization induced in collision with MeV protons. Solid state detectors with energy resolution around 150 eV are commonly used in PIXE technique, which is enough to separate characteristic X-ray lines from different elements. However, if the X-ray emission spectra are recorded with high-energy resolution the K β emission spectra start to reflect chemical environment of element. In present work, the high-energy resolution spectrometer in Johansson geometry [1] optimized for tender X-ray energy range was used to record K β emission spectra of several phosphorus (P), sulfur(S) and chlorine (Cl) containing compounds. In case of third-row elements K β emission spectra originate from valence-to-core (vtc) transition directly reflects the local symmetry of the compounds and ligand environments of the elements. The measured spectra are compared to the results of first-principle calculations based on density functional theory (DFT), included in the StoBe-deMon

molecular/cluster software package. The agreement between the measured and the calculated K β emission spectra permits to discuss valence orbitals in term of molecular orbital (MO) picture. The K β spectral shape of the XO $_4^{n-}$ and XO $_3^{n-}$ ions (where X is P, S or Cl) are decomposed to atomic orbitals [2]. The effects of ionic and covalent ligand bonding to PO $_4^{3-}$ are also presented [3]. Finally, *ab-initio* DFT calculation of the S K β spectral shape of lithium polysulfide (Li $_2$ S $_x$) compounds were performed, which can be used to study Li-S batteries in operando mode.

Acknowledgements: This work has been supported by Marie Curie Actions - Initial Training Networks (ITN) as an Integrating Activity Supporting Postgraduate Research with Internships in Industry and Training Excellence (SPRITE) under EC contract no. 317169.

[1] M. Kavčič et al., Rev. Sci. Instr. 83 (2012) 033113.

[2] M. Petrić et al., Inorg. Chem. 55 (2016) 5328-5336.

[3] M. Petrić, et al., Anal. Chem. 87 (2015) 5632-5639.

P-15: Chemical shifts of L X-ray absorption edges of thallium and valence state of thallium ion in its compounds and superconducting cuprates

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The X-ray absorption spectroscopy (XAS) is a very promising technique for investigating the structures and properties of materials. The position, shape of X-ray absorption discontinuities have been used to deduce structural and chemical bonding information on model compounds of thallium and its superconducting cuprates. The samples were characterized through X-ray diffraction. The LX-ray absorption edges of thallium and valence state of thallium ion in its compounds including superconducting cuprates were recorded by Rigaku X-ray Spectrometer. X-rays were monochromatized using a flat crystal of Si (1,1,1). It is seen that the chemical shift of L $_{III}$ absorption edges of thallium in the model compounds of thallium and superconducting cuprates is positive. The magnitude of the chemical shift lies in the range of 1.8 to 4.0. It is maximum in the case of thallic nitrate and minimum for thallic iodide. In all the superconducting cuprates, the chemical shift is similar to that in thallic nitrate suggesting thereby the presence of Tl $^{+++}$ ions in these cuprates. It is noted that in some compounds the thallium ions are in 1 $^+$ state and thallium-oxygen bonds have 38.7% ionic character, the magnitude of the chemical shifts are not the same. The L $_I$ spectra were simple in all cases. Splitting of the edge was not observed. In the monovalent compounds the 6s orbital is occupied, but 6d is completely vacant. A main peak corresponding to 2s to 6p transition was expected and since 6s is unavailable for transition to occur, no splitting of the L $_I$ edge was observed. The L $_I$ edge spectra of all the superconducting oxides were similar to Tl $_2$ O $_3$ and other trivalent thallium compounds. The intensity of this peak is slightly less in superconducting oxides as compared to that in Tl $_2$ O $_3$. An attempt has been made to explore if there exists a correlation between absorption shifts and the effective charges on the absorbing thallium ions. It is observed that the valence levels involved in the formation of chemical bonds are mainly responsible for the observed chemical shifts. The final level shifts its position with respect to that in the pure metal depending upon the effective charge on the absorbing atom. The results lead to the conclusion that the substitution of vanadium at thallium site does not change the

local environment around thallium ion. The L_{III} edge data in the complex superconducting oxides suggest octahedral environment about Tl^{+++} ion. The coordination numbers in these oxides estimated from curve-fitting analysis are almost same and are equal to six confirming the predictions of XANES analysis. The near-neighbor distances in these oxides are almost same within the uncertainty in the measurements. Their values agree with those in trivalent thallium compounds where thallium is coordinated to six oxygen neighbors.

P-16: Analysis of photon emission induced by light and heavy ions in time-of-flight medium energy ion scattering

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Medium energy ion scattering (MEIS) is commonly employed to characterize thin films by detection of backscattered particles. However, the combination of a pulsed beam and a time of flight (ToF) detection system additionally allows for the observation of secondary particles like photons. The present study shows a first systematic analysis of the photon yields from thin Au, TiN and HfO_2 films exposed to pulsed ion beams. ToF-MEIS experiments were conducted at the MEIS facility at Uppsala University [1]. Photons were detected by the same micro-channel plate detector as employed for the detection of backscattered particles. Besides the variation of target materials, different species and energies of incident ions as well as several scattering angles were studied. The photon yield exhibits a dependence on all these variables.

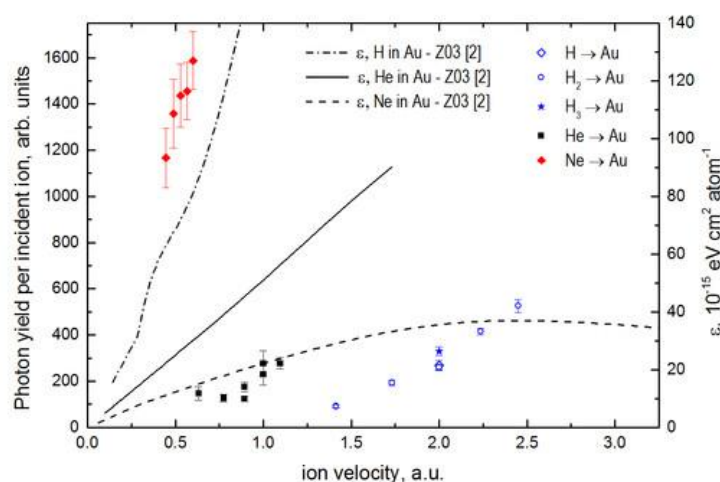


Fig. 1. Comparison between photon yields from Au films induced by different ion species. The used projectiles were H (ion velocity: 1.4–2 a.u.), ^4He (0.6–1 a.u.) and ^{20}Ne (0.44–0.6 a.u.). Also shown are the respective electronic stopping cross sections.

The figure compares photon yields from thin gold films induced by protons, ^4He ions and ^{20}Ne ions. To correct for the ion fluence from the ion source, the obtained yield is normalized by this quantity. Primary ion velocities in atomic units were 1.4 – 2 for protons, 0.6 – 1 for He and 0.44 – 0.6 for Ne. Also shown are the electronic stopping cross sections of the three ion species in gold. At comparable ion velocities (~ 0.6 a.u.) the photon yield for Ne is about ten times higher than for He. This difference is significantly higher than the one seen in the stopping cross sections (\sim factor 2.7). Photon yields for protons of such low velocities are not present in the

data set at hand. However, by interpolating from higher velocities it can be assumed that the photon yield from protons is even lower than the one from He. No significant difference was found in the photon yields from H, H₂ and H₃ ions. Furthermore, the photon yield depends on the target material. HfO₂, which is an isolator with a band gap of ~6 eV, exhibits a significantly higher yield than gold. This effect was observed both for proton and He projectiles. Likewise, the yields from ⁴He on Au and TiN films were compared. It was found that more photons are detected when using the TiN target.

[1] M.K. Linnarsson et al., Rev. Sci. Instr. 83 (2012) 095107.

[2] J.F. Ziegler, Nucl. Instrum. Meth. B 219-220 (2004) 1027-1036.

P-17: Feasibility study of combined Ambient Pressure MeV SIMS with PIXE

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Ion Beam Analysis comprises of numerous techniques among which MeV SIMS is the least known inside of the ion beam analysis community. Nevertheless, MeV SIMS, or the mass spectrometry of secondary ions sputtered with MeV heavy ions, is a powerful tool for molecular analysis and imaging of organic molecules. This surface sensitive technique finds its background in a technique known as PDMS (Plasma Desorption Mass Spectrometry) developed in 1970's by MacFarlane group with a purpose of analysing biomolecules. Both of the techniques, MeV SIMS and PDMS share the same underlying mechanism, i.e. electronic sputtering, which is still not fully understood. On the other hand, being a mass spectrometric technique of secondary ions makes MeV SIMS in some ways similar to conventional TOF SIMS. However, deploying a MeV instead of keV primary ions opens one special application for MeV SIMS which cannot be achieved with TOF SIMS. Since MeV ions can penetrate into air where they remain focused up to few millimetres, a development of ambient MeV SIMS can also be considered. The area of ambient mass spectrometry techniques has been rising rapidly in recent years, as the benefits of having analysis under ambient became recognised.

In the University of Surrey Ion Beam Centre first fully ambient pressure MeV SIMS setup has been commissioned. In this work we will explore the feasibility of combining Ambient Pressure MeV SIMS with PIXE which would provide both the molecular and elemental composition of the investigated material. The setup needed to couple these two techniques for analysis and imaging will be described, and the potential applications examined.

P-18: Growth and characterization of Pt-Si nucleation seeds for silicon nanowires synthesis

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Synthesis and application of Si nanowires have gained much interest in recent years in the area of thin film-based devices due to their low dimension [1]. However, the process of producing

them requires proper understanding and preparation of Pt-Si nucleation seeds on Si substrate as template. Solid-state dewetting has been reported to be the innovative method of producing well-ordered nucleation seeds necessary to control nanowire growth [2]. In this study, n-type silicon <100> wafer was used as a substrate and Pt coating as a catalyst. Pt layer, 20 nm thick, was deposited using electron beam evaporator at a deposition rate of 0.8 Å per sec in 5×10^{-6} mbar vacuum pressure. To study the formation of nucleation seeds, two set of samples were thermally annealed at 800°C and 950°C for 30 minutes and 2 hours, respectively. The Pt-coated Si samples were characterized using Rutherford Backscattering Spectrometry (RBS), X-ray diffraction (XRD), Scanning electron microscopy (SEM) and particle induced X-ray emission (PIXE). SEM revealed surface morphology with evenly distributed droplets of varying diameter, ranging from 10 nm to 80 nm, after annealing. To study the annealing effect on the interaction between Pt and Si, *in-situ* RBS (from 25°C to 400°C at $\sim 2^\circ$ per minute) was performed using mono-energetic beam of alpha particles (2 MeV) in vacuum pressure of 5×10^{-6} mbar. Silicide phase with PtSi stoichiometry, associated to the seeds, was observed to be the most stable and the last phase to form. It was confirmed using XRD to be orthorhombic with unit cell parameters $a=5.938$ Å, $b=5.59600$ Å and $c=3.60350$ Å. To further understand elemental composition and impurities in these samples, PIXE technique in a mapping mode was used and results analysed offline using GeoPIXE software [3]. PIXE maps taken from samples annealed at 800°C depicted homogeneous distribution of Pt as expected. Selected regions and point analysis revealed that the Pt and Si distribution in each droplet correspond to the stoichiometry of the PtSi phase. Similar attributes were also observed in the samples which were subjected to In-situ real-time RBS. These results paved a way to successful synthesis of Si nanowires for nano-devices fabrication.

[1] S. Strobel, C. Kirkendall, J.-B.Chang, Nanotechnology 21 (2010) 505301.

[2] A. Benkouider et al., Nanotechnology 26 (2015) 505602.

[3] <http://nmp.csiro.au/Geopixe.html>

P-19: Two-dimensional PIXE-channeling: determining small lattice distortions and lattice location of dopants in topological insulators

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Topological insulators are a class of novel materials displaying topological surface states protected by symmetry: crystal symmetry in topological crystalline insulators (TCI) such as $\text{Pb}_x\text{Sn}_{1-x}\text{Te}$, and time reversal symmetry in Z_2 topological insulators (Z_2 -TI) such as Bi_2Te_3 . Breaking these symmetries allows to tune the topological phase, for example, by breaking crystal mirror symmetry in TCI via a small rhombohedral distortion, and by breaking time reversal symmetry in Z_2 -TI via magnetic doping. We are investigating these two cases (rhombohedral distortion in TCI and lattice location of magnetic dopants in Z_2 -TI) using particle induced X-ray emission (PIXE) channeling. Unlike conventional implementations of ion channeling, for which only 1-dimensional angular scans are typically used, we measure the full angular dependence (2-dimensional channeling spectra) in the vicinity of major crystal axes, and fit the spectra with accurate simulations. This approach, which we have been developing and applying in the context of electron emission channeling, greatly enhances the capabilities

of channeling techniques [1]. As an example, Fig.1 shows PIXE-c measurements in the vicinity of [111] and [100] axes of PbTe and GeTe epitaxial thin films. Whereas PbTe has the ideal cubic rocksalt structure, GeTe exhibits a small rhombohedral distortion along the [111] axis. The effect of this small distortion on the channeling spectra is so strong that it is evident by mere visual inspection, when comparing the off-surface direction [100] of PbTe and GeTe: in GeTe, all planar channeling is suppressed except for one of the (110) planes (Fig. 1). By fitting such experimental data with accurate simulations, we aim to quantitatively characterize the rhombohedral distortion in TCI and the lattice location of magnetic dopants in Z_2 -TI, beyond what is currently achievable with other experimental techniques. While promising, this approach is still under development. Here, we present and discuss various developments which are currently underway, including simulation and fitting software, and more technical aspects (e.g. absorbers optimized for specific X-ray lines, and minimization of Carbon deposition during the long irradiation/measurement).

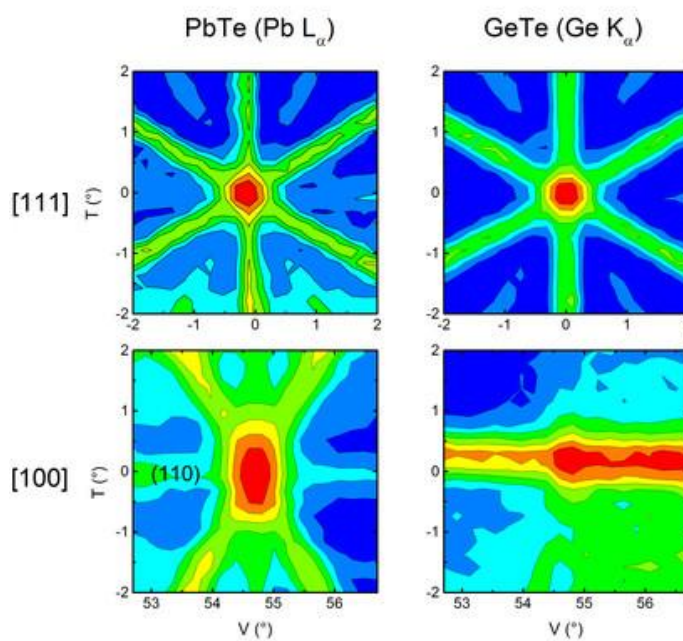


Fig. 1. Two-dimensional PIXE-c measurements of PbTe and GeTe along the surface direction [111] and off-surface direction [100].

[1] L. M. C. Pereira et al., Nucl. Instrum. Meth. B 332 (2014) 143-147.

P-20: *In situ* PIXE and RBS/c analysis during ion track formation in SiO₂

Marko Karlušić (1), Stjepko Fazinić (2), Kristina Tomić (1), Ivana Zamboni (2), Iva Božičević Mihalić (2)

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Materials modifications using swift heavy ions has found widespread technological applications, including track etch membrane production, hadron therapy, nuclear waste immobilization and

testing of electronic devices. However, basic understanding of underlying physical processes is still plagued by controversies and inconsistencies. At present, hotly debated topic is the origin of the velocity effect, i.e. why damage cross section (ion track size) increases for low ion energies in insulators [1,2,3]. Szenes proposed that damage cross section increase is related to activation of the Coulomb explosion mechanism due to high ionization of the matter along the ion trajectory [2]. Therefore, we performed high resolution PIXE measurements of Si K X-rays during ion irradiation and the ion track formation to evaluate suitability of PIXE for experimental test of Szenes interpretation. Also, we made complementary measurements of damage build-up during ion track formation in quartz SiO₂ using *in situ* RBS/c. The first obtained results of these *in situ* ion beam analyses are reported.

[1] M. Toulemonde et al., Phys Rev. B 85 (2012) 054112.

[2] G. Szenes, Nucl. Instrum. Meth. B 298 (2013) 76-80.

[3] M. Karlušić et al., New J. Phys. 19 (2017) 023023.

P-21: Elemental analysis of uranium pellets by PIXE in nuclear forensics

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Nuclear forensics is the analysis of unknown nuclear or other radioactive material to provide evidence for nuclear attribution in a legal context. Nuclear forensics is an essential component of national and international nuclear security response plans to events involving radioactive materials diverted outside of radioactive control. The goal of the analysis is to identify the composition, origin, and intended use of interdicted nuclear or radiological samples. Nuclear forensic analysis includes the multi-aspect characterisation of the material and correlation with its production history.

The analysis of samples covers a wide range of characteristics: elemental/chemical/isotopic composition, structure, age, etc. These properties to be determined require diverse analytic tools. An effective tool of elemental analysis is the PIXE spectrometry. Complementary analysis by PIXE and a bulk analysis method can provide detailed elemental composition of samples. Some elements are added to the uranium intentionally to modify the properties of the pellets. Other elements are impurities from the production technology. In some cases trace element contamination is added intentionally to pellets in order to support the tracing of them.

A set of samples, majority of them were low enriched (LEU) power reactor pellets either known or unknown origin were analysed by external and conventional PIXE. The determined elemental compositions are discussed and compared with that of obtained by other analytical techniques such as mass-spectrometry.

[1] Klaus Mayer, Maria Wallenius, Ian Ray, Analyst 130 (2005) 433-441.

P-22: Investigation of thickness and composition on aging of Polyimide thin films at high temperature using RBS Technique

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The need for faster devices with high voltage and high switching frequency capabilities, operating at higher than 150°C junction temperature is growing or up to 400°C in environmental temperature. However, such devices able to work under such conditions need appropriate packaging to sustain operation throughout their entire planned life cycle. Therefore, particular attention has to be paid to material selection when designing high temperature packaging. Polyimides (PIs) have received considerable attention due to their high thermal stability, mechanical and electrical insulating properties.

A series of 12 PI thin films was spin-coated onto Silicon substrate and then cured at 400°C for one hour in an oven under N₂ atmosphere. The final film thickness of 1.5 µm was obtained and measured before aging using a KLA Tencor profilometer. The obtained sample was divided into two parts. The first consist of six samples of PI deposited onto Silicon substrate. The second six samples were covered with a gold (Au) thin film with an approximated thickness of 180 nm by thermal evaporation. Each sample is aged at 300°C in open air at different times from 0h to 1500h.

RBS technique was employed to monitor the composition and the thickness of PI material versus aging times. The RBS measurement was done by using accelerated alpha-particle beams of energy 2 MeV. It was found that as the aging time increases the PI thickness in PI/Si samples decreases following an exponential decay, while that in Au/PI/Si it almost stable. This decrease in thickness is suggested to the surface degradation mechanism. It is argued that the presence of Au layer upon the PI/Si led to protection of PI against aging effects.

Moreover, a peak related to Silicon (Si) was observed on the top of the samples with increasing aging time. The reason of the appearance of this layer is assumed to be due to the impurity inside the oven.

P-23: Different adaptation strategies in seeds of Zn hyperaccumulating plant *Arabidopsis halleri*

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Zinc is an essential plant micronutrient but is toxic in excess. To cope with excess Zn, plant species must possess a strict metal homeostasis mechanism.

Arabidopsis halleri (L.) Brassicaceae is a European, perennial plant with significant heavy metal tolerance and ability to hyperaccumulate Zn and Cd. This species is a pseudometallophyte, thus

can be found on both metalliferous and non-metalliferous sites. In spite of extensive related work that has been carried out on *A. halleri* in different fields there is no data concerning chemistry and elemental distribution in seeds of this species.

Generally, plants exclude heavy metals from seeds which are a crucial stage in their development. Studies on hyperaccumulating plants show different metal concentration patterns in their seeds.

The aim of this study was to compare the elemental concentration and distribution in seeds from metallicolous (M) and non-metallicolous (NM) populations of *A. halleri* growing naturally. Seeds from four populations growing in Poland – two M – from Bolesław and Galman, and two NM from the Tatra Mountains and Niepołomice Forest – were collected during summer 2015. Three seeds per population were analyzed using the nuclear microprobe (PIXE and EBS) at the Materials Research Department of iThemba LABS. Concentrations and distribution of P, S, Cl, K, Ca, Mn, Fe, Cu and Zn in the seed sections were obtained. The analysis of micro-PIXE maps of elements' localization in *A. halleri* seed sections was carried out on ten separated regions: testa with extracted hilum, chalazal and micropular region, embryonic axis with root tip and hypocotyl, cotyledons and vascular tracts of cotyledons, and the root.

Significant and unexpected differences were found in the Zn concentrations and distribution between populations. The highest Zn concentration of the whole seeds was found in NM population (Niepołomice Forest) growing on soil with a very low concentration of Zn. In contrast, the lowest Zn concentrations of the whole seeds were detected in M populations growing on extremely polluted site in Galman and in NM population collected in the Tatra Mountains.

Regarding the seed sections, the highest concentration of Zn was detected in the chalazal and hilum region in samples from two NM populations and one M population (from Bolesław). Exclusion of metals from the embryonic tissues may be a strategy adapted to achieve reproductive success in the contaminated environment.

Surprisingly, in seeds from Galman (M) the differences between tissues were small and relatively high amount of Zn was found in the embryonic axis, tip of cotyledon and hypocotyl.

The main localization of K, P, Fe and S was in the cotyledon and embryonic axis of all analyzed seeds while Ca and Cl were accumulated mainly in the testa. Cd was detected in one M population only (Bolesław).

P-24: Mapping whitefish natal spawning grounds and hatcheries by measuring otolith core strontium

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Otoliths from juvenile whitefish were analyzed with particle induced X-ray emission spectrometry. The juvenile whitefish were collected from different hatcheries in Finland and had spent all their life in the same water habitat. The obtained concentrations were compared to results of analyses of otolith cores from adult whitefish caught at sea. Matching results reveal the hatchery.

Whitefish (*Coregonus lavaretus* (L.)) in the Baltic Sea can be divided into two groups according to their spawning strategies: one spawning in shallow bays along the coastline and one spawning upstream rivers. The wild river spawning whitefish is endangered and is in risk of becoming extinct. To estimate the amount of river spawning population is challenging as the two groups are visually identical and form mix populations at sea. The huge amount of stocked river spawning whitefish bred in hatcheries complicates the estimation further.

The core of the otolith of the adult fish holds information on the natal spawning ground. A high concentration of strontium in the core indicates an early age spent in salty water and a low concentration indicates hatching in a river or a lake. However, whitefish juveniles released to the sea from hatcheries will also have a low concentration of strontium in the core. Another problem is that some of the wild juveniles spend only a short time in the river before migrating to the sea. The area of the otolith corresponding to this time is very small. These fishes can therefore falsely be regarded as sea spawners. The stocked fish is normally kept in fresh water for almost a year before their release to the river mouth or sea and have therefore a huge fresh-water signature.

By analyzing otoliths from juveniles we will build a database that helps identifying the hatchery or the natal spawning ground of the adult whitefish captured at sea.

P-25: Non-destructive Analysis of the content of a Egyptian flacon – without opening it

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High-energy PIXE offers the advantage of a large possible analytical depth when looking for heavy elements. Thin gold foils have been detected behind 3 mm of lead glass. Thus, high-energy PIXE could be successfully applied to the analysis of the content of an Egyptian flacon. The flacon still had the original seal and the Egyptologists were reluctant to break it in order to gain information about the content.

The measurements, the data analysis and the results will be presented.

P-26: Focusing the ion beam on Byzantine pottery discovered at Oltina (Constanța County), Romania

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A set of ceramic shards unearthed in archaeological excavations at Oltina, in the South-Eastern part of Romania, dated to the 10th-11th centuries AD, were subjected to archaeometric investigations, in order to reveal the manufacturing techniques and raw materials employed by the potters from the Low Danube zone during the Byzantine ruling. This study is a continuation of the previous analyses on pottery from Harșova [1], and it was triggered by the possibility to identify possible regional commercial connections during that historical period.

The grouping of the shards according to stylistic reasons was refined by the subsequent petrographic study. Optical Microscopy (OM) observations detailed the potteries fabric by the textural characteristics, porosity and microstructure, surface treatments and firing. The petrographic conclusions agreed quite well with the initial separation of the shards into fine and coarse ceramics, but indicated an important variability in terms of mineral composition, homogeneity and porosity.

Micro-PIXE analyses of potteries were realized at AN2000 accelerator of LNL, INFN Italy, and led to the identification of chemical composition of the ceramic shards - bodies and decorations. Micro-PIXE mapping of the interfaces between the decorated surfaces and the ceramic bodies provided clues about the composition of the green glaze, decoration characterized by a strong enrichment of the lead oxide content compared to the corresponding ceramic body.

Principal Component Analysis (PCA) of the PIXE data evidenced two main categories of shards with distinct compositional signatures. Thus, the ceramic fragments made of kaolinitic clays were clearly dissimilar from the rest of the samples. Statistical analysis also identified several outliers; archaeological explanations for their presence in the studied assemblage were sought for.

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[1] Roxana Bugoi et al., Nucl. Instrum. Meth. B 348 (2015) 296-301.

P-27: Quantitative elemental analysis of Bronze Age situla by external micro-PIXE

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A situla, i.e. a bucket-shaped vessel from the Late Bronze Age was investigated by in-air micro-PIXE at the scanning nuclear microprobe in Atomki, Debrecen. The situla belongs to the so called Hajdúböszörményi hoard, which was excavated illegally in the Csege-halom area, near Hajdúszoboszló in Eastern Hungary. Firstly, the vessel appeared on the Internet in 2006 where someone tried to sell it on the black market. However, the Hajdúsági Museum filed a complaint against an unknown person. As a result, with the police intervention the object was prevented from selling abroad.

Altogether 21 measurement points including plates, loops, rings and nails were selected on the situla to get information about the composition of the different parts of the vessel. Quantitative elemental concentrations from S to Pb were determined by external micro-PIXE analysis which was carried out at the Laboratory of Ion Beam Applications of MTA Atomki [1]. A hierarchical cluster analysis was performed on the obtained dataset in order to group the different parts of the situla.

The quantitative analysis indicated that besides the main components of Cu and Sn the following trace elements were present: Fe, Co, Ni, Zn, As, Pb, Ag, Sb. Four groups were identified by the cluster analysis: plates with ~84wt% Cu concentration, Cu-rich (~87wt%) rivets, loops and nails with higher amount of Co and Ni, and finally Sn-Pb-rich ring, plate and nail.

[1] Zs. Török et al., Nucl. Instrum. Meth. B 362 (2015) 167-171.

P-28: Micro-PIXE mapping to identify provenance of prehistoric iron oxide based pigment

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Natural ochers and iron oxide based pigments are common in numerous archaeological contexts since the Paleolithic period. Elemental composition can provide information on the mineralogical composition and it has been demonstrated that trace element analysis of the iron-oxide phase is relevant for provenance identification [1,2]. However, these rocks are very heterogeneous at macro- and microscopic scales and can present variable proportions of accessory minerals. Moreover, when natural ochers and iron oxide are used as pigment or paint for decorating walls or objects, their presence in very thin layers on the surface of stones or

biomaterials such as bones and ivory can limit accurate fingerprinting. Micro-PIXE mapping takes into account the heterogeneity of the samples. The development of elemental PIXE imaging system such as recently implemented on the AGLAE accelerator (C2RMF, Paris [3]) has been used to select the areas of interest. In this work, PIXE imaging data sets obtained at various scales were used to identify heterogeneities of iron oxide based pigments. In rocks, iron oxide phase and non-ferrous minerals such as quartz and clays are differentiated. For archaeological artifacts, the most preserved layer of pigment is selected. As a result, only the iron phase is selected and analyzed for multivariate data analysis and hierarchical clustering. Pixel displaying low contents of iron is discarded from further data processing. The results show a better classification that enhances samples grouping in the framework of provenance identification. This procedure has been applied on two archaeological sites: the shelter of Pataud in France (28 000-26 000 years BP) and the El Mnasra (100 000-75 000 years) cave in Morocco.

[1] R.S. Popelka-Filcoff et al., J. Archaeol. Sci. 35 (2008) 752-762.

[2] L. Pichon et al., Nucl. Instrum. Meth. B 318 (2014) 27-31.

[3] L. Beck et al., Nucl. Instrum. Meth. B 273 (2012) 173-177.

P-29: Non-Destructive Study of Iron Gall Inks in Manuscripts

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Iron-gall inks were the most widespread writing ink in Europe from the early middle ages up until the 20th century. These inks are composed of Fe (II) sulphate, gallnuts, certain kind of gum (most often gum arabic) and sometimes other additives. The degradative effect of ink corrosion on paper supports is long known, but the complex mechanisms behind these deteriorative processes are still being researched.

This study was undertaken in order to better understand the state of preservation of iron-gall ink manuscripts, as well as the effects of conservation treatment performed upon them. Inks and paper supports were analyzed using non-destructive techniques. For elemental analyses PIXE, XRF and SEM/EDS were used. The elemental concentrations obtained by the external PIXE method were used for analysis of the paper and ink compositions as well as for tracing the compositional changes due to conservation treatment. Chemical composition was analyzed by FTIR spectroscopy. UV photography and IRR were used for ink differentiation and perception of migrations. All non-invasive methods used for object characterization were repeated after the conservation treatment.

Results indicate that ink characterization is possible through distribution of major and all minor and trace elements contained in the ink. Semi-quantitative analysis done by PIXE makes it possible to collect the so-called ink composition fingerprints expressed through the relative ratio between the major element in the ink composition (Fe) and minor elements. Such

fingerprint collection allows differentiation among various iron gall inks and establishing potential links between the chemical composition and the deterioration state of each manuscript respectively.

The analysis of manuscripts after conservation procedures (which included washing in ethanol/water mixtures, chelating treatments with calcium phytate and neutralization with calcium bicarbonate) shows a slight shift in the ratios between major and minor elements. This goes to show that the conservation treatments changed the chemical properties of paper and ink, even though no visual changes of the ink was detected. The lower amounts of Fe were observed in analyzed areas. Results indicate that the manuscripts were chemically stabilized.

P-30: Analysis of modern inks – comparison of PIXE and MeV-SIMS

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Modern ink materials, particularly ballpoint pens, tend to use a mixture of inorganic-based pigments, organic-based pigments and organic-based dyes in addition to solvents and other additives [1]. Due to non-destructive character, which is important when historical documents or documents important for forensic evidence are analysed Ion Beam Analysis (IBA) methods are often the methods of choice. In the present work, modern inks were analysed using Proton Induced X-Ray Emission (PIXE) and MeV Secondary Ion Mass Spectrometry (MeV-SIMS) and advantages and disadvantages of both techniques were discussed.

In the analysis of modern ink materials, PIXE and micro-PIXE can give us information on ink elemental composition, which can be a problem when organic-based pigments and dyes are analysed. In modern inks concentration of metals present in the inks is rather low and Ca signal coming from the paper is the most dominant one.

Contrary to PIXE, MeV-SIMS is giving information on ink molecular content from the uppermost layers which means that molecular composition of the inks is very well separated from the composition of the paper. Another important advantage of MeV-SIMS is that in a scan mode, allows for the differentiation of ink lines, and identification of deposition order of intersecting inks, by comparing unique m/z peaks and m/z peak ratios within single scans. This has been successfully carried out with all combinations of a set of 4 blue ballpoint pens from different manufacturers, and deposition order has been experimentally confirmed with experimental set-up for each deposition.

Research into non-destructive determination of the deposition orders of optically similar intersecting ballpoint pen lines is of high importance to forensic investigation as current methodologies for inks are destructive or quasi-non-destructive. Ion Beam Analysis techniques, although being destructive on a molecular level, provide microscopic damage that is generally not visible to the naked eye.

[1] J. Denman et al., Appl. Surf. Sci. 256 (2010) 2155-2163.

P-31: Comparison of PIXE and XRF analysis of airborne particulate matter samples collected on Teflon and quartz fibre filters

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In the framework of research projects for sampling and analysis of airborne particulate matter, Particle Induced X-ray Emission (PIXE) and Energy Dispersive X-ray Fluorescence (ED-XRF) techniques are routinely used in many laboratories in the world to determine the elemental concentration of the particulate matter samples. In this work an inter-laboratory comparison of the results obtained analyzing several samples (collected on Teflon and quartz fiber filters) with both techniques is presented: the samples were analyzed by PIXE (in Florence, at the 3 MV Tandetron accelerator of INFN-LABEC laboratory) and by XRF (in Elche, using the ARL Quant'X, Thermo Scientific Inc, USA, EDXRF spectrometer with specific conditions optimized for specific groups of elements). The results of the two sets of measurements are in good agreement for all the analyzed samples. A comparison of the Minimum Detection Limits for both techniques will be shown for all the measured elements and for the two different substrata.

Moreover, the comparison of PIXE and XRF results on Teflon and quartz fiber filters allows the quantification of possible self-absorption effects due to the penetration of the aerosol particles inside the quartz fiber-filters.

P-32: Fingerprint and footprint of atmospheric aerosol sources over a Hungarian city

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Aerosol pollution is a major environmental problem in urban areas due to negative impact of PM on health and on the built environment. In cities, aerosol particles can originate from local (e.g. biomass burning for residential heating, local industrial emissions or traffic) or from distant sources (e.g. sea salt, desert dust or regional industrial emissions). In Debrecen, Hungary, at an urban background station, concentration and elemental composition of PM₁₀ and PM_{2.5} is monitored regularly since 1993. On the basis of 24-hours samples collected twice a week we study the characteristics of the aerosol pollution of this region, including the effects of long range transport. The aerosol concentration is measured by gravimetry and the elemental composition (for $Z > 13$) is determined by Proton Induced X-ray Emission (PIXE) method at the macro-PIXE chamber in the IBA Laboratory of Atomki.

In order to get a picture about the geographical distribution of main pollution sources affecting a specific place, receptor modelling combined with backward trajectory calculation is applied. NOAA-HYSPLIT backward trajectory model [1] was used to define the movement of air masses, which reached Debrecen. Correlation analysis (Spearman's correlation) was applied to identify elemental fingerprints. However, backward trajectory usually indicates only the main directions of the transport and the subjective analysis of the paths is not always conclusive. A more comprehensive methodology for quantitative analysis of the observed footprints is adjoint dispersion modelling. Therefore our research was completed with source identification via footprints using the SILAM dispersion model [2], which provided integrated maps of distant sources affecting the observation site in each particular day. Footprints are the areas over where the air passes before being collected by the observational device.

Using correlation analysis we could separate some tracers (fingerprints) for remote anthropogenic sources, e.g. oil combustion/industry with S-V-Ni strong correlation. S is present in the aerosols in the form of sulphate, which is one of the tracer element of the heavy industry. The residence time of sulphate aerosol in the atmosphere is few days, therefore it should be associated with long range and regional transport. As an example footprints of sulphate aerosol are shown on Figure 1 on days when the concentration of PM_{2.5} was high and S appeared together with V and Ni. The potential source of this kind of pollution could be located in Eastern and Southern Europe for these days.

The results on possible regional and distant source areas obtained from the combination of the two approaches will be presented.

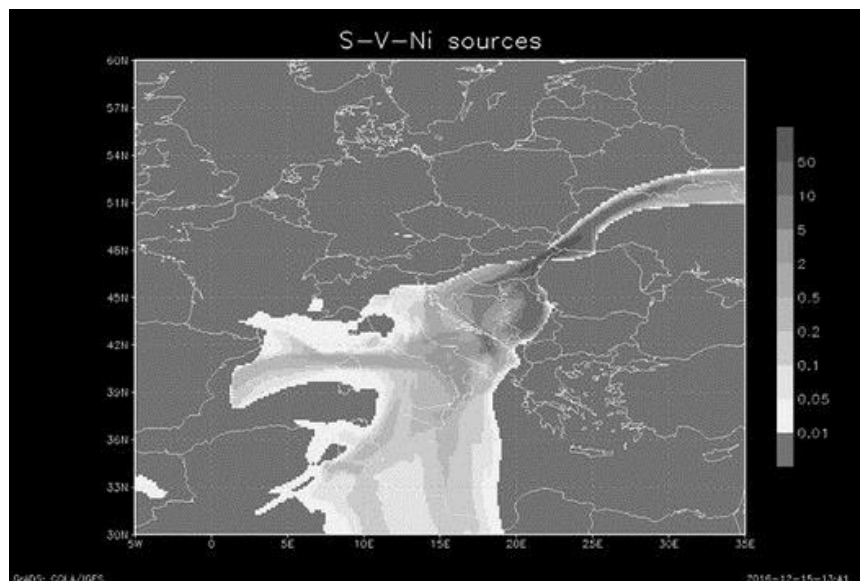


Fig. 1. Footprints of sulphate aerosol on dirty days, when S appeared together with V and Ni.

[1] A.F Stein et al., American Meteorological Society (2015) 2059-2077.

[2] M. Sofiev et al., Geosci. Model Dev. 8(2015) 3497-3522.

P-33: Study of aerosol pollution on public transport vehicles

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Transportation is part of our everyday life which contributes to the aerosol pollution of our environment. An average person spends every day 1 hour with travelling, and 20% of this with public transportation. In order to get information about the aerosol pollution and exposition of the passengers on public transport vehicles we measured the PM_{2.5}, PM coarse concentrations and elemental composition and mass size distribution of aerosols on different vehicles in Debrecen in September 2012 and in March 2014.

Aerosol samples were collected with a two stage personal sampler and a four stage personal cascade impactor on a bus, a new and an old tram, a trolley bus, an intercity train and on the Budapest metro. The PM concentrations were measured by gravimetry, and the elemental composition was determined by PIXE method.

The PM concentrations on the different vehicles varied between 100 and 400 $\mu\text{g}/\text{m}^3$, while the PM_{2.5} concentrations were found to be between 7 and 170 $\mu\text{g}/\text{m}^3$. The lowest concentrations were measured on the trams, while very high pollution levels were found on the bus, on the IC train and on the metro.

The composition of the indoor pollution was compared to the average outdoor aerosol composition. We found that in most cases the composition of the aerosol pollution inside the vehicles were similar to the outdoor air. Nevertheless some elements were present in higher concentration in some vehicles. We found high amount of Fe, Cr, and Mn on the train, the metro and the tram. The source of these elements was obviously the rails. Sn, Mo, and Zr were found on the train, which might have originated from the abrasion of the overhead wire. We observed the highest mass and elemental concentration values on the metro, which has its own underground microenvironment.

In order to determine the origin of the different element indoor enrichment factors were calculated. Besides the above mentioned elements Cl had indoor source (cleaning) on most of the vehicles.

In the mass size distributions the highest concentrations were measured on the coarse fraction. In the fine fraction the elements appeared in higher amount in the 2,5-1,0 and 1,0-0,5 μm size ranges. The lowest concentrations were measured on the 0,50-0,25 μm size range.

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