PIXE MEXICO 2007

XI INTERNATIONAL CONFERENCE ON PARTICLE INDUCED X-RAY EMISSION AND ITS ANALYTICAL APPLICATIONS

CONFERENCE PROGRAM AND ABSTRACTS

PUEBLA, MEXICO, MAY 25-29, 2007

ORGANIZED BY

UNIVERSIDAD NACIONAL AUTÓNOMA DE MÉXICO
BENEMÉRITA UNIVERSIDAD AUTÓNOMA DE PUEBLA
INSTITUTO NACIONAL DE INVESTIGACIONES NUCLEARES

IN COLLABORATION WITH
INTERNATIONAL ATOMIC ENERGY AGENCY
CENTRO LATINOAMERICANO DE FÍSICA
SOCIEDAD MEXICANA DE FÍSICA

ELEVENTH INTERNATIONAL CONFERENCE ON PIXE AND ITS ANALYTICAL APPLICATIONS

PUEBLA, MEXICO, MAY 25-29, 2007

CONFERENCE PROGRAM AND ABSTRACTS

Organized by:

Universidad Nacional Autónoma de México
Benemérita Universidad Autónoma de Puebla
Instituto Nacional de Investigaciones Nucleares

In collaboration with:

International Atomic Energy Agency
Centro Latinoamericano de Física
Sociedad Mexicana de Física (División de Física de Radiaciones)

Sponsored by:

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MESSAGE FROM THE PRESIDENT OF THE INTERNATIONAL ADVISORY COMMITTEE, G. W. GRIME

It is now over thirty years since the first international PIXE conference was held at the University of Lund, Sweden. Now we are looking forward to meeting for the eleventh conference at Puebla in Mexico. During these three decades, PIXE has matured from being an esoteric spin out of Nuclear Physics to a mainstream analytical technique which is making important contributions in fields as diverse as climate change, salmon fishery policy and cultural heritage. At the Puebla conference we can look forward to discussing the usual exciting mix of new developments in PIXE theory and techniques together with advances in traditional and novel areas of application. On behalf of the International Advisory Committee I would like to extend a warm invitation to all scientists to join us for the 11th conference in the hospitable and vibrant atmosphere of this historic city.

WELCOME MESSAGE TO PIXE 2007

The Local Organizing Committee of the Eleventh International Conference on PIXE and its Analytical Applications (PIXE 2007) is pleased to welcome all participants to this conference. This is the first time the PIXE conference has been awarded to a Latin-American country. Therefore, we hope it will be possible to show the international community how this region has contributed to the development of PIXE, other related methods, and their applications. Moreover, we are eager to maintain the friendly environment found in past conferences and to offer the opportunity to visit a few of the prehispanic and colonial attractions existing in Mexico.

Javier Miranda
Local Organizing Committee Chair

SPONSORS

Several organizations are playing an important role for the successful organization of the PIXE 2007 Conference, either as sponsors or advertisers. We must express our full gratitude to:

- Universidad Nacional Autónoma de México
- Benemérita Universidad Autónoma de Puebla
- International Atomic Energy Agency
- Sociedad Mexicana de Física
- Centro Latinoamericano de Física
- John Wiley & Sons
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MANUSCRIPTS FOR CONFERENCE PROCEEDINGS

In case the authors wanted to submit full papers for publication in the Conference Proceedings, they should have sent the text by e-mail to the Conference Secretary at most by **May 11, 2007**. Two printed copies and a diskette per paper must be delivered to the Conference Secretary during registration. The paper must follow the **conference paper template**, available on the Conference web page (as a MS Word document). The contributed papers will be reviewed by referees selected from the Conference participants. The authors must correct the manuscripts and return them before **July 6, 2007**.

ELECTRONIC CONFERENCE PROCEEDINGS

Accepted papers will be published with an *electronic format* in the **Conference Proceedings**. The Proceedings of the PIXE 2007 Conference will be recorded on CD and distributed by mail to participants several weeks after the reception of corrected manuscripts, to the address provided by the attendants. The Proceedings will also be available on the Conference home page.

SPECIAL ISSUE OF X-RAY SPECTROMETRY

Some of the invited and contributed papers will be selected by the Conference Program Committee and the International Advisory Committee, for publication as regular papers in a Special Issue of **X-Ray Spectrometry** (XRS), published by *John Wiley & Sons*. The papers will be chosen in order to offer a significant sample of current PIXE research, both for the general audience in the area of X-ray analysis and the specialized reader. The selected papers will be subjected to the ordinary refereeing procedure of XRS. Extension limit for invited papers is 6 printed pages, while that for contributed papers is 4 printed pages. Detailed preparation instructions will be sent to the authors via e-mail, or can be found at the XRS web site. A link to this site is given on the Conference home page. The manuscripts of the papers selected for publication in XRS should reach Conference Secretary by **August 10, 2007**. Corrected manuscripts must reach the Organizing Committee not later than **September 14, 2007**. Deadline to complete the publishing procedure will be set by XRS.

BEST POSTER AWARD

There will be an Award to the best poster presentation. This poster will be selected by a special Committee, appointed by the International Advisory Committee. The Award will be given during the Closing Ceremony, on Tuesday, May 29.

SOCIAL PROGRAM

Several social activities are planned during the Conference:

- First day get-together, to take place on Thursday, May 24, evening, after the first registration is closed, at the Posada San Pedro Hotel;
- Welcome reception, to which all participants and accompanying persons are invited. This reception will take place on Friday, May 25, at a site near the Conference Venue.
- Conference trip, organized for Sunday, May 27. The traditional trip in the PIXE Conferences will include the prehispanic site of Cacaxtla, as well as the colonial cities of Cholula and Santa María Tonanzintla. The trip will depart from the Posada San Pedro Hotel, in the early morning.
- Conference banquet, on the evening of Sunday, May 27, starting at 19:30, at the Fonda de Santa Clara restaurant.
- International Advisory Committee meeting, planned for Monday, May 28.

ACCOMPANYING PERSONS PROGRAM

A program for accompanying persons is already scheduled. All tours will depart from the Hotel Posada San Pedro. It will include the following activities:

- *Walking tour in Puebla*. The City of Puebla was declared since 1987 "World Heritage", by its architecture, archaeology, religious expressions, art crafts and gastronomy. The downtown area and several surrounding buildings with lots of testimonies of the past will be visited. These include The Cathedral (completed in 1649, the second largest in Mexico has the tallest bell towers in Mexico [69 m]), the Rosario Chapel, a world marvel made out of stucco covered with gold films, The Municipal Palace, the Alfeñique house (it is one of the best examples of the local version of Baroque). We will walk through streets with typical candy factories and talavera (fine china-like wares).
- *Franciscan convents*. Franciscan priests established town districts right after the Spaniards conquest of Tenochtitlan (present-day Mexico City), and initiated the conversion of indigenous groups to the Roman Catholic faith starting in 1524. Some of the Franciscan convents to visit in Puebla are: Tepeaca (a solid, defensive construction of 16th c, is one of the country's earliest and most impressive), Tecali de Herrera (also built in the 16th c. is missing it's roof, however the Renaissance façade is still intact; here we will visit the onix and marble art craft makers), Cuauhtinchan is another convent of the XVI century where remnants of a mammoth can be seen).
- *Atlixco-Volcán Popocatépetl*. Puebla is located in a large valley surrounded on four sides by the mountains and volcanoes of the Trans-Mexican volcanic belt. Puebla residents have a magnificent view of two snow-topped peaks, the Popocatépetl and Iztaccíhuatl volcanoes. The heterogeneity of the valley offers a variety of activities. The trip includes a visit to a Popocatépetl sight seen (this volcano dubbed "smoking mountain" by the Aztecs is the second highest volcano in Mexico, towering at just over 5,400 m. It is also one of the most active peaks in the country), San Baltazar Atlimeyaya (a trout farm), San Pedro waterfall, Atlixco (from Aztec term Atl-Ix-Co: "water in the valley" by its creek abundance, is the best place to admire a whole variety of flowers), Cabrera (flower green houses), and a Franciscan convent.

REGISTRATION AND SCIENTIFIC SESSIONS

Registration will be open on Thursday, May 24, at the Hotel Posada San Pedro, starting at 5:00 PM. After this, participants may register at the *Edificio Carolino*, *Salón Verde* I, every day from 8:30 AM, except on Sunday, May 27, when the desk will not be open.
Oral sessions will be held at the *Salón Barroco*, second floor of the *Edificio Carolino*. Speakers are requested to give their presentations one day before their presentation, so they can be uploaded on time. PowerPoint 2003 will be used by default, unless the speakers request other specific software or overhead projector.
There will be two poster sessions: **PI** on Saturday, May 26, and **PII** on Monday, May 28. The sessions will take place at the *Salón Verde* II or *Salón Candiles* of the *Edificio Carolino*. Posters must be mounted not later than 2:00 PM of the corresponding day. Materials for mounting (double-sided scotch tape) will be provided by the Organizing Committee. The posters must be removed by the end of the session.

USEFUL INFORMATION

Internet access
Most of the hotels offer free internet access. However, a few Internet Cafés are located in the surrounding area of the Conference site and hotels (see map in page).

Currency
The official currency is the Mexican Peso (MX$). As of April, 2007, the exchange rate is about MX$11.00 per US dollar. Although the hotels may exchange foreign currency, a better rate can be obtained in money exchange shops or banks.

Credit cards
Most hotels, restaurants and shops accept credit cards such as MasterCard, Visa or American Express.

Personal insurance
Neither the PIXE 2007 organization nor the organizers will be responsible for any medical expenses incurred by participants and/or accompanying persons and will not accept any liability for personal injuries sustained, or for loss or damage of property belonging to Conference participants, either during or as a result of the Conference or any tours. Participants are strongly advised to secure their own health and travel insurance before leaving their home country.

Water and fresh vegetables
It is strongly advised neither to drink tap water nor to eat some fresh vegetables. Bottled water can be easily obtained in the hotels, drugstores or convenience stores.

Restaurants

1. La Casona
2. Fonda Santa Clara
3. Fonda La Mexicana
4. Royalty
5. Sanborns
6. Taqueria La Oriental
7. Vips
8. Sanborns Conv. Center
9. Convention Center area

Others:

BUAP Carolino building (PIXE 2007) ★

Banquet: Fonda Santa Clara ●

Bank $

Drugstore ✚

Money exchange Ⓢ

Convenience store (24h) △

Arts and crafts ✡

Hotels

HI Holiday Inn

G Gilfer Hotel

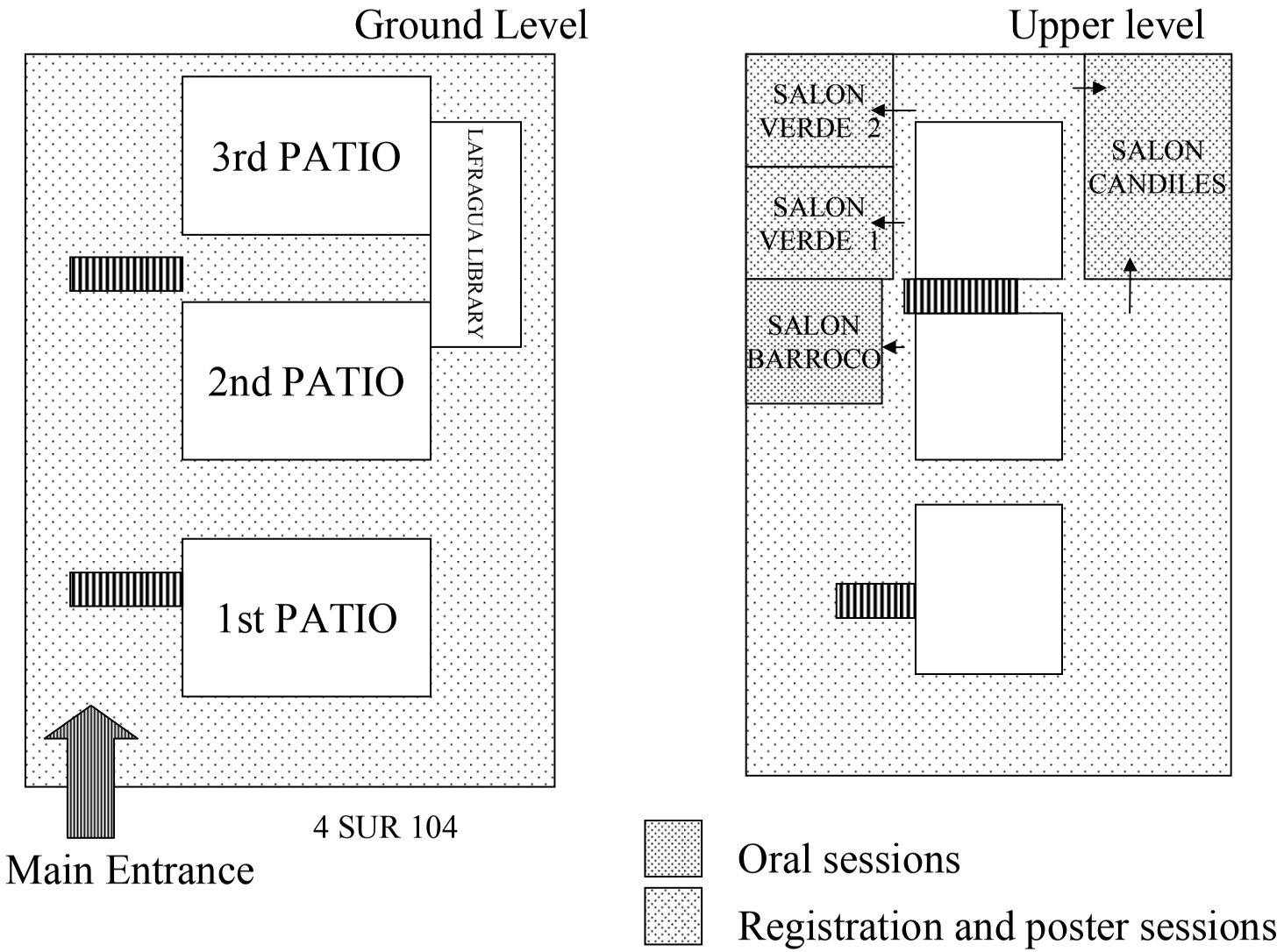
PSP Posada San Pedro

SA San Angel

PIXE MEXICO 2007

Puebla, Mexico, May 25-29,2007

Edificio Carolino



	Thursday	Friday	Saturday	Sunday	Monday	Tuesday
	May-24	May-25	May-26	May-27	May-28	May-29
8:30-9:00		**Opening**				
9:00-9:20		*Session A*	*Session E*		*Session H*	*Session K*
9:20-9:40		**G. Lapicki**	**N.R. Rebollo**		**K. Johnson**	**J. F. Dias**
9:40-10:00		M. Behar	A. Vaggelli	Conference	K. Dias da Cunha	C. Jeynes
10:00-10:20		S. Fazinic	N. Grassi	Trip	T.A. Cahill	B.L. Doyle
10:20-10:50		*Coffee break*	*Coffee break*		*Coffee break*	*Coffee break*
10:50-11:10		*Session B*	*Session F*		*Session I*	*Session L*
11:10-11:30		**K. Ishii**	**T. Calligaro**		A. S. Kerr W. Maenhaut	**A. Karydas**
11:30-11:50		Z. Szökefalvi-Nagy	Z. Smit		J. García-López	D. Sokaras
11:50-12:10		D. D. Cohen	L. Beck		C. Solís	V. Havranek
12:10-12:30		R. Correa-Devés	M. Kos		G. Pappalardo	R.G. Flocchini
12:30-14:00		*Lunch*	*Lunch*	*Lunch*	*Lunch*	**Closing**
14:00-14:20		*Session C*	*Session G*		*Session J*	
14:20-14:40		**A. Kreiner**	**G. Demortier**		**J. Höhne**	
14:40-15:00		E. Garman	G.W. Grime		F. Lucarelli	
15:00-15:20		W. Przybylowicz	D. Strivay		Workshop on	
15:20-15:40		R. Siegele	L. Pappalardo		GUPIX for Windows	
15:40-16:10		*Coffee break*	*Coffee break*		*Coffee break*	
16:10-16:30		*Session D*				
16:30-16:50		**A.I. Ektessabi**	Poster 1		Poster 2	
16:50-17:10		C.A. Pineda				
17:10-17:30	Registration	V. Purewal				
17:30-17:50		J. Mesjasz-Przybylowicz				
17:50-18:10		K. Sera				
			Round Table		**Round Table**	
19:00	Reception	Welcome Cocktail	**Archaeometry**	Conference Banquet	**Environment Sc.**	
			P.A. Mandó		W. Maenhaut	
			G.W. Grime		T.A. Cahill	
			Z. Smit		D.D. Cohen	

ORAL SESSIONS

Session A. Basic physical principles

Chairperson: J. Miranda

A-1. Invited Talk. **Ionization cross sections for high-energy PIXE.** *G. Lapicki*

A-2. **PIXE study of the influence of the Coulomb explosion on the molecular stopping power of C_2 in Si <100> direction.** *R. C. Fadanelli, M. Behar and J.F. Dias*

A-3. **Influence of chemical environment on the analysis of PIXE/XRF spectra of samples containing various compounds of 3d elements.** *S. Fazinić, M. Jakšić, S. Bamford, and L. Mandić*

Session B. Basic physical principles & Advances in experimental devices

Chairperson: F.D. McDaniel

B-1. Invited Talk. **Theory of atomic bremsstrahlung based on Binary Encounter Approximation.** *K. Ishii.*

B-2. **PIXE-PAGE analysis by scanning proton microprobe.** *A. Kocsonya, I. Kovács, Z. Szőkefalvi-Nagy, S. Lüthje, D. Hopff and M. Niecke.*

B-3. **Silicon detector deadlayer thickness estimates using proton bremsstahlung from low atomic number targets.** *D.D. Cohen, E. Stelcer, R. Siegele, M. Ionescu.*

B-4. **Elemental concentrations in aerosol samples determined by Artificial Neural Networks from PIXE spectrum.** *M.I. Dinator, R. Correa, I. Requena and J.R. Morales.*

Session C. Biology and biomedical sciences

Chairperson: Z. Szőkefalvi-Nagy

C-1. Invited Talk. **Application of a heavy-ion microprobe to the determination of microdistributions of a drug of potential interest for boron neutron capture therapy.** *A. J. Kreiner, P.Stoliar, M.E.Debray, M.E.Caraballo, A.A.Valda, J.Davidson, M.Davidson, J.M.Kesque, H. Somacal, H. DiPaolo, A.A. Burlon, and D. Minsky.*

C-2. **The characterisation of a contaminant-free support film for microPIXE analysis of biological samples.** *R.J. Southworth-Davies, A. Scothern, K. Leath, G.W. Grime and E.F. Garman.*

C-3. **Micro-PIXE studies of elemental distribution in mycorrhizal and nonmycorrhizal roots of *Ni*-hyperaccumulator *Berkheya coddii.*** *E. Orłowska, J. Mesjasz-Przybyłowicz, W. Przybyłowicz, and K. Turnau.*

C-4. **Localisation of trace metals in hyper-accumulating plants using μ PIXE.** *R. Siegele, N. Bhatia, M. Ionescu and D.D. Cohen.*

Session D. Biology and biomedical sciences

Chairperson: F. Folkmann

D-1. Invited Talk. **Applications of micro beam PIXE to investigations on neurodegeneration.** *A. Ide-Ektessabi.*

D-2. **Micro-PIXE analysis of bioconductive hydroxyapatite coatings on titanium alloy**. *C.A. Pineda-Vargas, M. Topić, T. Ntsoane and W.J. Przybylowicz.*

D-3. **The identification of historic biocide residues on herbarium material at the National Museum Wales.** *V. Purewal, B. Colston, S. Roehrs.*

D-4. **Nuclear microprobe studies of grasshopper feeding on nickel hyperaccumulating plants.** *M. Augustyniak, W. Przybyłowicz, J. Mesjasz-Przybyłowicz, M. Tarnawska, P. Migula, E. Głowacka, A. Babczyńska.*

D-5. **Daily changes of elemental concentration in a human body over 218 days obtained by quantitative analyses of beard samples.** *K. Sera, J. Itoh, Y. Saitoh and S. Futatsugawa.*

Session E. Complementary analytical techniques and other topics

Chairperson: B.L. Doyle

E-1. Invited Talk. **Luminescence of rare-earth doped zirconia: A phase stability study.** *N.R. Rebollo , F. González, J.L. Ruvalcaba-Sil, J. Miranda.*

E-2. **Micro-PIXE determination of Zr in rutile: an application to geothermometry of high-P rocks from the Western Alps (Italy).** *G. Vaggelli, A. Borghi, S. Calusi, R. Cossio, L. Giuntini, B. Lombardo, M. Massi.*

E-3. **Differential and scanning-mode external PIXE for the analysis of a painting by Antonello da Messina.** *N. Grassi, P. Bonanni, P.A. Mandò, A. Migliori, M. Massi.*

Session F. Arts and archaeology

Chairperson: J.L. Ruvalcaba-Sil

F-1. Invited Talk. **PIXE in the study of archaeological and historical glass.** *T. Calligaro*

F-2. **Concentration profiles in pigment layers.** *Ž. Šmit, P. Pelicon, M. Uršič, T. Trček-Pečak, B. Šeme, I. Langus, and K. Kavkler.*

F-3. **Characterization of white pigments and paint layers by simultaneous PIXE and RBS.** *L. Beck, P. C. Gutiérrez, J. Salomon, Ph. Walter, M. Menu.*

F-4. **PIXE and PIGE analysis of 18th century ceramics.** *M. Kos and Ž. Šmit.*

Session G. Complementary analytical techniques & Arts and archaeology

Chairperson: F. Lucarelli

G-1. Invited Talk**. Benefits of combined PIXE and AMS with new accelerators.** *G. Demortier and L. Calcagnile*

G-2. **Early Photographic Chemistry Investigated using Ion Beam Analysis and SIMS.** *D. McPhail, R. Chater, G.W. Grime, C. Jeynes, M.-L. Abel and N. Bell.*

G-3. **Discovery and characterization of an unknown blue-green Maya pigment: Veszelyite.** *R. Garcia Moreno, F. Mathis, and D. Strivay.*

G-4. **The contribution of the LNS portable PIXE system for the examination of gold preparations in the miniatures of the 492 code (Pontificale) preserved at the Museo Diocesano in Salerno.** *L. Pappalardo, M. Bicchieri, M. Nardone, G. Pappalardo, F.P. Romano, P.A. Russo, A. Sodo.*

Session H. Environmental sciences

Chairperson: M.A. Respaldiza

H-1. Invited Talk. **Aerosol characterization in the Mexico City Metropolitan Area by PIXE/PESA and application in analysis of the organic component.** *K.S. Johnson, V. Shutthanandan, Y. Xie, A. Laskin, B. de Foy, L.T. Molina, K. Dzepina, J.L. Jimenez, D. Salcedo, and M.J. Molina.*

H-2. **Application of PIXE technique for identification of occupational exposure to Tantalum.** *K.C. Dália, R. Jean, C.M. Lima, L. Barros, C.V. Santos Nascimento, G.E. Medeiros, L. Carneiro, K. Dias da Cunha.*

H-3. **PIXE and aerosols in the 21st Century: Continuous rules!** *T. A. Cahill.*

Session I. Environmental sciences

Chairperson: W. Przybylowicz

I-1. **Detection of atmospheric aerosol sources at São Paulo City by PIXE analysis.** *A.A. F. S. Kerr, T. G. Veríssimo, S. Gioia, M. Babinski.*

I-2. **Chemical composition and mass closure for PM2.5 and PM10 aerosols at K-puszta, Hungary, in summer 2006.** *W. Maenhaut, N. Raes, X. Chi, J. Cafmeyer and W. Wang*

I-3. **Analysis of hot particles from Palomares (Spain) using proton and He μ–PIXE.** *J. García López, M.C. Jiménez-Ramos, M. López Pérez, M. García-León and R. García-Tenorio.*

I-4. **Biomonitoring of airborne trace element in Mexico City using tree leaves.** *C. Solís, K. Saitoh and H. Zolezzi-Ruiz.*

I-5. **A possible application of the new Curium based XPIXE-α system to the monitoring of the atmospheric particulate matter.** *G. Pappalardo, De Sanoit, Marchetta, L. Pappalardo, F. P. Romano, F. Rizzo.*

Session J. Advances in experimental devices

Chairperson: K. Malmqvist

J-1. Invited Talk. **Cryogenic high resolution X-ray detectors.** *J. Hoehne.*

J-2. **External beam PIXE/PIGE measurements on aerosol samples at proton energies from 2 to 5 MeV.** *F. Lucarelli, A. Caciolli, G. Calzolai, M. Chiari and S. Nava.*

Session K. Materials science & Advances in experimental devices

Chairperson: L. Beck

K-1. Invited Talk. **PIXE: Elemental concentration and beyond.** *J. F. Dias.*

K-2. **Automated PIXE-RBS depth profiling using multiple PIXE spectra and simulated annealing.** *C. Jeynes and G.W.Grime.*

K-3**. Particulate screening using micro-PIXE and multivariate statistical analysis.** *B.L. Doyle, J.C. Banks, P.G. Kotula and A.J. Antolak.*

Session L. Advances in experimental devices

Chairperson: P. A. Mandò

L-1. Invited Talk. **3D Micro PIXE – a new technique for depth resolved elemental analysis.** *A.G. Karydas, D. Sokaras, C. Zarkadas, N. Grlj, P. Pelicon, M. Žitnik, R. Schütz, W. Malzer and B. Kanngießer.*

L-2. **A model for quantitative micro–PIXE analysis in confocal geometry.** *D. Sokaras, A.G. Karydas, W. Malzer and B. Kanngießer*

L-3. **Characterisation of the urban and suburban aerosol in the City of Prague.** *V.Havránek, V.Voseček, J.Schwarz, J.Hovorka.*

L-4. **Crocker Nuclear Laboratory and PIXE. A Historical Perspective.** *R.G. Flocchini.*

POSTER SESSION I

Basic physical principles

PI-1. **L X-ray production cross-section ratios for protons.** *B.N. Jones and J.L. Campbell.*

PI-2. **Empirical approximation for L_α production cross-sections**. *Ž. Šmit and A. Tancek.*

PI-3. **Mo L X-rays relative yield ion energy dependence.** *P. C. Chaves, M. Kavčič, M.A. Reis.*

PI-4. **Measurement of K–L radiative vacancy transfer probabilities in rare earth elements bombarded with 3 MeV-4 MeV protons.** *J. Reyes-Herrera and J. Miranda.*

PI-5. **K-shell vacancy production by impact of projectiles with a previous orbital vacancy.** *C. M. Romo-Kröger.*

PI-6. **Comparison of Gd-K and L X-rays RYIED and proton-NMRD.** *M.A. Reis, P.C. Chaves, A. Taborda, A. Carvalho.*

PI-7. **GUPIXWIN - a new software package for PIXE analysis.** *N. Boyd, A.Weatherstone, J.L. Campbell, J.A. Maxwell and M. Vormwald.*

Advances in experimental devices

PI-8. **A new target chamber for simultaneous Ion Beam Analysis at the University of Chile.** *P. A. Miranda, J. Wachter, and J. R. Morales.*

PI-9. **Some specific features of the Budapest-Hamburg proton microprobe.** *A. Kocsonya, P. Kostka, I. Kovács, Z. Szőkefalvi-Nagy, A. Krüger, and M. Niecke*

PI-10. **New Tandetron Laboratory at NPI.** *V.Havránek, V.Hnatowicz, A.Macková, V.Peřina, J.Novotný, V.Voseček, J.Bočan.*

PI-11. **Development of 3D micron-CT using PIXE.** *Y. Kawamura, K. Ishii, H. Yamazaki, S. Matsuyama, Y. Kikuchi, T. Yamaguchi, Y. Watanabe, R. Oyama, G. Momose and A. Ishizaki.*

PI-12. **Development of an ion microprobe setup for complex elemental analysis of individual microparticles.** *Zs. Kertész, A. Simon, Z. Szikszai, E. Dobos, G. Á. Szíki, I. Uzonyi.*

Biology and biomedical sciences

PI-13**. Elemental mapping of plants using submilli-PIXE camera.** *H. Yamazaki, K. Ishii, S. Matsuyama, Y. Kikuchi, Y. Takahashi, Y. Kawamura, R. Watanabe, K. Tashiro and C. Inoue.*

PI-14. **PIXE and PESA analysis of metalloprotein stoichiometry.** *G. F. Peaslee, P.A. DeYoung, L. A. Ellsworth, L. M. Kiessel, and J.D. Warner.*

PI-15. **Improved radiosensitive liquid core microcapsules for the targeting of chemotherapeutic agents.** *S. Haradat, S. Ehara, K. Ishii , H. Yamazaki, S. Matsuyama, T. Sato, S. Oikawa, T. Kamiya, K. Arakawa, K. Sera, J. Ito.*

PI-16. **Measurements of Sr/Ca in bones to evaluate differences in temperature.** *P. R. Santos, N. Added, J. H. Aburaya and M. A. Rizzutto.*

PI-17. **Simultaneous PIGE and PIXE analysis of dental composites using a single detector.** *E. A. Preoteasa, D. Gurban, A. Scafes, M. Gugiu, E. Preoteasa.*

PI-18. **Measurement of heavy metals in canned foods.** *L. R. M. da Silva, C. E. I. Dos Santos, L. Boufleur, R. Debastiani, M. L. Yoneama and J. F. Dias*

PI-19. **PIXE analysis of some medicinal plants usually extracted and drunk as tea, beverage, or used as spice or flavor in Nigeria.** *S.O. Olabanji, S.K. Adesina, D. Ceccato, M.C. Buoso, and G. Moschini.*

PI-20. **Elemental mapping of a post oak leaf using a proton microprobe.** *F.U. Naab, B.C. Boling, F.D. McDaniel, J.L. Duggan and D. Smith.*

Environmental sciences

PI-21. **Microbeam analysis of yellow sand dust particles.** *S. Matsuyama, K. Ishii, H. Yamazaki, Y. Kikuchi, Y. Kawamura, R. Oyama, T. Yamamoto, A. Ishizaki and M.Genki.*

PI-22. **The use of biomonitors and PIXE analysis in the study of air pollution in Mexico City.** *L. Cervantes, O.Avila, J.L. Ruvalcaba, J. Miranda, R. Muñoz.*

PI-23. **PIXE and μ-PIXE analysis of biological records in environmental studies.** *L.Calcagnile, K.Butalag, G. Quarta, L. Maruccio.*

PI-24. **Effects on the elemental concentration in growth tree ring due to Popocatepetl volcano exhalations.** *A. R. Cruz-Muñoz, L. Rodríguez Fernández, G. Calva-Vázquez, and J. L. Ruvalcaba-Sil.*

PI-25. **Relationship between soil composition and the distribution of three *Manfreda* (*Agavaceae*) in Mexico.** *N. Martínez-Nava, R. Ríos-Gómez, E. Solano-Camacho, M. Ayala, L. Rodríguez Fernández, J. Reyes-Herrera and L. Caballero-Pagaza.*

PI-26. **Uncertainty evaluation in quantities obtained from PIXE elemental analysis of atmospheric aerosols.** *A. Espinosa, J. Miranda, and J.C. Pineda.*

PI-27. **Aerosols from inside an Alaskan forest fire via PIXE and PESA.** *C. F. Cahill, Th. A. Cahill, and K. D. Perry.*

PI-28. **Atmospheric levels and elemental composition of fine and coarse aerosols during wet and dry season campaigns at two sites in Tanzania.** *W. Maenhaut, N. Raes, and S. Mkoma.*

PI-29. **Representation and variability of the elemental mass size distributions of Antartic coastal eerosol at Baia Terra Nova (Ross Sea).** *P. Mittner, D. Ceccato, V. Trovò, F. Chiminello.*

PI-30. **PIXE-PIGE combined set-up applied to geochemical characterisation of ice dust and continental sediments.** *F. Marino, G. Calzolai, S. Nava, M. Chiari, F. Lucarelli, V. Maggi, E. Castellano, F. Rugi and R. Udisti.*

PI-31. **Comparison of particulate matter morphology and composition of indoor and outdoor aerosols. Use of the analytical techniques EDS - SEM and PIXE.** *G.I.*

Alcaraz Bañuelos, E. Herrera Peraza, C. Solis, M. Delgado, A. Campos Trujillo, J. Carrillo Flores, E. Ramírez Espinosa.

PI-32. **Intercomparison of aeolian dust elemental concentrations via PIXE and ICP-AES.** *L. Rojo, T. Gill, and M. Barnes.*

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PI-33. **Identification of Saharian sand storms by PIXE from 1995 to 2006.** *M.A. Reis, G. Dias, A. Quaresma, P.C. Chaves.*

PI-34. **Composition of mineral aerosols generated in the Salt Basin of Far West Texas (USA) using PIXE and complementary techniques.** *A. E. Perez and Th. E. Gill.*

PI-35. **Trace metal distribution in humic substances of wastewater irrigated soils of central Mexico**. *I. E. Reyes S., C. Solís, N. E. García C., K. Isaac-Olivé, C. Romo.*

PI-36. **The Aznalcollar disaster: An in-depth PIXE study of the pirite mine spill of 1998.** *H. Calvo del Castillo, J.L. Ruvalcaba, M.A. Álvarez, T. Calderón.*

PI-37. **Space weathering of extraterrestrial silicates simulated by nanosecond pulse UV excimer laser and PIXE checking of chemical modifications.** *K. Butalag, L. Maruccio, G. Quarta and L. Calcagnile.*

PI-38. **Matrix composition contribution to elemental concentrations measured by PIXE.** *J.J. Ramírez T., J. López M., P. Villaseñor S., J.A. Aspiazu F., E. Ordóñez R., A. Gutiérrez, A.C. Ruiz F.*

Materials science

PI-39**. Traces elements present in Ecomaterial by PIXE.** *G. C.Diaz Trujillo, Ma. P. Haro, I. Cordova, Ma. E. Villafuerte, J. Miranda.*

PI-40. **PIXE depth profile Ge in Si-Ge film using algorithm of maximum likelihood.** *V.Levenets, A.Omelnik, A.Shchur, B.Shirokov, I.Kovalchuk.*

Arts and archaeology

PI-41. **PIXE analysis of artefacts from radiocarbon dated archaeological contexts.** *G. Quarta, L.Calcagnile, K.Butalag, L. Maruccio and M.D'Elia.*

PI-42. **Analysis of 19th century Mexican postage stamps by PIXE.** *T. E. Gill.*

PI-43. ***In situ* PIXE analysis of pigments used in a Leonardesque painting.** *N. Eastaugh, I. Gomez-Morilla, K.J. Kirkby, G.W. Grime.*

PI-44. **External beam analysis of Roman glasses,** *P.A. Rodrigues, L.C. Alves, G. Encarnação, R.C. da Silva.*

PI-45. **Scanning-PIXE analysis of ancient embroideries.** *A. Migliori, N. Grassi, P.A. Mandò, L. Giuntini and M. Massi*

PI-46. **Provenance of Belgian Merovingian garnets By PIXE on IPNAS Cyclotron.** *F. Mathis, O. Vrielynck, K. Laclavetine, G. Chêne, D. Strivay.*

PI-47. **Non destructive study of gilded tumbaga artifacts from the Chichén-Itza cenote.** *J. Contreras, J.L. Ruvalcaba Sil, J. Arenas Alatorre.*

PI-48. **PIXE and Ionoluminiscence for Mesoamerican jadeite characterization.** *J.L. Ruvalcaba Sil, L. Manzanilla, E. Melgar, R. Lozano Santa Cruz.*

PI-49. **Characterization of archaeological obsidians from Lagartero, Chiapas, Mexico by PIXE.** *D. Tenorio, S. Rivero, M. Jiménez-Reyes, T. Calligaro, F. Monroy-Guzmán, and L. C. Longoria.*

PI-50. **PIXE analysis of obsidians from Teotihuacan.** *J. Gazzola, M. Sánchez del Río, C. Solís and T. Calligaro.*

POSTER SESSION II

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PII-2. **New high energy and high resolution Lisbon PIXE set-up.** *P.C. Chaves, M.A. Reis, E. Alves.*

PII-3. **Status report of Sasaki Taro memorial PIXE Center.** *A. Terakawa, T. Sasaki, K. Ishii, U. Kawamura, K. Sera, H. Sasaki.*

PII-4. **External beam PIXE setup in Aarhus, Denmark.** *F. Folkmann, M. B. Jensen, M. H. Jørgensen and D. S. Otykier*

PII-5. **Radiographic technique for densitometric studies using heavy ion microbeams.** *J. Muscio, H. Somacal, A.A. Burlon, M. E. Debray, A. J. Kreiner, J. M. Kesque, D. M. Minsky and A.A. Valda.*

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PII-6. **Phase stability study of Y and Gd stabilized zirconia by luminescence methods.** *R.E. Noria, G. Reyes, N.R. Rebollo, F. González, J.L. Ruvalcaba-Sil, J. Miranda, and Ma. E. Villafuerte***.**

PII-7. **Characterization of natural and syntetic zeolites using ion beam analysis techniques.** *E. Andrade, J.M. Aceves, R. Miranda, C. Solis, J. Cruz, M.F. Rocha and E.P. Zavala.*

PII-8. **Cleaning wastewater from ammonium with the mineral vermiculite –Using PIGE for nitrogen monitoring.** *J-O. Lill, J. Rajander, , L. Harju, K-E. Saarela, A. Lindroos and S-J. Heselius.*

Biology and biomedical sciences

PII-9. **Tin (Sn) metal transport and its influence on essential elements in the yeast *Saccharomyces cerevisiae.*** *C.M. Viau, M.-L. Yoneama, J.F. Dias, C. Pungartnik, M. Brendel and J.A.P.Henriques.*

PII-10. **An investigation of metal ions in the exoskeletons of wasps using nuclear microscopy.** *D. L. J. Quicke, I. Gomez-Morilla, K.J. Kirkby and G.W. Grime.*

PII-11. **Application of micro-PIXE and dynamic analysis for the characterization of human hard tissues.** *M.E.M Eisa, C.A. Pineda-Vargas, U.M.E. Chikte, A.L. Rodgers, S. Naidoo, J.L. Conradie.*

PII-12. **PIXE and PIGE identification of a dental composite from a dental filling and assessment of elemental changes associated to its oral use.** *E.A. Preoteasa, E. Preoteasa, A. Scafes, D. Gurban.*

PII-13. **An elemental analysis of Periphyton: A natural source of phosphorus in the wetlands of the Mayan region of Quintana Roo, Mexico.** *S. Palacios-Mayorga, A. López-Suárez, L. Huerta, and A. Gómez-Pompa.*

PII-14. **PIXE analysis of some Nigerian pharmacological plants.** *S.O. Olabanji, O.R. Omobuwajo, D. Ceccato, M.C. Buoso, and G. Moschini.*

PII-15. **Characterization of wines from Rio Grande do Sul, Brazil.** *C. E. I. dos Santos, L. M. da Silva, L. A. Boufleur, R. Debastiani, M. L. Yonema and J. F. Dias*

Environmental sciences

PII-16. **Micro-PIXE study of heavy metal uptake and transport of aquatic plant species.** *Zs. Kertész, I. Kocsár, Z. Szikszai, E. Dobos, G. Á. Szíki and Gy. Lakatos.*

PII-17. **Trace metals in the sea grass *Thalassia Testudinum* from Mexican Caribbean coasts.** *C. Solís, K Isaac-Olivé), A. Martínez, E. Lavoisier, Z. Ruiz), LM Rivera.*

PII-18. **Application of the PIXE technique to the study of marine coastal environments using fishes as bioindicators.** *W. S. Fernandez, J.F. Dias, L.A. Boufleur, C. E. I. dos Santos, L.M. da Silva, R.Debastiani and J.F. Dias.*

PII-19. **Zinc profiles in archaeological and modern teeth.** *A.-M. M. Williams and R. Siegele.*

PII-20. **The role of PIXE within PATOS project, the first extensive field campaign for the aerosol characterisation in Tuscany (Italy).** *G. Calzolai, M. Chiari, F. Lucarelli, S. Nava, L. Paperetti and R. Udisti.*

PII-21. **Aerosol source apportionment by Positive Matrix Factorisation applied to daily and hourly concentration datasets obtained by PIXE.** *S. Becagli, G. Calzolai, M. Chiari, F. Lucarelli, A. Mannini, T. Martellini, S. Nava, L. Paperetti, R. Udisti and E. Yubero.*

PII-22. **High airborne Chlorine in micro or sub-micrometer particles.** *M.A. Reis, G. Dias, A. Quaresma, P.C. Chaves, N. Franco, N. P. Barradas, L.C. Alves.*

PII-23. **Seasonal variability in atmospheric aerosol levels and elemental composition during 2006 at Uccle, Belgium.** *W. Maenhaut, N. Raes, H. De Backer, and A. Cheymol.*

PII-24. **Detailed aerosol and elemental mass size distributions during winter and summer campaigns in Ghent, Belgium.** *N. Raes and W. Maenhaut.*

PII-25. **Comparison of Debrecen fine fraction aerosol data with others collected in some European collaboration.** *E. Dobos, I. Borbély-Kiss, Zs. Kertész, Gy. Szabó and E. Koltay***.**

PII-26. **Time resolved elemetal component study of urban aerosol in Debrecen, Hungary.** *Zs. Kertész, E. Dobos, B. Fenyős, R. Kéki, and I. Borbély-Kiss*

PII-27. **Elementary composition of particles $PM_{2.5}$ present urban areas of Baja California, Mexico.** *M.C.Castañón Bautista, G.C.Diaz Trujillo, F. Wakida, J. Miranda.*

PII-28. **PIXE technique used fro characterization of human exposure to mineral sand dust particles.** K Dias da Cunha, C.V. Barros Leite.

PII-29. **Analysis by PIXE of underground water from Ixtacxochitla, Puebla.** *F.González, E. Romero, J. Vargas, C. Solís, and J. Miranda.*

PII-30. **Heavy metal, radionuclides, ben/z/ahyrene accumulation in soil of inhabited settlement nearby NSC KIPT.** *V. Levenets, V.Azhazha, A.Masilov, A.Shchur, A.Omelnik, V.Diorditsa, I.Gnedaya.*

PII-31. **Development of sample preparation method for engine lubricating oil analysis using in-air PIXE.** *K. Saitoh, T. Ishikawa, H. Iso, S. Hasegawa, A. Fushimi, S. Kobayashi, K. Tanabe, T. Konishi and H. Imaseki.*

Earth sciences

PII-32. **Particle size/composition relationships of wind-eroding sediments, Owens Lake, California, USA.** *L. Rojo, T.E. Gill and D. Gillette.*

PII-33. **PIXE Based Geochemical Characterization of the Pluvial Lake Palomas – Samalayuca Dunes Corridor System, Chihuahua, México.** *M. Domínguez-Acosta, and T.E. Gill.*

PII-34. **PIXE Based Geochemical Characterization of the Pluvial Lake Palomas System, Chihuahua, México.** *M. Domínguez-Acosta, and T.E. Gill.*

Arts and archaeology

PII-35. **Determination of elemental distributions in cherts using μ-PIXE.** *S. Matteson, F.U. Naab, J.L. Duggan and F.D. McDaniel.*

PII-36. **PIXE and the anthropic circulation of obsidian during the Neolithic of central and western Mediterranean.** *F.-X. Le Bourdonnec, G. Poupeau, S. Dubernet, P. Moretto, T. Calligaro and M. Compin.*

PII-37. **Provenance Studies by PIXE of Obsidians from Laguna de los Cerros, Veracruz, Mexico.** *F. Ramírez, J.L. Ruvalcaba Sil, A. Cyphers.*

PII-38. **Long distance transport of Neolithic variscite ornaments along the European Atlantic arc demonstrated by PIXE analysis.** *G. Querré, F. Herbault and T. Calligaro.*

PII-39. **TTPIXE analysis of roman coins from Ilipa (II-I B.C.).** *B. Gómez-Tubío, M.A. Respaldiza, F. Chavez, I. Ortega-Feliu and M.A. Ontalba-Salamanca.*

PII-40. **Micro-SR-XRF and micro-PIXE studies for archaeological gold identification – The case of Carpathian (Transylvanian) gold.** *B. Constantinescu, R. Bugoi, M. Radtke, T. Calligaro, J. Salomon, L. Pichon, S. Röhrs and D. Ceccato.*

PII-41. **PIXE analysis of a 12th century stained-glass window.** *M.-P. Etcheverry, T. Calligaro, I. Baudoin-Louw, F. Licenziati S. Djanarthany, B. Magassouba, P. Trocellier and I. Pallot-Frossard.*

PII-42. **Non destructive analysis of daguerrotypes by simultaneous PIXE-RBS.** *A. de la Torre Saucedo, J.L. Ruvalcaba Sil.*

PII-43. **The Grolier Codex: A PIXE & RBS study of the possible Maya document,** *H. Calvo del Castillo, J.L. Ruvalcaba, T. Calderón, M. Van der Meeren and L. Sotelo*

PII-44. **Study of damage induced by ion beam in white pigments.** *P. C. Gutiérrez, L. Beck*

PII-45. **PIXE analysis of trace elements in middle age human and animal bones.** *L. Maruccio, G. Quarta, K.Butalag, V. Gaballo, P.Arthur and L.Calcagnile*

PII-46. **Proton Beam Characterization of Bone Remains from the Middle Mesoamerican Formative.** *L . Couoh, J.L. Ruvalcaba Sil.*

ORAL SESSIONS

A-1. Invited Talk — Basic physical principles

Ionization cross sections for high-energy PIXE

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Theories for K-shell ionization in the high-energy (10-100 MeV) range of PIXE [1], with an extension to GeV-protons [2], are reviewed to untangle consequences of various relativistic treatments of protons and the relativistic nature of the K-shell electron as well as its polarization by these high-energy protons. An alternative scaling to the parameterization implemented recently [3] in the GUPIX software [4] will be examined. The predictions of these theories and the adequacy of proposed fits for K-shell ionization cross sections will be analyzed for their departures from the nonrelativistic PWBA evaluated with the exact limits for momentum transfer [5], and scrutinized against the data.

1. A. Denker et al., *Nucl. Instrum. Meth. B* 2002; 189: 315; 2004; 213:677; *X-Ray Spectrom.* 2005; 34:376.
2. R. Anholt et al. *Phys. Rev. A* 1976; 14:2103; I.B. Vodopyanov et al. *J. Phys. B* 1996; 29: 2534.
3. A. Denker, J. Optiz-Coutureau, J.L. Campbell, J.A. Maxwell, T. Hopman, *Nucl. Instrum. Meth. B* 2004; 219-220:130.
4. J.A. Maxwell, W.J. Teasdale, J.L. Campbell, *Nucl. Instrum. Meth B* 1995; 97: 407.
5. Z. Liu, S. Cipolla, *Comp. Phys. Commun.* 1996; 97: 315.

A-2 Basic physical principles

PIXE study of the influence of the Coulomb explosion on the molecular stopping power of C_2 in Si <100> direction

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The study of the molecular stopping power is more complex than the one concerned with single atoms. In this case non trivial effects come into play. Among them it should be mentioned the so called Coulomb explosion. When the molecule penetrates in the material, it loose their electrons, and consequently an electrostatic repulsion force acts between the ions and the particles move away from each other as they travel through the medium. The present contribution reports on a quantitative determination of the Coulomb explosion of C2+ molecules channeled through the Si <100> direction by using the Si K-x-ray production. With this aim we have used the 3 MV tandem accelerator of the Ion Implantation Laboratory which provided C+ and C2+ beams at 1.2 MeV/amu. The Si <100> crystal was mounted on a three axis goniometer located in a chamber that was in a vacuum better than 10-7 mbar. The Si <100> alignment was performed using a 1 MeV He+ beam being the backscattering particles detected by a Si surface detector. The 1.74 keV Kα x rays emitted from the target were detected by a Si(Li) detector with an energy resolution of about 160 eV at 5.9 keV. Measurements of close encounter events giving raise to x-ray emission under channeling conditions provide a sensitive method to investigate the redistribution of the ion flux inside the channel. Therefore we have performed angular scans around the Si <100> axis using C+ ions and C2+ molecules. The results show that at small polar angles the x-ray yield was always larger for the C2+ molecules in comparison with the C+ ions. From this difference we were able to extract the energy of the Coulomb explosion of the C2+ molecule which is 13 +- 4 eV at the studied energy. This experimental result is in fairly good agreement with theoretical calculations and numerical simulations.

A-3 Basic physical principles

Influence of chemical enviromnent on the analysis of PIXE/XRF spectra of samples containing various compounds of 3d elements

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It is well known that the K_β/K_α intensity ratios of elements in the $22 \leq Z \leq 32$ region (3d elements) depend on the chemical environment. The differences of up to 10% were reported for K_β/K_α intensity ratios between various compounds of these elements. Experimental data show additional dependence of K_β/K_α intensity ratios on the excitation mode. However, it is the common practice in the analysis of PIXE or XRF spectra to neglect this dependence of K_β/K_α intensity ratios of 3d elements on the chemical environment. Instead, the measured K_α and K_β peaks in spectra are fitted with the help of the existing theoretical or experimental data-bases of determined intensity ratios for single elements, with corrections for target self-attenuation, possible attenuation of filters and differences in detector efficiency. In some circumstances this usual practice of spectral analysis could lead to analytical mistakes like for example reporting of non-existing elements (false hits). In this work we prepared a number of samples in the form of thick pellets by mixing various compounds of 3d elements. 2 MeV protons (PIXE) and photons (XRF) have been used to excite corresponding spectra. The following compounds have been used to prepare samples: TiO, Ti_2O_3, VO, V_2O_5, Cr_2O_3, $K_2Cr_2O_7$, MnO_2, $KMnO_4$, Fe_2O_3, and Co_3O_4. Related spectra have been analysed with the main goal to estimate the possible influence of the chemical environment on reporting the false hits in spectra. The results of the analysis have been discussed and presented.

B-1. Invited Talk — Basic physical principles

Theory of atomic bremsstrahlung based on Binary Encounter Approximation

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Continuous X rays produced in light-ion atom collisions producing continuous backgrounds and therefore determining the detection limit of PIXE, have been experimentally and theoretically studied 1]. It is shown that the experimental results over the wide range of projectile-ion energy from 0.5 MeV to 40 MeV are explained by four sources of radiative processes: nuclear bremsstrahlung (NB), atomic bremsstrahlung (AB), secondary-electron bremsstrahlung (SEB), and quasi-free electron bremsstrahlung (QFEB). The production cross section of AB was calculated based on the PWBA theory and reproduced well the experimental continuous background produced in an aluminum target [2]. However, in the case of gold target, there is a large difference between the theory and experiment. This can be improved by taking account of a screening effect [3], but it is not complete. We introduce a calculation method of AB cross section based on Binary Encounter Approximation [4].

1. K.Ishii and S.Morita, *Int. J. PIXE*, 1 (1990) 1.
2. K. Ishii and S.Morita, *Phys. Rev.* A30 (1984) 2278.
3. K.Ishii, H.Yamazaki, S.Matsuyama, W. Galster, T.Satoh, and M.Budnar, *X-ray Spectrometry*, 34 (2005) 363.

B-2 Advances in experimental devices

PIXE-PAGE analysis by scanning proton microprobe

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Metal content of metalloproteins can be located and even quantified by the so-called PIXE-PAGE method. In this technique the proteins are separated by thin layer electrophoresis (by polyacrylamide gel electrophoresis (PAGE) in most cases) and the properly dried gel sections are directly analysed by PIXE using "band-shaped" proton milli-beam. Adaptation of the method for the use of micro-beam is promising in several points:

i. more detailed elemental mapping of the tiny bands can be possible,
ii. the scanning allows two-dimensional analysis,
iii. the fast, continuous scanning will certainly reduce the thermal deterioration of the gel allowing to use higher beam current, and
iv. the disturbing artifacts caused by possible dust-like impurities can be easily filtered out in the data evaluation process

As a first step a series of measurements were performed to determine the elemental detection limits in order to estimate the minimum amount of proteins supplied for electrophoresis. For several technical reasons this amount is limited in a PAGE run restraining the sensitivity of the PIXE-PAGE method. Taking full advantage of the unique X-ray detector arrangement in our PIXE chamber - a large area (80 mm^2 Si(Li) detector of 220 mSr can be installed directly behind the gel sample – the detection efficiency bacame considerably higher in comparison to that of obtained in the standard milli-beam setup. The background, on the other hand, was reduced by a properly chosen combination of C, Al and Be foils between the thin sample and the detector both to stop the beam and to reduce the x-ray background emitted in the stopping process. In micro-PIXE-PAGE measurements on cytochrome *c* bands a detection limit of 0.7 ng was obtained for Fe. Preliminary results on real samples from maize (*Zea mays* L.) root plasma membranes are also presented.

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B-3 Advances in experimental devices

Silicon detector deadlayer thickness estimates using proton bremsstahlung from low atomic number targets.

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Heavy charged ions, with MeV energies, produce both characteristic and bremsstrahlung radiation in the 1-15 keV X-ray energy range for thin and thick targets. The characteristic X-ray peaks are used in Particle Induced X-ray Emission (PIXE) analysis methods. These PIXE techniques are very sensitive and to also be accurate they generally require X-ray semiconductor detector efficiencies to be exceedingly well determined. This requires an accurate knowledge for all absorbing material between the target and the detector crystal itself. It is difficult to obtain insitu individual thickness estimates for each of these absorbing materials which may include filters, beryllium windows, ice and crystal detector deadlayers.

Recent, work shows that the detected PIXE bremsstrahlung radiation has several components quasifree, secondary electron and atomic bremsstrahlung as well as Compton scattered gamma rays For MeV ion bombardment of materials lighter than aluminium and heavier than copper the characteristic X-rays peaks do not generally interfere with the bulk of the bremsstrahlung.. For 1-4 MeV proton bombardment this bremsstrahlung generally peaks in the 1-5 keV X-ray region and is ideally suited to determine absolute semiconductor detector efficiencies in this traditionally difficult but important energy region.

This paper describes a novel method for the estimation of silicon detector deadlayer thicknesses in the range 0-1 μm in the presence of all other non silicon absorbers which maybe several orders of magnitude thicker. The method uses the measured bremsstrahlung from 2-4 MeV protons on both thin and thick beryllium targets. It does not require an accurate knowledge of the bremsstrahlung production cross sections only that they are smooth and continuous with X-ray energy around the silicon K edge energies from 1.5-2 keV. Data for current Si(Li) detectors with deadlayers of 0.1μm will be presented in the presence of 75μm thick beryllium window absorbers.

B-4 Advances in experimental devices

Elemental concentrations in aerosol samples determined by Artificial Neural Networks from PIXE spectrum

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An application of artificial neural networks (ANN) has been performed to determine elemental concentrations in atmospheric aerosol samples. A feed-forward ANN was used in the learning phase, directly from concentration data obtained in real time. The network was trained with a back propagation algorithm in order to learn how to obtain the elemental concentrations in these samples. The inputs to the ANN analysis were experimental data obtained from the PIXE irradiations plus one from each element in the spectrum. The ANN output was the elemental concentration. A set of thirty seven PIXE spectra from aerosols collected in Santiago, Chile, provided the raw data which were used in the ANN application. Following a random selection, thirty spectra were used in the training phase, while the rest was used to test the ANN capability in the determination of unknown concentrations. Networks of similar topology were independently trained for each one of the five elements considered in this study, Al, Si, S, K, and Fe. These networks operated in parallel, thus allowing a simultaneous determination of the elemental concentrations. These results were in good agreement with those obtained by standard PIXE analysis. This ANN application to the study of atmospheric aerosols has proved to be reliable and easy to extend to a large number of samples of similar characteristics.

C-1. Invited Talk Biology and biomedical sciences

Application of a heavy-ion microprobe to the determination of microdistributions of a drug of potential interest for boron neutron capture therapy

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In recent years a heavy-ion high-energy microprobe has been installed at the Tandar Laboratory of the Argentine Atomic Energy Commission in Buenos Aires. High resolution X-ray spectroscopy following micro-PIXE (Particle Induced X-ray Emission with micrometer-sized beams), with a focused ^{16}O ion beam at 50 MeV incident energy, was used to study the microdistribution in tissue samples of small animals of a drug of potential interest for Boron Neutron Capture Therapy (BNCT). In addition a STIM Silicon surface barrier detector was installed at 0°, interchangeably with a normalization Faraday cup, to obtain high spatial resolution density maps by measuring directly the energy loss of the projectiles as they traverse the sample. Focusing was performed with a heavy-ion scanning high-precision magnetic quadrupole triplet Oxford microprobe. Microdistributions of the prospective BNCT-compound CuTCPH, a carborane-containing tetraphenylporphyrin with one Cu atom in its molecular structure, have been obtained in thin tissue sections of different organs of tumor-bearing and normal Syrian hamsters injected with the boron compound by employing the heavy ion microbeam. Samples from squamous cell carcinomas induced on the right Cheek Pouch of Syrian Hamsters (HCP), were cryo-sectioned with a cryo-microtome and freeze-dried before irradiation. Two-dimensional maps of elemental concentration were obtained by scanning the beam over the samples demonstrating the feasibility of the technique for the specific application. Very non-uniform Cu concentrations were found in all sections pointing to the need of improving the drug delivery strategy.

C-2 Biology and biomedical sciences

The characterisation of a contaminant-free support film for microPIXE analysis of biological samples.

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Unique identification of metals bound to macromolecules is a challenge in structural biology, and although mass spectrometry methods have progressed significantly, an unambiguous assignment is often problematic. MicroPIXE (particle induced X-ray emission) with 2-3MeV protons on liquid and crystalline proteins has been used successfully in both identifying atoms and measuring their stoichiometric ratio (calibrated using the sulphur peak and internal normalisation of the sulphur atoms in cysteines and methioniones) to an accuracy of ±20% on over 50 samples [1,2]. Liquid or crystal samples are currently mounted on 2μm Mylar ($C_{10}H_8O_4$) backing film during analysis. The Mylar was known to be contaminated with calcium and phosphorous: thus problems in determining elemental concentration arose if the biological sample contained either of these elements [3]. Calcium is often functionally important in biochemical processes and many proteins bind it. Phosphorous can also be used as an internal standard for samples containing DNA or RNA.

A 4μm thick Prolene film [4] (CH_2) routinely used for sample mounting in WDXRF(Wavelength Dispersive X-Ray Fluorescence) experiments appears to be free of the phosphorus and calcium contaminants that are present in the 2μm Mylar ($C_{10}H_8O_4$) backing film currently used in microPIXE. A further compelling advantage of Prolene is that it contains no oxygen, making RBS spectra interpretation much more straightforward. MicroPIXE analysis of the prolene showed that it contains no contaminants above the minimum detectable limit, and this film is thus allowing us to perform accurate measurements of the calcium and phosphorus content of biological samples. The advantages of Prolene will be illustrated using recent examples of protein analysis.

1. Garman, E. *Structure* (1999) 7, R291-R299.
2. Garman, E. and Grime, G. (2005) *Progress in Biophysics and Molecular Biology*. 89/2, 173-205.
3. Yates, D., Garman, E., Gemmell, N., Gomez-Morilla, I. and Grime, G. W. (2004). In *Proceedings of the 10th International Conference on Particle Induced X-ray Emission and its Analytical Applications*, ISBN 9616303627. Ed. Milos Budnar, Published by Institute Josef Stefan, Ljubljana, Slovenia.
4. Fluxana GmbH & Co. Bonhoeffeweg 1, D-47551, Bedburg Hau, Germany. *www.fluxana.com*

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C-3 Biology and biomedical sciences

Micro-PIXE studies of elemental distribution in mycorrhizal and nonmycorrhizal roots of *Ni*-hyperaccumulator *Berkheya coddii*

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Recently, the presence of mycorrhizal symbiosis was demonstrated in the Ni hyperaccumulating plant, *Berkheya coddii*. This South African plant is growing on ultramafic soils and seems to be an excellent model for phytoextraction and phytomining. The principal function of mycorrhiza is an enhanced supply of essential nutrients from the soil by extraradical mycelium. At the same time mycorrhizal colonization affects heavy metal transfer from the soil to the plants but this response varies depending on the fungal species and isolates.

The aim of the present study was to assess the influence of the different mycorhhizal fungi on the uptake of elements by *B. coddii*. The plants were cultivated under laboratory conditions on sterilized ultramafic soil and inoculated with native mycorrhizal fungi, *Glomus intraradices* from non-polluted soil or left noninoculated. The elemental distribution in *B. coddii* roots (thick matrix) and fungal hyphae (intermediate matrix) was investigated with a nuclear microprobe. Micro-PIXE and BS (Proton Backscattering) were simultaneously used. GeoPIXE II software package was used for quantitative elemental mapping complemented by evaluation of data extracted from arbitrarily selected micro-areas.

Elemental maps clearly showed different elemental distributions throughout the roots. The uptake of elements and their distribution strongly depended on the presence of mycorrhiza and different fungal strains.

C-4 Biology and biomedical sciences

Localisation of trace metals in hyper-accumulating plants using μ PIXE

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PIXE is a very sensitive technique that can fast and reliably measure a wide range of elements simultaneously with high sensitivity. Using a focused microbeam elemental distributions can be mapped with high spacial resolution. This is very useful for a wide variety of samples, especially for biological samples, where it provides information about the concentration of various elements in different types of tissue.

Over the past 5 years we have used this technique to study a wide variety of metal hyper accumulating plants, accumulating different metals from Manganese to Cadmium. Sample preparation is the single most important issue in order to achieve good and reliable results. One of the problems of the sample preparation is to dry the specimens for micro-PIXE without relocating the to metals or other elements. We will discuss different sample preparation techniques and their influence on micro-PIXE.

We will also show examples micro-PIXE maps of the elemental distributions of metals and other structural elements in various parts of plant tissue and how these maps can be used to understand the strategies the plants use to cope with hight concentrations of these normally toxic metals.

D-1. Invited Talk Biology and biomedical sciences

Applications of micro beam PIXE to investigations on neurodegeneration

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During the past two decades there have been increasing efforts to measure and quantify biological constituent elements (such as Fe, Zn, Cu, and Ca), the excessive accumulation of these elements, as well as incorporation of toxic minority elements into the human tissues. Distribution of contaminating elements and free radicals in the human tissues is also a matter of concern. In this talk, I introduce the results if some of the works performed in our laboratory since 1992.

The possibility of influence of metallic elements on neurodegeneration is a subject that needs further investigation and micro beam PIXE provides us with strong means in this field. Our studies extended from Alzheimer's and Parkinson's disease to ALS. Other techniques such as electron and synchrotron radiation were also systematically used in these studies. The techniques and problems associated with the preparation of samples for PIXE analyses will also be discussed in details.

D-2 Biology and biomedical sciences

Micro-PIXE analysis of bioconductive hydroxyapatite coatings on titanium alloy

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Bioconductive materials and in particular implants using Ti alloy (Ti_6Al_4V) coated with hydroxyapatite (HAp) have proved to be a suitable surgical procedure. However, experience has shown that these implants not always have the required reliability to guarantee their expected life-span of approximate 15 years. In this research, experimental Ti alloy-implants coated with HAp and incubated in a simulated body solution (r-SBF) under controlled physiological conditions were study by nuclear microscopy. Selected cross-sectional implants, containing both Ti alloy substrate and HAP coating, were analysed by nuclear microprobe (NMP) with protons of 1.5 and 3.0 MeV at the iThemba LABS facility. The software package GeoPIXE II was used to reconstruct spectra stored as files in the event-by-event mode. Major elements (Ti, Al, V, Ca and P) as well as trace elements (Si, K, Fe, Zn and Sr) were determined. The effect of longer incubation time was of particular interest. Results confirmed that secondary Ca-deficient defect hydroxyapatite precipitated from the simulated body solution onto the HAp coating surface after prolonged incubation. This newly formed layer is thought to be of vital importance for bonding of implants with living bone tissue.

D-3 Biology and biomedical sciences

The identification of historic biocide residues on herbarium material at the National Museum Wales

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The National Museum Wales (NMW) houses c. 250,000 higher plant specimens, with material dating back to the 18th century. Herbaria have been a major source of botanical research and reference for centuries and the collections have increased over time from donations and collecting.

Due to its organic content, botanical material is susceptible to insect and fungal attack. Even aged, dried material is a source of sugar and protein. Institutions and collectors have prevented such attacks through the application of pesticides. Chemicals containing compounds of arsenic, lead and mercury were common place, and have remained stable over time. Consequently, present-day handling of theses collections presents a potential health risk to staff and visitors through inhalation and skin absorption, particularly since the quantity and nature of the pesticide applied is unknown. Occasionally, the residues are visible, but research has shown that, herbarium sheets which appear untouched, have been previously treated, and contain high concentrations of toxic metal ions.

The use of a UV hand-held lamp has helped to identify sheets that have been treated, even though treatment is not visible to the naked eye. The UV causes areas to fluoresce on the herbarium mount sheet. These areas were analysed by atomic absorption spectroscopy (AAS) and particle induced x-ray emission (PIXE) and have been found to correlate with pesticide applications.

This research has provided data for the identification and quantification of the pesticides applied. The information has enabled safe standard procedures to be implemented to protect personnel, and has also provided a rapid, effective method of identifying contaminated samples within the collections and provided a means to prioritise which collections require immediate re-mounting. This has enabled the removal of a large amount of hazardous chemical from the herbarium environment, and allowed for safe disposal.

D-4 Biology and biomedical sciences

Nuclear microprobe studies of grasshopper feeding on nickel hyperaccumulating plants

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Grasshopper *Stenoscepa sp*. is one of the very interesting insects feeding on the South African nickel hyperaccumulating plants. Ingested high amount of Ni does not disturb the development of this insect. This particular ability stimulated search for symptoms of the adaptation to survive in such extreme conditions.

The elemental distribution in the insect body was investigated with a nuclear microprobe (micro-PIXE and BS). GeoPIXE II software was used for quantitative elemental mapping complemented by evaluation of data extracted from arbitrarily selected micro-areas.

Micro-PIXE analysis in *Stenoscepa* sp. tissues showed the highest Ni level in the gut and Malpighian tubules.

The activity of glutathione-dependent enzymes and glutathione (GSH) content in the IInd stage larvae tissues were measured. One of the ways to survive under permanent Ni intoxication conditions is an intensified GSH synthesis. GSH concentration in tissues of the grasshoppers was very high, about six times higher than in larvae of other Acrididae species from areas contaminated with heavy metals in Europe. Catalase activity was 5-10 times lower in comparison to other Orthoptera. Glutathione reductase (GR) activity was unexpectedly low (at detection limit level). It is likely that the studied grasshopper may use thioredoxine system for regeneration of the reduced form of GSH.

D-5 Biology and biomedical sciences

Daily changes of elemental concentration in a human body over 218 days obtained by quantitative analyses of beard samples

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We developed and reported a standard-free method for beard (including mustache and whiskers) samples last year. This method enables us to quantitatively analyze powdered beard samples of extremely small quantity taken with ordinary electric shaver. In order to investigate intake of essential elements and exposure to toxic elements, daily changes of elemental concentration in the body, which reflect daily ingestion of foods and water, give us essential information, and beard analyses are expected to give us valuable information. Samples were taken from the same person in the morning and at night over successive 218 days and 543 samples were analyzed in total. Moreover, concentration changes with passage of time in a day were also studied. As a result, both short-term and long-term changes have been observed and their correlation with the food intakes is investigated. For example, it is found that concentrations of sodium, potassium and chlorine show the same trend both in short- and long-term changes, which indicates that they mostly exist in the chemical forms of NaCl and KCl in a human body. Difference of elemental concentration between the beard samples collected in the morning and at night is also discussed. It is found that the standard-free method for beard samples is quite useful for investigating daily changes of elemental concentration in a body and it is expected to give us information about the pathways of human exposure to toxic elements.

E-1. Invited Talk Complementary analytical techniques

Luminescence of rare-earth doped zirconia: A phase stability study

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Recent developments of zirconium-based ceramic coatings, for thermal insulation applications, have led to the implementation of rare earths (REs) as a mean to improve their thermal capabilities. The introduction of REs as alternative dopants to zirconia (ZrO_2), has opened the possibility to benefit from their luminescent properties; an example is the recent application of selected REs as luminescent sensors embedded in the coating to monitor erosion at different depths or to measure *in-situ* temperatures during service, through their response to laser stimulation. The aim of this work is to extend the technological use of luminescent dopants as indicators of the phase evolution on RE-doped zirconia.

Thermal conductivity reductions achieved by RE additions to zirconia, enable potential operating temperature increments. However, a successful implementation of these emerging materials is conditioned to their ability to resist thermal phase degradation. The present work proposes the use of Eu and Tb as sensors to monitor and compare the phase de-stabilization through luminescence changes; it also intends to identify possible influences of these dopants on phase stabilization. The specimens for this work are powders prepared by reverse co-precipitation of precursor solutions. Phase evolution upon ageing at high temperature is monitored using traditional micro structural characterization techniques as well as Ion-luminescence and photoluminescence. Particle induced X-ray emission is also used as a complementary technique to identify impurities and to determine the relative amounts of the constituting elements. Ion-luminescence emerges as a promising alternative technique to characterize RE-doped zirconia compositions.

E-2 Advances in experimental devices

Micro-PIXE determination of Zr in rutile: an application to geothermometry of high-P rocks from the Western Alps (Italy)

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The Western Alps of Northern Italy mostly consist of lithotectonic units which re-crystallised and were metamorphosed at high depth in a subduction zone. During their exhumation to shallow crustal levels, however, the high pressure mineral assemblages of blueschist and eclogite facies were pervasively re-equilibrated under low pressure conditions, making it difficult to estimate the metamorphic peak P-T conditions.

Rutile [TiO_2] is a typical high-pressure mineral, occurring as relict phase in low-P re-equilibrated metamorphic rocks. Recent studies of minor element abundances in metamorphic minerals suggest that, in thermodynamic systems buffered with quartz and zircon, Zr incorporation in rutile is a temperature–dependent process that can be modelled quantitatively.

An application of the Zr-in-rutile geothermometer to blueschist and eclogite facies rocks of the Western Alps, pervasively re-equilibrated under low-P conditions, is presented in this contribution.

Rutile crystals occurring in metapelites were analysed at the external scanning proton microprobe facility, placed on a beam line of the new 3MV Tandetron accelerator at the LABEC laboratory of INFN in Florence. A 3 MeV proton microbeam with ~10 μm spatial resolution and beam current of 1-2 nA was used. The PIXE spectra and maps were processed by Gupix and Geopixe dedicated software packages.

Preliminary results of Zr-in-rutile thermometry on Alpine metamorphic rocks appear to be consistent with the metamorphic history and T conditions derived from phase relations and conventional geothermometry, indicating that determination of Zr abundances in rutile by the micro-PIXE technique is a precise tool to reconstruct metamorphic peak temperatures of the high-P event.

E-3 Arts and archaeology

Differential and scanning-mode external PIXE for the analysis of a painting by Antonello da Messina

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Extensive investigations have been performed at the LABEC Laboratory in Florence on the painting "Ritratto Trivulzio" by Antonello da Messina, one of the great Italian masters of XV Century. The work was carried out in collaboration with art historians and restorers of the Opificio delle Pietre Dure, with the aim of discovering materials and techniques used by Antonello, universally considered to be an innovator in Renaissance painting.

In spite of the well known difficulties in PIXE analysis of paintings, useful information on paint layer composition and stratigraphy of the analysed masterpiece has been obtained, exploiting the high versatility of our Tandetron facility. On the one hand, differential PIXE was performed with proton beams up to 5.5 MeV energy; on the other hand, the analytical capabilities of our external microbeam setup were exploited, obtaining elemental maps by scanning the beam over the sample.

Differential PIXE measurements were performed as single-spot analysis in different areas of the painting, using external proton beams (500 micron size, energies from 1 to 5.5 MeV, current intensities from 5 to 50 pA). The interpretation of these preliminary data pointed out the need of further analysis in selected areas, where some issues remained to be clarified. This was possible by using the external microbeam facility: elemental maps from areas of several mm2 were obtained, with a 3 MeV proton beam of about 30 micron size and 50 pA intensity.

The whole of the collected data allowed us to characterise the different areas in the painting: the hat and the incarnato; the dark background, where an estimate of the paint layer thickness was possible; the mantle of the portrayed gentleman: here, the use of different pigments in different layers, with an irregular surface distribution on a sub-millimetre scale, was acknowledged as the technique used to create colour shades.

F-1. Invited Talk — Arts and archaeology

PIXE in the study of archaeological and historical glass

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The PIXE method, especially when carried out with an external beam, is a remarkable tool for the scientific investigation of objects of cultural heritage. We present here two case studies addressing three important issues of archaeological and historical glass: de-formulation of ancient glass recipes, determination of provenance of raw materials and understanding weathering processes endangering these precious artefacts. It is also the occasion to specify the place of the PIXE technique among other analytical methods based on radiations for the study of this specific material. In the first example, PIXE was used to determine the provenance of a natural glass, obsidian, constituting the support of two paintings of the Spanish master Murillo (17^{th} century) on display in the Louvre museum. The comparison of the chemical fingerprints obtained by PIXE with an obsidian composition database established by INAA and XRF showed that the rectangular obsidian panels were imported from Mexico. These investigations also permit to highlight the complementarity of these analytical techniques. In the second example, PIXE was applied to a 12^{th}-century stained glass window from the Saint-Denis basilica (Paris) to determine the various medieval glass types (soda- and potash-lime) employed in this panel. In addition, by combining p-PIXE with α-PIXE, PIGE, p-RBS and α-RBS, alteration markers of the glass surface, useful in conservation science, were evidenced.

F-2 **Arts and archaeology**

Concentration profiles in pigment layers

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Differential PIXE measurements based on the variable proton impact energy were used to probe the depth distribution of metal-based pigments in paint layers. The method represents further improvement of the differential measurements in metals [1]; however, the present approach is more semi-quantitative as important information on chemical compound in the target has to be inferred: the pigment compounds and the embedding matrix. The measurements were performed at the Jožef Stefan Institute using proton beam in the air. Two types of targets were used: fresco fragments and oil paintings. Frescoes were selected for their known matrix (limestone) which can be monitored through its calcium X-rays. The concentration profiles in frescoes were determined using two different normalization procedures: a) the sum of all weight fractions of the compounds present was set to unity (similar to the analysis of metals [1]); b) the number of projectiles was measured, exploiting the argon signal from the air; in the latter case, the experimental geometry was carefully measured using standard targets. The de-convolution method relied on slicing the target into layers characterized by mean production depths; the matrix inversion was replaced by a min $\chi 2$ problem. From the practical point, the method was applied on the paintings of Slovenian impressionist from the beginning of the 20th century. It was shown that the pigments were excessively mixed with different white pigments (mostly lead), necessary to support thick plastic strokes of the pen.

[1] Ž. Šmit, M. Holc, *Nucl. Instr. Meth*. B219-220 (2004) 524-529.

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F-3 **Arts and archaeology**

Characterization of white pigments and paint layers by simultaneous PIXE and RBS

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PIXE is now routinely used for analyzing paint layers. Various set up have been developed to investigate the elemental composition of samples or wood/canvas paintings. However, quantitative PIXE analysis is sometimes difficult to interpret due to the layered structure, the presence of varnish and organic binder. Backscattering spectrometry (BS) can be a useful complementary method to overcome these limitations. By using 3 MeV protons for PIXE and BS simultaneously, it is possible to perform quantitative analysis including C and O for which the non-Rutherford cross sections are intense. Furthermore, by using the same conditions for PIXE and BS, the experiment time and the potential damage by the ion beam were reduced. The results obtained with the external beam of AGLAE on a large set of pure white pigments (lead white, basic lead sulphate, calcium sulphate, gypsum, calcite, zinc oxide, titanium oxide and lithopone) and on various mixing ratios of pigment and linseed oil will be shown. Simultaneous combination of PIXE and BS lead to a complete characterization of the paint layers: elemental composition, thickness, varnish layer thickness and proportion of the organic binder were determined.

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F-4 **Arts and archaeology**

PIXE and PIGE analysis of 18th century ceramics

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Among the ceramics production of the 18th century Europe, the creamware is a special phenomenon. It was developed in England (the most important producer was Josiah Wedgwood) as a replacement for highly expensive porcelain of the mid-18th century. The production spread all over Europe. Most of the new factories were imitating English products. In the vicinity of today's Slovenia, the most renowned factories were in Vienna and Graz (Austria), and in Trieste (Italy). In Ljubljana three creamware manufactures were formed at late 18th and early 19th century. Their products were highly elaborate. They were exported all over nearby countries (Germany, Austria, and Italy). The most important producers in Ljubljana were Sigismund Zois's ceramics factory and Brother Wasser's creamware factory. Both used their marks, but only a part of objects were in fact signed. As for forms and decoration, all the producers in Ljubljana, Graz, Vienna, and Trieste used very similar schemes, based on English patterns.

In the ceramics collection of the National Museum of Slovenia there are around 1000 creamware objects, around 200 unsigned. The curator's task is to establish whether they were produced in Ljubljana or maybe imported from other countries. Since art historical methods (grouping the objects on the basis of colour, pattern, and form) proved to be ineffective, we searched for another, more exact solution. In order to prove the results and establish the provenance database, we decided to determine composition of the ceramics by in-air PIXE. The material appeared rather pure, so detection of the elements heavier than iron was not possible. Discrimination and grouping was done according to the light elements in the clay, which were determined by proton-induced gamma analysis (PIGE). We also distinguished between the structure of glaze and the body itself. The method is useful for analyzing all kinds of glazed ceramics.

G-1. Invited Talk Complementary analytical techniques

Benefits of combined PIXE and AMS with new accelerators.

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PIXE and associated IBA techniques for the study of archaeological artefacts are now widely used by interdisciplinary teams. These non destructive surface techniques were generally performed with single ended particle accelerators in the late 70's when external beams became available in nuclear physics laboratories involved in applications to archaeological problems. New facilities, especially in Europe, are now available with tandem accelerators using negative incident ions and often equipped with microprobes in- and outside vacuum target ports. These negative ion sources allowing eliminating nitrogen ions from the ^{14}C beams are also used to implement the performances in dating of organic materials by comparison with the time consuming procedure involving the counting of low energy and low intensity β particles. The implementation concerns the lower quantity of necessary material to be consumed as well as the determination of older accessible ages.

Combination of the elemental non destructive analysis of artefacts by IBA multi-elemental techniques (but mostly PIXE) with the AMS measurement of the age of the organic material discovered in the close vicinity of these artefacts (mainly available for recent excavations) gives new tools to archaeologists to improve their diagnostic in the study of the composition of ancient objects and of their workmanship in ancient times by using the same experimental facility for IBA and AMS. For potteries and metallic samples IBA and AMS are to be applied on different samples excavated in the same environment but for organic archaeological samples IBA and AMS techniques could be sequentially used on the same material.

Results of these combined techniques on artefacts of various origins recently studied at CEDAD (CEntre of DAting and Diagnostic investigations) will be presented.

G-2 Arts and archaeology

Early Photographic Chemistry Investigated using Ion Beam Analysis and SIMS

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Photographs are an important part of our cultural heritage, and have been representing accurate historical images of people, places and events for nearly two hundred years. A number of important collections of photographs are housed in museums and archives around the world and it is vitally important to find the best conditions in which to store them, in order to preserve them for future generations.

The aim of our research is both to elucidate the chemical processes used in early photographs and to determine the mechanisms and rates of deterioration of these objects as a function of their environmental conditions, in order to arrive at the best conditions for their storage and restoration. We are characterising these complex multilayer structures using a wide range of analytical techniques, with an emphasis on those which can be used on complete photographs without sampling, or which require minimal sample size.

Microbeam PIXE and RBS are being used to determine the identity, concentration and depth profiles of metals used in light sensitive materials, while SIMS is used to investigate near-surface chemical changes caused by long term storage. The long term goal will be to use external beam IBA on unsampled photographs, but in initial experiments we have been studying small fragments of a photograph in vacuum. By comparing PIXE and RBS spectra recorded at normal incidence with elemental maps of cross sections we can gain confidence in interpreting the complex normal incidence spectra.

In this talk we will compare and contrast the information given by FIB SIMS, PIXE/RBS and TOF SIMS, applied to a sample kindly donated by the Getty Institute.

G-3 Arts and archaeology

Discovery and characterization of an unknown blue-green Maya pigment: Veszelyite

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Green mineral pigments have been used since Antiquity throughout the world. In pre-Columbian art, green, a colour directly related to life and fertility, has been employed to make ritual objects. Based on some micro-chemical stain tests, which frequently reveal the presence of copper, references cite malachite and chrysocolla, both very common in Mexico, as the main green mineral pigments mastered by pre-Columbian civilisations. Nevertheless, the application of complementary analytical techniques on Maya archaeological funerary artefacts has helped to document and precise different pigments' recipes that indicate the extended use of other green copper pigments.

Blue-green mosaic and polychrome masks and funerary offerings from the royal tombs of Calakmul, Mexico, were analysed by Scanning Electron Microscope equipped with Energy Dispersive X-ray spectroscopy (SEM-EDX), X-ray Diffraction (XRD), and Particle Induce X-Ray Emission (PIXE) in the Centre Européen d'Archéométrie of Liege University (CEA-ULg), and the Centre de Recherche et de Restauration des Musées de France (C2RMF). This led to the first identification of the use of veszelyite, a rare hydrated copper-zinc phosphate, as green pigment. This rare mineral might come from mines located in Puebla. Analyses of geological samples of veszelyite have been done to confirm the characterisation of this Maya pigment, which might help determine pre-Columbian trade routes of precious and luxury raw materials and objects in Mesoamerica, between Central Mexico and the Maya area, for the Classic period (A.D. 250-800).

PIXE MEXICO 2007

XI International Conference on PIXE and its Analytical Applications
Puebla, Mexico, May 25-29,2007

G-4 **Arts and archaeology**

The contribution of the LNS portable PIXE system for the examination of gold preparations in the miniatures of the 492 code (Pontificale) preserved at the Museo Diocesano in Salerno

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The 492 code of the Museo Diocesano of Salerno is dated back to a period from the end of XIII century and the incoming of the XIV one. At present time it is preserved at the Museo Diocesano in Salerno, after having been submitted to an important and innovative restoration and conservation treatment at the Istituto Centrale per la Patologia del Libro (ICPL) in Rome.

The miniatures are of excellent quality and the pigments (lead white, minium, lapis-lazuli, mosaic gold, yellow-ochre, earths, etc.) are generally in poor state of conservation. Gold is present as "foil": many layers of thin gold-leafs are applied on the *asisum* (the preparation for gold illuminations).

The manuscript has not been completed by the miniaturists; in some cases only the preliminary design is present, in other cases the preparation is not covered by the gold-foil. The above circumstances give the unique opportunity to investigate the different techniques used by the miniaturists and in particular the composition of the gold preparation and their comparison with the medieval recipes.

The gold preparations of the Salerno 492 code were examined by means of the LNS portable PIXE-alpha spectrometer. Complementary XRF and micro-Raman techniques were also used. Quantitative data enable to establish their composition. Also results on Mosaic Gold are reported and discussed.

H-1. Invited Talk — Environmental sciences

Aerosol characterization in the Mexico City Metropolitan Area by PIXE/PESA and application in analysis of the organic component

K.S. Johnson[(a)], V. Shutthanandan[(b)], Y. Xie[(b)], A. Laskin[(b)], B. de Foy[(a,*)], L.T. Molina[(a,**)], K. Dzepina[(c)], J.L. Jimenez[(c)], D. Salcedo[(d)], and M.J. Molina[(a,e)]

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Atmospheric pollution in the Mexico City Metropolitan Area (MCMA) remains a serious concern for the welfare of its residents and the surrounding environment. Aerosols emitted from a multitude of local and regional sources threaten human health, affect climate through direct and indirect effects, impair visibility, and participate in a variety of heterogeneous reactions. To investigate aerosol composition and emission sources in the MCMA, size-segregated samples of particulate matter ≤ 2.5 μm (PM2.5) were collected during the MCMA-2003 Field Campaign for chemical analysis by Particle-Induced X-ray Emission (PIXE), Proton Elastic Scattering Analysis (PESA) and Scanning Transmission Ion Microscopy (STIM), which were followed by source apportionment by Positive Matrix Factorization (PMF) [1]. Specific emissions sources were identified at a time resolution of 6 hr and included soil/dust, biomass burning and industrial emissions. Speciation of total mass into major chemical components indicates that approximately 50% of non-volatile PM2.5 during MCMA-2003 consisted of carbonaceous compounds, consistent with the finding that soot and organic aerosols comprised nearly two-thirds of total PM2.5 [2]. PESA hydrogen was apportioned to sulfate ((NH4)2SO4) and organic compounds assuming loss of nitrates, water, and semi-volatile organics prior to PESA analysis. Good agreement was observed in mass concentrations of PIXE sulfur (as SO4-2) and sulfate measured in real-time by aerosol mass spectrometry (AMS) and in the relative variation of time profiles for PESA organic H and AMS organic aerosol mass. The organic H obtained from our analysis was attributed to low vapor pressure organic aerosols which would be consistent with the presence of high molecular weight compounds. This information contributes to the understanding of urban aerosol chemistry and demonstrates for the first time the complimentary nature of PIXE/PESA and AMS techniques.

1. K.S. Johnson, B. de Foy, B. Zuberi, L.T. Molina, M.J. Molina, A. Laskin, Y. Xie, V. Shutthanandan, *Atmos. Chem. Phys*. 6, 2006, 4591-4600,
2. D. Salcedo, T.B. Onasch, K. Dzepina, M.R. Canagaratna, Q. Zhang, J.A. Huffman, P.F. DeCarlo, J.T. Jayne, P. Mortimer, D.R. Worsnop, C.E. Kolb, K.S. Johnson, B. Zuberi, L.C. Marr, R. Volkamer, L.T. Molina, M.J. Molina, B. Cardenas, R.M. Bernabé, C. Márquez, J.S. Gaffney, N.A. Marley, A. Laskin, V. Shutthanandan, Y. Xie, W. Brune, R. Lesher, T. Shirley, J.L. Jimenez, *Atmos. Chem. Phys.,* 6, 2006, 925-946

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H-2 **Environmental sciences**

Application of PIXE technique for identification of occupational exposure to Tantalum

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The aim of this study was to evaluate the occupational exposure to tantalum bearing particles in a pyrochlore processing plant. In this plant pyrochlore is processed to obtain Fe-Nb alloy. PIXE technique was used to analyze excreta (urine and feces) samples provided by workers and by individuals from the control group, food and water samples. The results of the monitoring programs for evaluating exposure of the workers to dust particles generated during the process to obtain Fe-Nb alloy have shown the availability of Tantalum bearing particles in the respirable fraction of aerosol. Although the workers were exposed to dust particles in the respirable fraction of aerosol, the bioassay results have shown that there is no correlation between Ta concentration in the respirable fraction of aerosols and in worker's urine samples. Ta concentration in urine samples were in the range of 21.99 to 111.32 μg/day and in feces samples 96.13 to 878.66 μg/day. The ratio between Ta concentration in urine and feces samples from each workers is less than 1, suggesting that the ingestion is the main pass way of Ta incorporation. Ta concentrations in urine and feces samples from control group were in the range of 32.18 to 103.09 μg/day and 84.29 to 632.32 μg/day, respectively. Ta concentration in food samples was below the detection limit, but Ta concentration in drink water was 112 μg/L. A biokinetic model adopt by ICRP was applied to estimate Ta incorporation by workers. The ration between Ta concentrations in urine and feces samples determined using the model was compared to the experimental values. The results show that the transfer factor (f1) adopted by ICRP to Ta does not represent the actual behavior of Ta in body fluid.

H-3 **Environmental sciences**

PIXE and aerosols in the 21st Century: Continuous rules!

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The advantages of the high momentum and well focused proton beams for aerosol analysis is an advantage so great as to offer the opportunity to dominate essentially every other method of inorganic aerosol analysis. The key point is that atmospheric sciences now recognizes that the rapid changes in aerosol size and composition as a function of time are so important that integrated (24 hr, $PM_{2.5}$) filter based methods are not adequate for the challenges of the 21st century, and in many cases they can be seriously misleading. While continuous methods have been achieved for major aerosols components (mass, organic carbon, elemental carbon, optical absorption, sulfates, and nitrates), although often at considerable cost, measurements of the gross and tracer elemental species have proven to be extremely difficult. This realization, together with advances in continuous sampling by size and time, places PIXE programs in an extremely favorable position. These advantages can be multiplied by using complementary techniques to obtain mass (soft β ray transmission, STIM [1]), total organic aerosols (PESA [1]), light elements (RBS, forward scattering), optical behavior (uv/vis/IR optical transmission and reflection) and morphology (SEM). Aerosols are vital in resolving some of the key environmental problems in the 21st Century. Two of the most important are tying the strong statistical association between aerosol mass and human health to specific components of aerosols with well defined sources, and understanding the role that aerosols have on global climate change, since aerosols are at present the source of roughly 80% of all the uncertainty in the global climate models.

1. Graham Bench, P.G. Grant, D. Ueda, S.S. Cliff, K.D. Perry, and T. A. Cahill. *Aerosol Science and Technology* 36 (2002) 642-651.

I-1 Environmental sciences

Detection of atmospheric aerosol sources at São Paulo City by PIXE analysis

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At the São Paulo Metropolitan Region (RMSP) leave today 19.3 million inhabitants. An inventory of the local environmental agency evaluates that essentially 95% of the air pollution come from the vehicular fleet and the remaining 5% from the 46,642 industrial units. This research is part of a main project designed to study the source of particulate matter in the region, during summer and wintertime, including eventual transport from some other nearby areas. In this specific work we studied the atmospheric aerosol at São Paulo, with fine time resolution, in the transition to a weekend. Our aim was to achieve a better resolution of the non-automotive sources, which intensity reduces during the weekend. Atmospheric aerosol samples collected every 3 h, from July 14th to 18th, 2006, were used to study the sources of particulate matter at the São Paulo city. Samples were collected using a stacked filter system, in order to fractionate the aerosols in their fine and coarse fractions (equivalent aerodynamical diameter, D<2,5µm and 2,5µm<D<10µm, respectively). The concentrations of particulate matter on both fractions were determined; trace elements contents were also measured on both fractions using PIXE technique. Four sources of coarse particulate (soil, industrial process, fossil fuel burning, and sulfates) and four sources of fine particulate (industrial process, fossil fuel burning, soil, and a source of Al and K) were identified using Principal Components Analysis, and meteorological information. The results showed that the high-resolution sampling enabled a good definition of the principal sources associated to the components, despite the relatively limited number of samples available in this study.

I-2 Environmental sciences

Chemical composition and mass closure for $PM_{2.5}$ and PM_{10} aerosols at K-puszta, Hungary, in summer 2006

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A comprehensive chemical aerosol characterisation was carried out at K-puszta, Hungary, from 24 May until 29 June 2006. Up to 11 June it was unusually cold at the site, but from 12 June onward it was warm. Several filter samplers were deployed in parallel, typically for separate day and night collections, and a total of 68 parallel collections were made. Among the samplers were two PM2.5 samplers (one with a 0.4 μm pore size Nuclepore polycarbonate filter, the other with 2 Whatman QM-A quartz fibre filters in series) and two PM10 samplers (with the same filter types as the PM2.5 samplers). All samples were analysed for the particulate mass (PM) by weighing. The Nuclepore polycarbonate filters were analysed for up to 29 elements by PIXE and for major anions and cations by ion chromatography [1]. The quartz fibre filters were analysed for organic and elemental carbon by a thermal-optical transmission technique [2]. Aerosol chemical mass closure calculations were done for the PM2.5 and PM10 aerosol. As gravimetric PM data we used the data from the Nuclepore polycarbonate filters. For reconstituting this PM, eight aerosol types (or components) were considered, whereby two aerosol types (including the important crustal matter component) were deduced from the PIXE data. Organic matter contributed by far the most to the PM2.5 and PM10 PM; it was responsible for 40-50% of the average PM during both the cold and warm periods. Noteworthy were the much larger percentages of crustal matter during the warm period than during the cold one (both for PM2.5 and PM10). In the PM2.5 aerosol, crustal matter accounted for 17% of the average PM during the warm period, but only for 3.1% during the cold period. For the PM10 aerosol, the percentages were 28% in the warm period and 10% in the cold one.

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2. M.E. Birch, R.A. Cary, *Aerosol Sci. Technol*. 25 (1996) 221.

I-3 Environmental sciences

Analysis of hot particles from Palomares (Spain) using proton and He μ–PIXE.

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More than 40 years ago took place in Palomares (Spain) an aircraft accident which involved the destruction of two nuclear weapons. A portion of the remaining transuranic contamination in the affected soils is present in the form of small high activity concentration particles (10-100 μm), also called "hot particles". Two radioactive particles stemming from Palomares area have been analysed with the nuclear microprobe of the National Accelerator Centre (CNA) in Seville. Compositional analysis, mappings and depth distribution of different elements has been performed by a simultaneous combination of Particle Induced X-ray Emission (PIXE) and Rutherford Backscattering Spectrometry (RBS). In a previous paper [1], oxygen, carbon, uranium and plutonium have been identified as the main components of the particles.

This work aims to explore the advantages of combining μ-PIXE and μ-RBS using a 5.5 MeV ^{4}He beam with conventional analysis using 3 MeV protons. The measurements performed with α–particles are more sensitive to the sample surface, while the results collected with protons are indicative of the average composition of the hot particles. In this way, important information about the depth distribution of Pu and U has been obtained. In some of the analysed points the surface of the hot particles was found to be partially depleted of Pu and U, which could be related to leaching and weathering processes.

1. J. García López, M.C. Jiménez-Ramos, M. García-León and R. García-Tenorio, accepted for publication in *Nucl. Instr. and Meth.* **B**.

I-4 Environmental sciences

Biomonitoring of airborne trace element in Mexico City using tree leaves.

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Airborne trace element composition was monitored using two urban species of trees (*Ficus benjamina & Ligustrum lucidum*) at Mexico's City Metropolitan Area. Leaves were collected from six parks in rainy season, end of rainy season and dry season during 2003 and 2004. Elemental composition was determined on unwashed leaves with PIXE (Particle Induced X-ray Emission) analyses using the PIXE at Nishina Memorial Cyclotron Center, Japan Radioscope Association. Differences were observed between the relative composition of particulate matter estimated from the leaves and published values of the relative composition of aerosols from Mexico City collected on filters [1]. Cluster analysis showed a high association among metals associated with secondarily formed aerosols (S, V and Ni) and among primary particulate matter (Mn, Zn, Cu, Ni, Co, V). Canonical analysis showed the leaves from the six parks group into three main zones (Northeast and Center, Northwest and Southwest, and East). Northeast and Center samples correspond to the allocation of the principal factories of the industrial zone of the Metropolitan Area of México´s City in the North Eastern zone of the city. Absolute Principal Component Scores Analysis was conducted to determine the relative contribution of apportion sources to the total mass.

1. J. Miranda, V.A. Barrera, O.S. Espinosa, A. Galindo, R.C. Núñez-Orozco Montesinos, A., Leal Castro, J. Meinguer, *Nucl. Instrum. and Meth.* B (2004) 157-160.

I-5 Environmental sciences

A possible application of the new Curium based XPIXE-α system to the monitoring of the atmospheric particulate matter

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The Radioisotope 244Cm emits 5.76 and 5.80 MeV alpha particles; also 9% yield of x-rays are emitted in the energy range of 12.13 to 21.98 keV. A spectrometer based on the use of curium source has been widely used during the Mars explorations. At the LNS/INFN laboratories of Catania a portable curium based spectrometer, named XPIXE-alpha system, has been designed and realised. The main characteristics of the new portable XPIXE-alpha spectrometer are described. PIXE and XRF mechanisms are simultaneously induced on samples being analysed. Due to the different behaviour of the ionisation cross section of alpha particles and x-rays this source enables to well excite both light elements and medium ones. The above characteristics are particularly suited for the analysis of atmospheric particulate matter in which major light elements are tracers of the crustal component (Si, Al, Ca and Fe) or tracers of the sea water component (Na and Cl) or as aerosol secondary component (S). Results of low intensity prototype are presented and limitations are discussed.

J-1. Invited Talk Advances in experimental devices

Cryogenic high resolution X-ray detectors

J. Hoehne

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Cryogenic detectors are very sensitive, energy-resolving, low-threshold photon and particle detectors which have been developed over the last decades for a variety of applications in particle physics and astrophysics. More recently, cryogenic detectors have also been applied as high-resolution, photon-counting detectors for energy-dispersive X-ray spectroscopy (EDS) and X-ray fluorescence analysis (XRFA). Cryogenic detectors can provide an about 10 times better energy resolution for X-rays than achievable with "conventional" energy-dispersive detectors, such as HPGe and Si(Li) detectors.

VeriCold has developed the POLARIS cryogenic detector system which serves industrial as well as R&D applications. The system which comprises of a superconducting sensor element as well as a cryogen-free cooler is fully automated and includes all standard spectral acquisition, manipulation and display features.

The basic working principle of cryogenic detectors will be introduced as well as recent scientific results. Advantages and limitations for the application of cryogenic detectors for PIXE applications will be discussed.

*Web page: www.vericold.com

J-4 Advances in experimental devices

External beam PIXE/PIGE measurements on aerosol samples at proton energies from 2 to 5 MeV

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At the 3 MV Tandetron accelerator of the LABEC laboratory of INFN in Florence, an external beam facility fully dedicated to PIXE and PIGE measurements of elemental composition of atmospheric aerosol is operational. PIGE is routinely performed simultaneously with PIXE to quantify the underestimation of light elements concentration due to the low energy X-rays absorption within the sample; this effect can not be simply theoretically evaluated since the sample matrix and thickness are not known *a priori*.

In order to correctly apply PIGE in thin target approximation, the proton beam energy should be selected so that the γ-ray emission cross section remains sufficiently constant despite the proton energy loss in the sample. Thus, γ-ray emission yields for $^{19}F(p,p'\gamma)^{19}F$ (E_γ = 110 and 197 keV), $^{23}Na(p,p'\gamma)^{23}Na$ (E_γ = 441 keV), $^{27}Al(p,p'\gamma)^{27}Al$ (E_γ = 843 and 1013 keV) and $^{28}Si(p,p_1\gamma)^{28}Si$ (E_γ = 1779 keV) have been measured under typical experimental conditions (i.e. external beam) for proton beam energies from 2 to 5 MeV. Several suitable proton energies have been indeed determined. As regards Na analysis, an important marine aerosol tracer, the effectiveness of this approach has been tested both on certified standards and aerosol samples collected at different sampling sites; a 30% underestimation from PIXE data has been found for PM_{10} aerosol samples.

A thorough investigation of PIXE minimum detection limits (MDLs) as a function of proton beam energies from 2 to 5 MeV has been carried out to find out the best energy for PIXE meausurements on aerosol samples collected on different substrata, namely Teflon, Quartz fibre, Kapton and Nuclepore. Note that minima in the ^{19}F γ-ray emission yields mark useful energies for PIXE measurements on aerosol collected on Teflon (CF_2) filters, since the Compton γ-ray background in PIXE spectra is reduced, thus enhancing the MDLs.

K-1. Invited talk — Material science

PIXE: Elemental concentration and beyond

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Materials science is an ever-changing field which demands the development of new approaches to challenging problems. For instance, semiconductors like silicon still play a key role in applications for advanced technologies, and the SiO_2/Si system has been the interface of choice in microelectronics for over forty years. However, new technologies are pushing the gate oxide thickness to the ultra-thin limit, likely leading to a replacement of this oxide by a higher dielectric constant (high-K) material. Among several IBA techniques, PIXE has been employed in the analysis of materials due to its special features, which make it suitable for a variety of applications. As far as advanced materials are concerned, PIXE has been employed mostly in the detection of impurities and in studies related to lattice location and displacement of elements. In order to push the PIXE technique even further, it is important to know all basic processes involved in the ion-matter interaction. Moreover, the PIXE technique itself can be applied in fundamental research which can eventually lead to a deeper insight of the physics behind basic processes. In this work, it will be presented some difficulties related to the analysis of thin films and how the knowledge of some fundamental aspects of the ion-matter interaction might be important for future developments and applications of the PIXE technique. In particular, the role of the stopping power will be discussed. As an example, it will be shown how this technique, carried out with ionic hydrogen cluster, can provide important information about the physics involved in the interaction of swift ions with matter.

K-2 Advances in experimental devices

Automated PIXE-RBS depth profiling using multiple PIXE spectra and simulated annealing

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There are many examples of strongly non-uniform layered structures which can not be solved by RBS and related particle scattering spectroscopy because the elements of interest are at minor element concentrations, are light relative to the substrate or are too heavy to be resolved from other constituents. Recent examples of these that we have encountered include the determination of depth profiles of Cl in ZnS and Y in CrAlYN.

However, these elements have a unique PIXE signal which is affected through X-ray absorption by the detailed depth profile of the element. Provided the X-ray absorption correction can be modelled accurately then the depth profiles of the elements of interest may be extracted from multiple PIXE spectra collected at different geometries. This approach has already been described (e.g. [1]), but the difficulty of calculating the absorption effects means that its use has until now been restricted to depth profiles which can be described using simple few-layer models.

In this paper we describe the use of the DataFurnace code [2, 3] to approach this problem for profiles of arbitrary shape. This code fits IBA spectra automatically with the global minimisation algorithm of simulated annealing, and has recently been extended to accept PIXE spectra. Thus we can use the particle scattering spectra to determine the major element profiles and also fit the PIXE and particle spectra simultaneously and self-consistently. DataFurnace does a correct calculation of the X-ray absorption for an arbitrary layer structure with no limit on the number of layers, using the algorithm of Reis [4], and is accurate for the cases where fluorescence may be neglected. The method will be illustrated using results from measurements carried out both simultaneously (multiple PIXE detectors at different angles) and sequentially (single PIXE detector with sample rotation).

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2. C. Jeynes, N.P. Barradas, P.K. Marriott, G. Boudreault, M. Jenkin, E.Wendler, R.P. Webb, *Journal of Physics D-Applied Physics* 36 (2003) R126.
3. C. Pascual-Izarra, N.P. Barradas, M.A. Reis, *Nucl.Instrum.Methods* B 249 (2006) 820-822
4. M. A. Reis, Nucl.Instrum.Methods B68 (1992) 300.

K-3 Advances in experimental devices

Particulate screening using micro-PIXE and multivariate statistical analysis

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Forensic analysis of particulates harvested from filters and other matrices is usually done by first screening to identify particles of interest (POIs) and then individually picking and analyzing these POIs in a labor-intensive process. Advances in μPIXE combined with Multivariate Statistical Data Analysis methods1 offer new and complementary tools for automated nondestructive identification and characterization of POIs with high selectivity, specificity, and sensitivity. Recent results have demonstrated the capability to perform both in-vacuo and ex-vacuo analyses of field-collected samples. These μPIXE techniques measure the major, minor and trace (ppm) element composition of particles and also allow for ultra-trace (ppb) element analysis using mass spectrometry by locating and marking POIs in the collection media. Such measurements contain important information about the origin, processing and transportation of evidence collected at crime scenes or acts of terrorism. The Sandia AXSIA2 multivariate statistical analysis has already demonstrated the capability to automatically produce high quality elemental concentration images taken with μPIXE3, and in this paper we show how AXSIA can also be used to classify the evidentiary value of particulates. A new ex-vacuo system is described that uses a transmission μPIXE geometry and a high resolution optical scanner for suspicious particle identification and stage navigation, and an in-vacuo particle analysis system has been developed to identify extremely small (<5 □m) POIs.

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2. Kotula, P.G. et al., *Microsc. Microanal.* 9 (2003) 1-17.
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L-1. Invited Talk Advances in experimental devices

3D Micro PIXE – a new technique for depth resolved elemental analysis

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A novel experimental technique, 3D Micro- Particle Induced X-ray Emission (PIXE) is described in the present work. 3D Micro-PIXE is realized by using an X-ray optic in front of the detector, thus creating a confocal arrangement together with the focused proton micro-beam. This confocal setup defines a probing volume from which information on elemental distribution is obtained. If a sample is moved through the probing volume, depth resolved measurements become possible with a resolution in the micrometer regime. This development was motivated by the corresponding successful implementation of two X-ray lenses in a typical X-ray Fluorescence (XRF) set-up [1, 2]. For the experimental realization of this technique, the nuclear microprobe of the Jožef Stefan Institute was used [3] in order to establish, characterize and apply the confocal setup for 3D Micro PIXE for the first time [4].

The experimental setup was characterized with respect to its spatial and depth resolution. The 3D Micro PIXE measurements confirmed the potential of the technique to resolve elemental distribution in separate layers in a complex structure overcoming the depth analytical limitations of standard ion beam techniques. As an application example of this new non-destructive analytical technique, an archaeological ceramic fragment exhibited a black gloss layer (of about 20 μm thickness) onto a porous ceramic body was analyzed and a first approach to simulate the complex experimental results is presented. The potential of 3D Micro-PIXE to provide advanced qualitative information on the elemental distribution in the sample is discussed and compared with the 3D Micro-XRF.

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2. B. Kanngießer, W. Malzer, A. Fuentes Rodriguez, I. Reiche, *Spectrochimica Acta* B, 2005, B 60, 41.
3. P. Pelicon, J. Simčič, M. Jakšić, Z. Medunić, F. Naab and F.D. McDaniel, *Nucl. Instr. and Meth.* B, 2005, 231, 53
4. A. G. Karydas, D. Sokaras, C. Zarkadas, N. Grlj, P. Pelicon, M. Žitnik, R. Schütz, W. Malzer and B. Kanngießer, submitted

.

L-2 Advances in experimental devices

A model for quantitative micro–PIXE analysis in confocal geometry

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In a typical Micro-PIXE set-up the insertion of an X-ray optic, such as a polycapillary half lens, in front of the X-ray detector in combination with the focused charged particle beam defines a confocal geometry. This geometry is characterized by the creation of a probing-volume that is produced by the intersection of the X-ray lens focus with the focus of the proton micro-beam. By moving the sample across the probing-volume, the characteristic X-ray intensities produced only within this probing-volume are detected. This experimental procedure will result, finally, into an intensity profile versus depth for each detected element. We have demonstrated very recently the feasibility of such kind of depth resolving elemental analysis with Micro-PIXE [1].

The aim of this work is to present a quantitative model that simulates the Micro-PIXE intensities in confocal geometry providing the possibility for depth resolved analysis of composite-stratified materials. The proposed quantification approach uses an analytical description for the energy dependent spatial response function of the X-ray lens at the focal region. Incorporating the beam profile, the probing-volume is analytically described through a sensitivity function expressed in spatial coordinates in an analogues manner to the one for 3D Micro-XRF in confocal geometry [2]. First, the X-ray intensity is calculated taking into account the well known processes that involve in the PIXE process (ionization, stopping power, and self-attenuation). The convolution of the PIXE intensity with the sensitivity function produces simulated PIXE intensity profiles. This is the first step towards a full quantitation for Micro-PIXE in a confocal geometry. The influence of various experimental parameters (beam energy, spatial resolution of the X-ray lens, micro-beam dimensions, irradiation and detection angles), as well as of the sample characteristics like the structure (thickness, composition) of the individual layers that compose the analyzed sample, on the simulation of the PIXE intensity profiles is examined and discussed. Examples of the advantages and benefits of the Micro-PIXE analysis in confocal geometry are given.

1. A. G. Karydas, D. Sokaras, C. Zarkadas, N. Grlj, P. Pelicon, M. Žitnik, R. Schütz, W. Malzer and B. Kanngießer., submitted.
2. W. Malzer and B. Kanngießer, *Spectroch. Acta*, 2005, Part B' 60, 1334.

L-3 Advances in experimental devices

Characterisation of the urban and suburban aerosol in the City of Prague

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A one year comprehensive study of the composition of Prague urban and suburban aerosol composition has been curried out form February 2004 to April 2005. The project consists of concurrent sampling on two above roof sites in the Prague City. The first site is located at northern west suburban of Prague in the Institute of Chemical Process Fundamentals (ICPF), the second one is located downtown of Prague city on the roof of the 4 storey building of Faculty of Natural Sciences of Charles University at Albertov quarter. Both sites were equipped with following sampling devices and on-line instruments: scanning mobility particle sizer (SMPS) with CPC model 3022, 10 stage Berner low pressure cascade impactor (Berner and Lürzer (1980)), PM1 URG PM1 cyclone sampling head, Teflon (Zefluor, Pall), PM2.5 (Leckel PM2.5/1m3/h sampling head, teflon (Zefluor, Pall)/quartz(whatman QMA) filter pack) and PM10 (Leckel PM 10/1m3/h sampling head, quartz/quartz filter pack)filter samplers and Gent SFU (Nuclepore 8µm/Nuclepore 0.4µm) sampler. Next there were used instruments (Horiba) for the measurement of NO, NO_2, NO_x, O_3, CH_4, and non-methanic hydrocarbons and basic meteorological measurements like ambient temperature and humidity, wind velocity, wind direction and total sun radiation. The samples are analysed by IC, PIXE, INAA, AAS and OC/EC. Mainly the PIXE, PIGE a IC results on the SFU and BLPI samples will be discussed in our contribution.

L-4 Advances in experimental devices

Crocker Nuclear Laboratory and PIXE A Historical Perspective

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Researchers at Crocker Nuclear Laboratory initiated the development of a PIXE system in the early 1970s. After an evaluation of several different energy alpha and proton beams 4.5MeV protons emerged as the beam of choice. The paper presents a discussion of the decisions that were made to ensure a system that would provide precise and accurate results on lightly loaded air filters. A further condition that was imposed is that the cost had to be economically palatable to funding agencies. In addition to discussing the development of the system, specific applications are presented. The first application involves the analysis of samples collected during the US National Parks IMPROVE program. There are currently 150 sampling sites that generate samples every three days. The second example is the analysis involved with the evaluation of the US Department of Agriculture's attempts to assess the impact of agriculture on air quality. Finally a cooperative program between Crocker Nuclear Laboratory and the University of Chile, Faculty of Science to determine air quality in Antarctica is presented. All of these endeavours present unique challenges. Similarities and differences will be presented. The presentation of results and an evaluation of the challenges present leads to a discussion of the requirements and constraints that possibly will be placed on systems and experiments of the future. These requirements are not only based on the physics of the system, in fact, the challenges here may be small compared to the demand placed by legislative of regulatory bodies. It is becoming increasingly important for researchers to straddle the boundaries between the analytical, legislative and regulatory communities. Questions discussed in the paper include the role of standards and reference materials; the generation and interpretation of minimum detectable limits, especially in the presence of multiple interferences and the reproducibility of results in various systems. Hopefully the experiences and evolution of Crocker Nuclear Laboratory's PIXE system will benefit those either just developing or utilizing the PIXE technique.

.

POSTER SESSION I

PI-1 Basic physical principles

L X-ray production cross-section ratios for protons

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The great majority of data from measurements of proton-induced L X-ray production cross-sections have been converted into ionization cross-sections using L subshell fluorescence yields and Coster-Kronig probabilities recommended in a 1979 review. A new critical review [1], based on a much expanded data set, recommends values that in some cases differ significantly from those of the earlier work.

The present work seeks to test the combination of the ECPSSR-DHS ionization cross-sections and the new subshell yields [1] by measuring the X-ray intensity ratios Lβ/Lα and Lγ/Lα for six heavy elements using a Si(Li) detector coupled simultaneously to both analog and digital signal processors. Proton energies of 2.0 and 2.5 MeV were chosen to ensure that possible united atom and intra-shell effects would be small, and also to make the work directly relevant to the PIXE database. Careful attention was paid to spectrum fitting in order to reduce hitherto ignored sources of error: Voigtian lineshapes were used and Compton steps on the low energy side of peaks were included in the peak model. Only the most intense diagram line in each of the L1, L2 and L3 series was varied within the fit, the remaining lines being locked to their principal line via the assumption that theoretical Dirac-Fock radiative transition rates are accurate.

Agreement between measured and predicted Lβ/Lα and Lγ/Lα ratios was significantly better (viz. within a few percent) when the former fluorescence and Coster-Kronig parameters were replaced by the 2003 values. This result provides a significant improvement in the database used for PIXE analysis. However, serious gaps remain in measured fluorescence and Coster-Kronig yields and a full MCDF theoretical treatment is not yet available, so much room for improvement remains.

1. J.L. Campbell, *At. Data Nucl. Data Tables* 85 (2003) 291-315.

PI-2 Basic physical principles

Empirical approximation for L_α production cross-sections

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In analytical applications, the L lines of the elements heavier than silver or tin are frequently used. It is usually the strongest $L\alpha_{1,2}$ line induced by protons which is of practical importance. Though a reliable data base of L production cross section is certainly needed, the values equivalent to the K-shell data base are not available. The reason may be sought in a smaller number of measurements, in a more complicated case of three L subshells exhibiting migration of vacancies, and in the data base of intershell transition rates which is still being refined. Only two groups of authors tried to obtain the mean production and ionization cross sections [1,2]; only the first results are published, yet the second contributed to the GUPIX data base. Since the data [1] are available in a tabular form only, we propose here a practical analytical approximation which would enable simple interpolation and use of the data in thick target algorithms. The approximation has no ambition of following the physical models of the ionization process, so the logarithm of $L\alpha_{1,2}$ production cross section is given as an eight parameter rational function of the logarithm of proton energy. The accuracy of fit is within several percent in the high energy region, about 5 % in the vicinity of the discontinuous jumps of the data [1], and about 10-20% in the low energy region below 200 keV.

1. M.A. Reis, A.P. Jesus, *At. Data Nucl. Data Tables* 63 (1996) 1.
2. I. Orlić, C.H. Sow and S.M. Tang, *At. Data Nucl. Data Tables* 56 (1994) 159 and unpublished.

PI-3 Basic physical principles

Mo L X-rays relative yield ion energy dependence

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In previous works, it was shown that relative yields of W L x-rays emitted under proton irradiation depend on the ion beam energy and can be used to establish intensity ratio variation patterns (IRVP) [1], which may then be used to distinguish between different chemical species of the same element. In the present work, Mo L_{α} , $L_{\beta 3,4}$ and $L_{\beta 2,15}$-spectra were obtained using the high resolution Johansson type crystal spectrometer at the Micro Analytical Center of Josef Stefan Institute in Ljubljana, and Mo, $MgMoO_4$ and $(NH_4)_6Mo_7O_{24}.4(H_2O)$ L-spectra were obtained using a Si(Li) detector at ITN in Lisbon, and several proton beam energies between 0.3 and 2.3 MeV (which correspond to reduced velocities values between 0.6 and 1.7). Spectra deconvolution, whenever required, used a Bayesian Inference process to reduce errors due to spectra fitting. In the case of the Mo foil and the $MgMoO_4$ samples, ultra pure material were used to avoid any possible target contamination. In the case of $(NH_4)_6Mo_7O_{24}.4(H_2O)$ this was not possible so P.A. material was used. In this communication the observed $L_{\beta 2,15}/L_{\beta 3,4}$ ratio variability presented but unexplained in a previous work [2] will be discussed based on new evidence and comparison of data relative to different chemical species. Other Mo intensity ratio variation patterns will also be presented and discussed.

1. M.A. Reis, P.C. Chaves, J.C. Soares, *Nucl. Instrum. Meth. B* 229 (2005) 413-424.
2. P.C. Chaves, M.A. Reis, N.P. Barradas, M. Kavčič, LI Sub-shell X-rays Relative Intensity Dependence on Ion Beam Energy; 19th International Conference on the Application of Accelerators in Research and Industry, August 20 - 25, Fort Worth, Texas USA, 2006, *Nucl. Instrum. Meth. B* (in press).

PI-4 Basic physical principles

Measurement of K–L radiative vacancy transfer probabilities in rare earth elements bombarded with 3 MeV-4 MeV protons

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One of the main difficulties in the measurement of concentration of rare earth elements in soil samples using PIXE is the overlap of the L-lines emitted by these elements with the K X-rays from more abundant lighter elements, such as Mn, and Fe. Therefore, the possibility of using more energetic proton beams to induce the K X-rays of the rare earths might be explored, although information about ionization cross sections or other atomic parameters is still scarce. Therefore, in this work the K-shell X-ray production cross sections and intensity ratios for rare-earth elements have been measured following irradiation with proton beams having energies between 3 MeV and 4 MeV. From the X-ray intensity ratios, the radiative vacancy transfer probabilities from the shell K to the L sub-shells were determined. The experimental data were compared to theoretical predictions, such as the Plane Wave Born Approximation or the ECPSSR theory, for the ionization cross sections, the semiempirical fits of Venugopala-Rao et al. [1] for X-ray line intensities, and the Scofield [2] theoretical predictions for radiative vacancy transfer probabilities. The results showed a fair agreement between theory and experiment.

The authors acknowledge the technical assistance of Mr. K. López and Mr. F.J. Jaimes. Work supported in part by PAPIIT-UNAM (contract IN-103307).

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2. J.H. Scofield, *At. Data. Nucl. Data. Tab.* 14 (1974) 121.

PI-5 Basic physical principles

K-shell vacancy production by impact of projectiles with a previous orbital vacancy

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The cross sections determination for the ionization of a target atom with a specific projectile atom is fundamental for the understanding of this atomic interaction phenomenon. It is also important to improve results in quantitative analysis with the technique Particle Induced X-ray Emission, PIXE. In quantitative PIXE analysis, matrix effects became a fundamental question. Bulk material affects the examination of the specific elements in a sample. Depending on the analyzed element, the projectile particle type and its energy; components of the bulk material can produce a major effect on the X-ray production yields. Background in the X-ray spectrum, detection limits, particle stopping powers and X-ray photons attenuation, are some factors to be considered for the quantitative analysis. Also the ionization cross sections could be affected by the previous state of the projectile, conditioned by the passage in the material producing multiple ionizations and changes in its charge state. It has been reported that ^{40}Ar ions can acquire 2p vacancies by the passage in matrixes containing ^{32}S and ^{16}O. The vacancy in the projectile ion modifies the normally expected mechanism of ionization in a specific target atom. Different matrix compositions and geometries (sample in the surface or immerse in the bulk) are analyzed in relation to the ionization cross sections.

PI-6 Basic physical principles

Comparison of Gd-K and L X-rays RYIED and proton-NMRD

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In previous works, it was shown that, for W under proton irradiation, the x-ray relative yields were dependent on the energy of the irradiation ion beam. This was then named Relative Yield Ion Energy Dependence (RYIED) and was seen both for the L [1] as well as for the K shell x-rays[2]. In the present work, this effect is further explored and comparisons are made to data originating in a totally different analytical technique, namely the analysis of the proton longityudinal relaxation time dependence on the Larmour frequency (proton-NMRD). This technique when applied to water solutions of chelates of paramagnetic ions, provides information on the electronic configuration of the chelate, including the electrons surrounding the core ion. Experimental evidence related to RYIED effects strongly suggests an important connection between intensity ratio variation patterns and the electronic configuration where the emmiting ion is embeded. This work, focus on Gd K-spectra collected using a CdTe detector and Gd L-spectra collected using a Si(Li) detector, obtained for irradiation with protons having reduced velocities between 0.17 and 0.4 for the K-shell x-rays and between 0.3 and 1.2 for the L-shell x-rays, corresponding to proton energies between 0.3 and 3.8 MeV.

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PI-7 Advances in experimental devices

GUPIXWIN - a new software package for PIXE analysis

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The GUPIXWIN package combines a simple and familiar Windows interface with a fast Fortran engine derived from the former GUPIX code. GUPIXWIN offers a significant functionality, flexibility, and ease-of-use improvement over the former GUPIX. Version 1.2 was presented at GUPIX Schools events in Paris and Florence in late 2005, and feedback from these events facilitated extensive feature additions and interface improvements, most of which were released as v1.3. GUPIXWIN offers guidance via help buttons at all stages and a comprehensive manual. Common mistakes are easily avoided thanks to input screen layouts being logical and drawing the user's attention to subtle problem areas.

GUPIXWIN handles thin, intermediate, thick and layered samples, and offers both trace element solution in a known matrix and iterative matrix element solution. The user may define a matrix via atom numbers, or by concentrations of specified pure elements, oxides, or arbitrary chemical compounds. Detector options now include Moxtek polymer windows for work with low-energy X-rays. Extensive file output options are centralized in an 'Output Manager'. All of GUPIXWIN's file output is presented in the universally readable formats CSV and ASCII text.

The top-hat filter approach is still used for background stripping, but channel-dependent filter dimensions are offered, as is a two-region filter that is optimum for weak high-energy peaks. In addition to the conventional peak pile-up approach a peak+continuum pile-up option is included.

The recently released version 2.0 contains an expanded batch mode which now includes support for two-detector PIXE systems. In this mode, the iterative matrix solution is peformed first and the resulting concentrations are automatically used to calculate the trace element solution. Consistency checking is simple thanks to side-by-side presentation of the matrix and trace output data.

PI-8 Advances in experimental devices

A new target chamber for simultaneous Ion Beam Analysis at the University of Chile

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A new experimental chamber has been installed at the 3.75 MeV Van de Graaff accelerator laboratory at the University of Chile. The aim of this facility is to perform simultaneous PIXE, PIGE, RBS and PESA analyses with a millimetre size beam. This chamber, manufactured by CINEL-Strumenti Scientifici, is designed for automatic analysis of a maximum of 30 samples affixed to a wheel driven remotely by a PC card interface and is currently equipped with a Si(Li) detector and two surface barrier detectors for RBS and PESA analysis. In addition, a NaI(Tl) gamma-ray detector will be used to conduct simultaneous proton-induced gammas emission (PIGE) analysis. The spectra are acquired using a multiparametric data acquisition system based on the CAMAC standard with PelROOT interfacing software. The calibration of the PIXE set-up has been done with Micromatter thin films and other thick standards. The GUPIXWIN Version 2.0 software package recently installed in our laboratory is used to process the PIXE spectra and the results were compared with certified values to obtain the experimental constant H (solid angle and correction factor) which represents the set-up geometry and the system for charge monitoring. A similar procedure for RBS analysis has been done by means of the SIMNRA code. The chamber will be used in different studies like atmospheric aerosol samples, archaeometric materials, biological tissues and others.

PI-9 Advances in experimental devices

Some specific features of the Budapest-Hamburg proton microprobe

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Thanks to its home made design and construction the meanwhile successfully re-installed Hamburg proton microprobe (the Budapest-Hamburg proton microprobe since then) has some specific and unique features. In addition to the usual backward positioning (in our case at 120^{o} angle with respect to the proton beam direction) the design of the target chamber allows a unique detector arrangement geometry to maximize the X-ray detection efficiency: a large area (80 mm^2) Si(Li) detector can be placed just behind the sample to be analysed. The final fine focusing of the 2.5 MeV proton beam can be performed by a dedicated computer controlled procedure, where the intensity of the secondary electron pileup events produced by circular scanning on a hexagonal copper grid of 65 μm spacing is maximized by varying the currents of the focusing quadrupoles. The observed asymmetry of the smallest available beam size – less focusing can be done horizontally compared to the vertical one - can mainly be attributed to mechanical vibrations. To detect these vibrations and to eliminate their resolution deteriorating influence a seismic sensor is attached to the target chamber. If the signal of this sensor is higher than a pre-determined reference value, the data collection can be automatically inhibited. Results of these efforts are also presented.

PI-10 Advances in experimental devices

New Tandetron Laboratory at NPI

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A new Tanderton laboratory has been recently build up at the Nuclear Physic Institute of the Academy of Sciences of the Czech Republic at Rez near Prague. The accelerator is The new Tandetron 4130MC from HVEE with terminal voltage 3MV. The accelerator itself has been set into the operation by HVEE technicians at the end of the 2005. Since then we have installed two ion beam lines for the ordinary and heavy ion RBS analysis, TOF-ERDA, High energy ion implantation and Ion channelling analysis. These two lines are already used for experiments. We also started installation of the third ion beam which will be equipped with flexible target chamber for simultaneous analysis by PIXE, PIGE, RBS. We also plan to extend this line for the external beam applications. The fourth line will be dedicated for ion microprobe and it is planed to be finished during years 2007-2008.

More information about our laboratory can be found at http://neutron.ujf.cas.cz/vdg/home.html

PI-11 Advances in experimental devices

Development of 3D micron-CT using PIXE

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We are developing "micron-CT", using micro-PIXE for in vivo imaging. This system comprises an X-ray CCD camera (Hamamatsu photonics C8800X9) with high resolution (pixel size: 8x8μm2, number of pixels : 1000x1000) and a X-ray point source with a spot size of 1.5x1.5μm2 which is generated by irradiation of a microbeam on a pure metal target. Thus we can acquire projection data with high resolution. The sample is placed in a tube of a small diameter, and rotated by a stepping motor. The 3D images were reconstructed from the obtained projection data by using cone-beam CT reconstructon algorithm[1,2,3]. X-ray spectra produced by heavy charged particle bombardment, exhibit a much smaller continuous background compared to electron bombardment. Therefore, X-rays produced by ion beam can be used as a monochromatic and low energy X-ray source. The feature is very effective to investigate small insects . Moreover we can get elemental distribution image of object by choosing appropriate characteristic X-rays corresponding to the absorption edge. On the other hand, the conventional X-ray CT, where usually continuous X-rays are used, provides images of the electron density in the object. Using this system, we were able to get 3D images of a living ant's head with 6 μm spatial resolution. By using Fe-K-X-rays (6.40keV) and Co-K-X-rays(6.93keV), we can investigate the 3D distribution of Mn(K-absorption edge=6.54keV) in ant's head. Therefore, micron-CT is a powerful tool for biological studies.

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3. T. Yamaguchi, K. Ishii, H. Yamazaki, S. Matsuyama, Y. Watanabe, S. Abe, M. Inomatata, A. Ishizaki, R. Oyama and Y.Kawamura, *I.J. PIXE*, Vol.15 (2005), 195-202.

PI-12 Advances in experimental devices

Development of an ion microprobe setup for complex elemental analysis of individual microparticles

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Besides the bulk analysis of the airborne particulate matter, individual particle investigations performed on electron or nuclear microprobes provide useful additional information about the size, morphology and chemical composition of atmospheric aerosol particles. However, due to the new directions in atmospheric aerosol research it is no longer sufficient to determine the inorganic elemental composition of individual aerosol particles, but information about the light elemental (H, C, N, O) content together with the structure of the particles is required.

In this work we present a measurement setup and data evaluating system installed at the Debrecen ion microprobe facility which is able to determine the quantitative elemental composition and distribution of individual microparticles even for light elements by using complementary elemental techniques.

Due to recent improvements the determination of quantitative elemental concentration from C to U and the building-up of 2D true elemental maps with a spatial resolution of 2μm became attainable for complex inhomogeneous thin samples [1,2]. We extended these capabilities in the direction of the quantitative detection of light elements including H, Li, B, C, N, O, F. The analytical techniques available are STIM to determine the area density of the sample, ERDA to detect the H content, PIXE-PIXE to detect characteristic X-rays originating from elements with Z>5, RBS to determine the matrix composition, to crosscheck the validation of the data and the beam dose measurement, and if needed, DIGE for determining the light element ($3 \leq Z \leq 9$) content.

The above described analytical methods will be suitable not only for the analysis of airborne particles, but the integrate study of complex inhomogeneous thin samples (like biological samples) will also be achievable, opening up a wide range of applications.

1. Zs. Kertész *et al*., *Nucl. Instrum. Meth. B* 231 (2005) 106.
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PI-13 **Biology and biomedical sciences**

Elemental mapping of plants using submilli-PIXE camera

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Phytoremediation is a technology for cleaning metal-contaminated soils using plant physiology. *Pteris vittata*, which is known as a hyper-accumulator of As, was analyzed by an in-air submilli-PIXE camera. This PIXE analysis system provides spatial distribution images of elements in a region several cm^2 with a resolution of *ca.* 0.5 mm. 3 MeV proton beam (10 nA beam current, <0.5 mm beam diameter) from a 4.5-MV single-ended Dynamitron accelerator was extracted to open air through 12.5 μm thick Kapton film. Proton beams were scanned 15x15 mm^2 on a surface of plant samples which were fixed to a target frame just after the beam exit window. The distance from the beam exit window and a sample is around 5mm. X-ray energies and beam positions were simultaneously measured in order to obtain a spatial distribution of elements. X-rays from targets were measured with two Si(Li) detectors; No.1 detector (7.5 μm thick Be window, 10 mm^2 active area) with a low geometric efficiency is well suited for detection of an element of low atomic number, and No.2 detector (12.5 μm thick Be window, 50 mm^2 active area) with a 100-μm Mylar absorber allows detection of X rays > 4 keV and the removal of recoil protons. The list mode data acquisition system can sort the data for a selected element / energy region and generate an elemental image even while the data are accumulated. Elemental images of leaves were obtained *in-vivo* without sample preparation. Elemental map of the leaves showed that arsenic was accumulated in the edges of *Pteris vittata* leaves. From these findings, it is possible to reveal the distribution of heavy metals and their location in the plant using the submilli-PIXE camera. PIXE analysis is an effective tool for undertaking phytoremediation research.

PI-14 **Biology and biomedical sciences**

PIXE and PESA analysis of metalloprotein stoichiometry

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We have developed a novel combination of techniques for determining the stoichiometric ratio of metal to protein in metalloproteins after polyacrylamide gel electrophoresis. Native polyacrylamide gel electrophoresis was used for protein separation and after drying the gels, they provide thin samples necessary for ion beam analysis. Proton Elastic Scattering Analysis (PESA) is used to determine the areal densityof the thin targets by quantifying the energy loss of the transmitted ions. Particle-Induced X-ray Emission (PIXE) is used to determine the concentrations of heavy metals in the protein locations within the gel. The combination of these two ion beam analysis techniques allows us to quantify the stoichiometric metal-to-protein ratio in metalloproteins. Inital work has focused on proof-of-principle with cytachrome-c, while current work centers on the production and quantification of Cu-metallothionein from the CUP1-2 locus in *Sacchromyces cervisiae*. The vacuum preparation methods, ion beam analysis methods as well as the preliminary results will be presented.

PI-15 **Biology and biomedical sciences**

Improved radiosensitive liquid core microcapsules for the targeting of chemotherapeutic agents

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Microcapsules consisting of alginate and hyaluronic acid that can be decomposed by radiation are currently under development. Previously, we have found that microcapsules comprising of alginate and hyaluronic acid, and Yttrium are efficiently decomposed by radiation. In this study, we introduce that our microcapsules were much improved by Calcium and Yttrium polymerization. We also introduce amount of released their core contents by colorimetric analysis, and their frequencies of decomposition, using micro PIXE camera.

Solutions of 0.1% (wt/vol) hyaluronic acid were mixed into a 0.2 % alginate solution. To these mixtures, Carboplatin (l mg) and indocyanine green (12.5μg) was added and the resulting material was used for the capsule preparation. The capsules were prepared by spraying the material into mixtures of 4.34 % solution of CaCl2, supplemented with from 0 to 5.0 x 10-3 %Y.

These capsules were irradiated by a single dose of 0.5, 1.0, 1.5 or 2 Gy 60Co γ ray radiation. Immediately after irradiation, the frequencies of decomposed microcapsule was determined, using a micro Particle Induced X-ray Emission (PIXE) camera, and amount of released their core contents were measured, using colorimetric analysis by indocyanine green.

The frequencies of decomposed microcapsules by micro PIXE camera strongly correlated with released amount of their core contents by colorimetric analysis. Microcapsules that were polymerized 4.34 % solution of CaCl2 supplemented with 5.0 x 10-3 %Y revealed the maximum decomposition and releasing of their core contents with 2 Gy irradiation. They were 48.9±4.3 % in frequency of decomposition, and 68.9±3.1% of releasing their core contents.

Our liquid core microcapsules suggest a new potential use for radiation: the targeted delivery of the chemotherapeutic agents or radiosensitizers. This offers the prospect of increased combined effectiveness of radiation with chemotherapy or radiosensitization and decreased adverse side effects.

PI-16 Biology and biomedical sciences

Measurements of Sr/Ca in bones to evaluate differences in temperature

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The replacement of calcium atoms by strontium in aragonite crystals ($CaCO_3$) was frequently observed due to the fact that both are from Alkaline earth metal group. The substitution rate depends on crystal type, chemical and physical growth conditions, including temperature. Analysis of aragonite from sea shells and coral skeletons shows a signifcant correlation between the Sr/Ca ratio in these crystals and the sea water temperature obtained by satellites and ship readings [1, 2]. In this work we present the results of a study that correlates Sr/Ca ratio and temperature during the formation of another calcium crystal, the hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), main mineral compound of teeth and bones from vertebrates. For that we investigated bones of animals of different species. These animals, independent of its thermoregulation pattern (endothermic or ectothermic), have variations of internal temperature along the body. Usually the warmest parts are the parts closer to the heart and to the brain, and the coldest are the extremities, like feet. One interesting application of this work is to differ warm-blodded animals from cold-blodded ones just by measuring Sr/Ca ratio in their bones. Bones from a crocodile (Caiman yacare) and two dogs (a poodle and a non defined race) were analized using Thick target PIXE. A 1.78 (18) MeV external proton beam was used to probe the surface of bones in external PIXE setup at Laboratory of Materials Analyses by Ionic Beams at University of São Paulo (LAMFI-USP). An accumulated charge of about 10 μC per sample was used. Emitted X-rays were collected using Si-PIN detectors (140 keV for Fe). In order to improve Sr X-rays detection, a 0.5 mm plastic filter was mounted to attenuate Ca X-rays, between the samples and the Si-PIN detector, allowing an increase of the beam current intensity. Results show that the Sr/Ca ratio is lower in bones from the body's warmer parts and higher in colder parts indicating a similar behavior with results for coral skeletons.

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PI-17 Biology and biomedical sciences

Simultaneous PIGE and PIXE analysis of dental composites using a single detector

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PIGE is widely used for the analysis of light elements as a complementary method to PIXE. In particular, fluorine is analyzed by the 110 and 197 keV γ radiation from the $^{19}F(p, p'\gamma)^{19}F$ reaction at proton energies around 3 MeV. Fluorine is of utmost importance both for the maturation of teeth and for caries prevention, and added in certain dental composites prevents secondary caries. We previously analyzed dental composites by PIXE and ERDA [1]. Aiming at a more simple approach, we examined the feasibility of simultaneously detecting fluorine by PIGE together with heavier elements by PIXE using a single low energy HP Ge detector, and we tested this technique on dental composites. Flat surface samples of the composites Ariston and Tetric Ceram (Ivoclar-Vivadent, Lichtensein), F2000 compomer (3M Dental, USA) and of three Romanian Restacril products were prepared by polymerization. An old Tetric dental filling from a patient tooth was studied also. Reference materials included Teflon, NaF and aluminium. The samples were irradiated at 45° with 3.0 MeV protons from a tandem van de Graaff accelerator and the PIXE+PIGE spectra were collected between 0 and 300 keV. The newly prepared composites evidenced mainly Ca, Zr, Ba, Yb and Hf by PIXE and F by PIGE. However, F was lost from the old Tetric dental filling. In addition, a γ line at ~171 keV was seen, produced by the proton bombardment of ^{23}Na (170.47 keV) and/or ^{27}Al (170.68 keV). In the composites, F and Al arise as YbF_3 and fluoroaluminosilicate glass. The PIGE detection limits were of ~0.2%, but they can be improved by a small increase in proton energy. The results proved highly relevant for analyzing the composites and their changes in the oral environment, and encourage the technique's further development.

1. E.A. Preoteasa *et al.*, *Nucl. Instrum. Meth. B* 189 (2002) 426.

PI-18 **Biology and biomedical sciences**

Measurement of heavy metals in canned foods

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The objective of this work is to determine the presence of heavy metal and their concentrations in canned food using the PIXE technique. The sample preparation consisted of drying the products (dehydration) either through the liofilization process and/or thermal treatment. After that, the materials were macerated and pressed into pellets. The products analyzed so far have been: tomato extract, milk cream, beer, tuna and sardine. Other packages for the same product have been analyzed as well. It was observed a greater concentration of Fe in the tomato extracts that remained longer in the cans. The canned milk cream also present greater concentration of Fe than the milk cream stored in cardboard box packing. In the case of the beers, the elementary concentrations do not vary with the packing, but they are different for each one of the analyzed trademarks. Samples of canned sardine and tuna had been analyzed, and the preliminary results indicate that the cans of both products of the three trademarks present traces of Cr in its internal coatings, while the cans of tuna also presented traces of Al. The differences of elementary concentrations between sardine and tuna vary with the trademark of the product. In general, it appears that there is no evidence of metal absorption by the materials stored in cans. The next step for the continuity of the project consists of measuring the major elements of the sample with the RBS technique in order to proceed with the quantitative analysis of the samples.

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PI-19 **Biology and biomedical sciences**

PIXE analysis of some medicinal plants usually extracted and drunk as tea, beverage, or used as spice or flavor in Nigeria.

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Some medicinal plants usually dried and extracted in cold or preferably hot water and drunk as a local tea or beverage, alone or in an admixture in particular ratios were analyzed using PIXE technique. Such preparations may be sweetened with honey, sugar or any sweetening agent. They are believed to serve usually as tonic in which case they can help in blood formation or as beverage like cocoa products. These medicinal plants include *Sorghum bicolor (L) Moench* Grammineae, *Harungana madagascariensis (Lam ex Poir)* Gutteferae (Dragon blood tree), *Curcuma longa (L) syn. Curcuma domestica Valeton* Zingiberaceae (Turmeric), *Zingiber officinale Roscoe* Zingiberaceae (Ginger), *Cymbopogon citrates (DC) Stapf* Grammineae (Lemon grass), *Harugana madagascariensis (Lam ex Poir)* Guttiferae, *Rumex acetosa*, and mixtures or combinations of samples. PIXE measurements were carried out using 1.8 MeV collimated proton beam from the 2.5 MeV AN 2000 Van de Graaff accelerator at Istituto Nazionale di Fisica Nucleare, Laboratori Nazionali di Legnaro, Padova, Italy. The results showed the presence of 20 different elements at different concentrations, three plants had traces of gold, and none of the plants contained any heavy toxic metals such as Pb, As, Cd, and Hg. The metabolic roles of the detected elements were discussed. The results of this novel study are presented and discussed.

PI-20 **Biology and biomedical sciences**

Elemental mapping of a post oak leaf using a proton microprobe

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Elemental distribution in a post oak leaf was measured using the Particle-Induced X-ray Emission (PIXE) technique and a proton microbeam at energy of 3 MeV and spatial resolution of 10 μm. The elements detected in this sample were Mg, Al, Si, P, S, Cl, K, Ca, Cr, Mn, Fe, Cu, Zn, Br, Rb, and Sr. Among them, spatial differences in the concentration of nine elements were observed between the vascular and mesophyll tissue. Si, Cl, K, and Ca were mostly concentrated in vascular tissue, while Mg, P, S, Cr, and Mn were for the most part concentrated in the mesophyll. The distribution of Ca appeared to follow cell wall contours. The distribution of some of these elements is compared to the function of the elements in living tissue and future possibilities for this type of investigation are discussed.

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PI-21 **Environmental sciences**

Microbeam analysis of yellow sand dust particles

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The microbeam system was applied to analysis of yellow sand dust particles. Yellow sand dust particles from the Asian continent sometimes cause turbid conditions in Japan, especially in spring. These particles are sometimes deformed by mixing with anthropogenic aerosols. For better understanding of the deformation mechanism, analysis of single particles is indispensable. For this purpose, we developed a microbeam analysis system with a spatial resolution better than 1 μm [1]. The system is composed of two X-ray Si(Li) detectors for PIXE analysis, an annular Si ion-implanted detector for RBS analysis, a Si ion-implanted detector for off-axis STIM and a Si-PIN photodiode for direct STIM. The combination of PIXE, RBS and off-axis STIM methods enabled simultaneous analysis for hydrogen to metal elements and revealed the chemical composition of these particles.
Yellow sand dust particles were impacted on a thin polycarbonate film and analyzed by using the microbeam system. After quantification of these particles, the chemical composition of each particle was obtained. Aluminum, silicon, calcium and oxygen are the main elements of the particles, originating mainly from soil dust of Alumino-Silicate particles. These particles contain sulfur and heavier elements and are deformed by mixing with anthropogenic aerosols. Sodium, magnesium and chlorine elements which come from marine aerosols are also included in these particles and may be absorbed during transportation from the Asian continent. Single particle analysis of yellow sand dust will lead to a better understanding of their deformation process during transportation.

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PI-22 **Environmental sciences**

The use of biomonitors and PIXE analysis in the study of air pollution in Mexico City

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An environmental study of air contamination in the Valley of Mexico is under investigation based on the use of biomonitors. The epiphyte lichen *Flavopunctelia flaventior*, which is rather sensitive to air pollution and has a great capability to absorb and accumulate airborne mineral elements, including heavy metals, was chosen as biomonitor in order to study trace element atmospheric pollution in an attempt to classify several locations according to their contamination level from airborne heavy metals accumulated in the lichen. The thalli of lichen samples were collected in a controlled area, La Marquesa National Park and subsequently transplanted to 13 points located in stations of the automatic network of atmospheric monitoring in the metropolitan area of the Valley of Mexico. This region includes urban and industrial places and one rural station. The study area is located 2240 m above sea level and covers a surface of 9,560Km^2, of which 1500 km^2 is urban with about 20 million inhabitants; it includes as well 3.5 million vehicles and 35,000 industries. The samples of transplanted lichen were exposed quarterly in different seasonal periods during the year from 2002 to 2004. Pellets were prepared from ashes of the exposed lichens. The contents of Cr, Cu, Fe, Ni, Mn, Pb, Zn and other trace elements were determined by PIXE using an external proton beam setup. This work presents the results of analysis of correlation factors between elements and main components using the Statistical Package for the Social Sciences (SPSS). The elemental distribution in the area of study and the environmental conditions are discussed.

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PI-23 **Environmental sciences**

PIXE and μ-PIXE analysis of biological records in environmental studies

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The experimental potentialities of the 3 MV Tandetron accelerator installed at CEDAD, University of Salento, Lecce, Italy have been improved by the installation of two new IBA (Ion Beam Analysis) beam lines: a PIXE-PIGE external beam set-up and a nuclear microprobe equipped with an Oxford Microbeam focusing triplet. We present the results obtained in the analyses of biological records and particulate air matter performed by external beam PIXE and in vacuum μ-PIXE. Dendrochronological dated tree rings sequences, were sampled, using an increment borer, from live Pinus Pinea trees, grown in two of the most industrialized areas in Southern Italy, the industrial districts of Brindisi and Taranto. The analyses allowed to reconstruct the temporal variations of the trace elements content and to relate them to known changes of the magnitude of the polluting emissions. The relationship between the trace elements content, the distance of trees from the polluting sources and climatoligical features such as the direction of the prevalent winds is also discussed. From the same area particulate air matter was also sampled and characterized, in vacuum, by μ-PIXE using 3 MeV protons and a Si(Li) Gresham x-ray detector. The compositional results of single particles allowed to identify the possible pollution sources and to obtain preliminary information about their relative magnitude.

PI-24 **Environmental sciences**

Effects on the elemental concentration in growth tree ring due to Popocatepetl volcano exhalations

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The Popocatepetl volcano located at 70 km South-east from the downtown of Mexico City has increased significantly its activity since 1991. Several exhalations of gases, dust and ashes have taken place reaching the Mexico Valley and the city of Puebla. Those events have affected the atmospheric conditions in the surroundings, producing changes in the composition of aerosols and having an impact on the environment of the whole region. Also, they have affected the ecological conditions in the soils.

Dendrochemistry studies of tree rings agree in the argument that the environmental factors and composition of the soil determine de elemental composition and the properties of the wood. It is expected that the effects of the changes mentioned above are recorder in the elemental composition of the growth tree ring [1-3].

In this work we present a study of the elemental concentration in tree rings from pines of the forest at Iztapopocatepetl National Park, in the region called "Paso de Cortés", located in the North Slope of the volcano few kilometers from the crater. For this study pines of the specie *Pinus montezumae* (Lamb. var. Lindleyi) were considered. Ten tree cores were extracted by a 5 mm diameter stainless steel Pressler drill and dried at 90^{o}C during 48 hours. The elemental concentrations were determined by PIXE using an external proton beam set-up. Each ring in the tree core was irradiated by a collimated 0.5 mm x 3 mm proton beam under a helium atmosphere. Only the rings corresponding to the last 30 years were analyzed in order to compare the last 15 years of volcanic activity with the prior 15 years without volcanic emissions. Changes in the concentration of the elements S, Mn, Fe, Cu and Zn were observed.

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PI-25 **Environmental sciences**

Relationship between soil composition and the distribution of three *Manfreda* (*Agavaceae*) in Mexico

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The *Manfreda* (A*gavaceae*) are well known herbaceous plants that growth along the Mexican territory. However the M. nanchititlensis is a specie endemic of the Temazcaltepec region located 130 km west of Mexico City and botanists do not have enough information about its growth conditions yet. As soil is one of the fundamental elements of the environment for vegetation support, this work presents a study of the soil characteristics at Temazcaltepec region in places where there are predominant presence of *M. nanchititlensis*. In order to make a comparison, the soil characteristics for other two species well known were determined too. The *M. scabra* and *M. maculate* were considered. The *M. scabra* has a wide distribution along the Mexican territory and it is a specie resistant to perturbed soils. The *M. maculta* is specie located mainly in the central part of Mexico and growth in altitudes from 1400 to 3000 m over the sea level.

The soils samples were collected in different places where each species are predominant. The National System of Soil Classification of the U.S. Department of Agriculture was considered to describe the soil taxonomy and profile. The pH and electric conductivity of the soils were measured using conventional methods. The composition of $Z > 13$ elements were measured by PIXE using 0.75 MV proton beam. Soils corresponding to the *M. nachititlensis* present low conductivity and the lowest concentration of Ca and Cr.

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PI-26 **Environmental sciences**

Uncertainty evaluation in quantities obtained from PIXE elemental analysis of atmospheric aerosols

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In most of the published studies dealing with the elemental analysis of atmospheric aerosols, carried out, for example, with Particle Induced X-ray Emission (PIXE) or X-ray Fluorescence (XRF), concentrations of the elements are combined to obtain further results about other quantities. Examples of this are the calculation of variables related to Soil, Sulfate, Non-Soil Potassium, or Organic Matter [1-3]. Many times the experimental uncertainty is overlooked in these new calculations, or there are even confusions regarding nomenclature. Although a careful description of the uncertainty evaluation in individual elements is given by Maenhaut [4], it dos not seem to be applied in all studies. Furthermore, the elemental concentrations used to compute the derived magnitudes are often correlated, as is the case of Al, Si, Ca, Ti, and Fe in the Soil variable. In this work, an evaluation of the experimental uncertainty of some of these quantities is presented, specially when there are correlated quantities, based on the ISO Guide for the Evaluation of Uncertainty [5]. Here, elemental concentrations in samples of PM_{10} and $PM_{2.5}$ collected in downtown Mexico City are used to illustrate the method, so it can be followed to evaluate straightforwardly the experimental uncertainty in this kind of studies.

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PI-27 **Environmental sciences**

Aerosols from inside an Alaskan forest fire via PIXE and PESA

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The warming of the Arctic has as a consequence increase in the frequency, severity, and duration of wildfires in the boreal forests, with release of stored terrestrial carbon reserves into the atmosphere. The Frostfire study was designed to examine the role of fire in mixed boreal forest/tundra land type via a prescribed fire east of Fairbanks, Alaska, summer, 1999. Aerosols were collected in 8 modes by a DRUM impactor in a battery powered fire shielded 10 m tower at a site within the prescribed fire zone. Samples were taken over an 18 hr period and analyzed in 40 min increments by PIXE and PESA, recording the passage of the flame front and the subsequent smolder phase. The standard IMPROVE algorithm was used to convert hydrogen by PESA into organic matter. The passage of the flame front in 3 hrs was characterize with by fine potassium and optically absorbing aerosols at 0.35 ± 0.2 μm aerodynamic diameter, with a potassium/organic ratio of 3%. There followed an extended smolder phase from the tundra of 7 hr with organic matter in the 0.75 ± 0.2 μm diameter and a potassium/organic matter ratio of 0.7% that represented over 2/3 of all the organic matter emitted by the fire. Little evidence of optically absorbing carbon was observed in this latter phase. These data help explain the anomalously high organic matter seen in Alaska each summer that is not assigned to fires via standard chemical mass balance signatures. The smolder phase organics are optically similar in size to sulfate aerosols seen at eastern US should have short wave cooling impact on the Earth's albedo.

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PI-28 **Environmental sciences**

Atmospheric levels and elemental composition of fine and coarse aerosols during wet and dry season campaigns at two sites in Tanzania

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Three aerosol sampling campaigns were conducted at two sites in Tanzania, first in the 2005 wet season (May-June), which was actually rather dry, then in the 2005 dry season (July-September), and finally in the 2006 wet season (March-May). The sites were at Dar es Salaam (a kerbside) and at Morogoro (a rural site; on the main campus of the university), about 200 km to the west of Dar es Salaam. Among the aerosol samplers was a Gent PM10 stacked filter unit sampler with sequential Nuclepore polycarbonate filters, providing coarse (2-10 μm diameter) and fine (<2 μm) size fractions. Depending upon the season and the location, either 24-hour collections or separate day-time and night-time samplings were performed. The Nuclepore filters were analysed for the particulate mass (PM) by weighing, for black carbon (BC) with a reflectance technique, and for up to 28 elements by PIXE [1]. The median levels of the PM10 PM for the 3 campaigns were 23, 45, and 13 μg/m^3 at Morogoro and 46, 58, and 40 μg/m^3 at Dar es Salaam. The average percentages of the PM10 mass in the fine size fraction during the 3 campaigns were 35, 49, and 37% at Morogoro and 31, 27, and 33% at Dar es Salaam. Most elements were predominantly associated with the coarse size fraction; notable exceptions were BC, S, K, and Pb, and at Dar es Salaam also V, suggesting that BC and these elements originated mainly from anthropogenic sources. Some typical anthropogenic elements, such as Zn and Pb, exhibited 20-70 times higher median PM10 levels at Dar es Salaam than at Morogoro. The high median levels for these elements at Dar es Salaam (e.g., 270 ng/m^3 for Zn and 113 ng/m^3 for Pb during the dry season campaign) are likely mainly due to traffic.

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PI-29 **Environmental sciences**

Representation and variability of the elemental mass size distributions of Antarctic coastal aerosol at Baia Terra Nova (Ross Sea)

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A multiannual experiment on Antarctic coastal aerosol is being performed during the summer season at the site Campo Icaro (Lat. 74º42'43"S, Long. 164º06'58"E). Size-segregated aerosol samples collected with an SDI 12-stage impactor are submitted to PIXE analysis and the size distribution of the mass concentration "EMSD" (ng m-3) of up to 9 elements, (with $Z \geq 11$) is obtained in the size range 12.÷0.047μm.

We present the partial results (11 samples) of the 2002/2003 campaign, which display a substantial reduction of the background level with respect to the published 1999/2000 results, and allow a successful representation of the EMSD's of 7 crucial elements (Na, Si, S, Cl, K, Ca, Fe) with one or two lognormal functions, in the full dimensional range, in almost all cases.

The scientific results include, for each individual sample: strong variability of a crustal component, cr, (identified by the strong correlation between Si and Fe and by their ratio); impressive stability of the submicrometric S (accumulation) mode; strong variability of the relative displacement of the EMSD's of Na and Cl, corresponding to a size-dependent Cl-depletion of the sea-salt component, ss; a strongly variable size-dependance of the difference, nss (non-sea-salt S), between S (total) and ssS (sea-salt S) in the supermicrometric region; the relative contribution of K and Ca to ss and cr; the detection of sub-micrometric modes.

Major involved scientific problems include: (a) strength of the fluxes to and from S and ss aerosol of gaseous S and Cl compounds; specific properties of gas-particle interactions; (b) amount of internal mixing between cr and ss components; its possible relevance for chemistry, optical aerosol properties and sea-air transport processes; (c) the interpretation of optical data concerning the interaction between solar radiation and aerosol (d) the connection between aerosol variability and variability of meteorological and environmental parameters.

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PI-30 **Environmental sciences**

PIXE-PIGE combined set-up applied to geochemical characterisation of ice dust and continental sediments.

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Geochemical characterisation of atmospheric dust particles deposited over the Antarctic ice sheet is extremely useful in recognizing the actual polar dust sources and, therefore, the present day and past atmospheric pathways. The climatic and environmental conditions experienced by source areas in different climatic regimes can be inferred by geochemical investigation on either ice dust and sediments (continental, fluvial or lacustrine) from the dust source areas.

The PIXE technique has proven to be a reliable tool for major and minor elements investigation of polar ice dust. This technique was applied for the direct measurement on filters of the insoluble dust fraction (after ice melting and without any other sample pre-treatment), with analytical detection limits less than 1 ppb.

By means of the combined use of PIXE and PIGE, particular attention was paid here to improve accuracy on quantitative determinations of lighter elements (mainly Na, Mg, Al and Si) in different sized materials. Results of measurements performed on size-selected Certified Mineral Standards (sampled as bulk – size up to 50 μm - and size selected < 5 μm), together with the first results obtained on the finer fraction (<5 μm) of PSA (Potential Source Area) samples, will be presented.

This approach, with the paleoclimatic information derived from the geochemical (isotopic, major and trace elements) analysis on polar dust being compared with that obtained from continental proxy records, will help in understanding source-related environmental changes and Earth's climate system dynamics.

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PI-31 **Environmental sciences**

Comparison of particulate matter morphology and composition of indoor and outdoor aerosols. Use of the analytical techniques EDS - SEM and PIXE.

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Air pollution in indoor and outdoor environments may constitute a serious risk for human health. The chemical and physical properties of particulate matter in indoor environments is influenced both by internal and external sources. It has been shown that in the current building design, walls are not effective in the removal or stopping of particles coming from the exterior. This aspect is very important if we consider that people spend most of the time indoors (an estimated 80%). The objective of the present work is to establish a comparison between levels of particulate matter with diameters of less than 10 micrometers (PM_{10}), between indoor and outdoor environments and its elemental compositions. Analysis of the differences between summer and winter season are also included. The sampling area was the South-eastern sector of Chihuahua City, Northern Mexico. Samplings periods covered the months of July 2005 to February 2006, using a high volume sampler for outdoor sampling and personal monitors for indoor environments. Scanning Electron Microscopy (SEM) was used for particle size analysis and Energy Dispersive Spectroscopy (EDS) was used for individual particle analysis. Correlation between concentrations of PM_{10} in both environments calculated with Pearson's coefficients was higher than 70 %. Seasonal differences in dimension and morphology of particulate matter were observed. The elemental analysis was performed with PIXE. The main elements considered for comparisons were Mn, Ti and Fe.

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PI-32 **Environmental sciences**

Intercomparison of aeolian dust elemental concentrations via PIXE and ICP-AES

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In this study we compare elemental concentrations of aeolian dust samples obtained via PIXE with elemental concentrations obtained via ICP-AES. Bulk dust samples were collected at Owens (dry) Lake, California during a sequence of three separate wind events in March 1993 along a 1.2km north-south transect. The samples were analyzed by Proton Induced X-ray Emission (PIXE) as solid pressed pellets and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) after digestion. PIXE analyses revealed the presence of 19 elements; additional trace elements were detected at ppm levels by ICP-AES. Al, K, Ti, Mn, Fe, Ni, Cu, Zn, As, and Sr were detected by both analytical techniques. Ba, Pb, Cr, Co, Mo and Cd were detected by ICP-AES only and Na, Si, Ca, S, Mg, Cl, Rb, Br, Ga were detected by PIXE only. PIXE revealed higher concentrations in most of the elements than ICP-AES. For instance, Al was at least 75% higher via PIXE then ICP-AES and for lower concentration elements such as As, PIXE was at least 27% higher than ICP-AES. Scatter plots and correlation factors depict the differences obtained from each method. The best correlations between the two methods were for Ti, Sr, Zn, and Mn with an R^2 of 0.71, 0.73, 0.79 and 0.86, respectively. Al, Ni, and K presented no correlation with R^2 = 0.007, 0.006, and 0.0018, respectively. As and Cu concentrations were predominantly higher in the third dust storm when analyzed by PIXE while Ni was predominantly higher in the third dust storms when analyzed by ICP-AES. Ni was detected by PIXE in 28% of the samples and 100% via ICP-AES. Outliers and variability may be explained by differences in the established detection limits for each analytical method, difference in analytical conditions, differences in sample preparation, and/or cross-contamination from sampling instruments.

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PI-33 **Earth sciences**

Identification of Saharian sand storms by PIXE from 1995 to 2006

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Since 1995 airborne particle samples have been collected in Portugal in the framework of various campaigns and monitoring programs. Although small changes in sampling strategy occur, the possibility of building up a 12 years long data series lead to preparing a fully compatible set, based on these originally dispar approaches. One of the most remarkable results attained as a consequence of this effort, is the identification of various Saharian dust episodes based exclusivelly on PIXE data analysis, including statistical data analysis. During the whole 12 years period, events showing a Saharian signature are present with a different annual distribution, and evidence can be found for a time dependente change in the tipical aerosol composition observed during these events. In a previous work it was seen that during this type of ocurrences, aerosol composition at Azores and near Lisbon showed very high resemblance, mainly if small time delays were introduced in data prior to comparison. This implies that it seems realistic to assume that the aerosol being deposited over the whole South Eastern area of the North Atlantic due to Saharian dust events, are being subject to a changing composition process, which may impact on the local ecosystem. In this work these results will be compared to conclusions present in the literature reated to the dynamics of airborne particles originated on the Sahara, and possible implications emerging from these comparisons will be discussed.

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PI-34 **Earth sciences**

Composition of mineral aerosols generated in the Salt Basin of Far West Texas (USA) using PIXE and complementary techniques

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The Salt Basin in Far west Texas, USA, is a site of active aeolian sand transport and dust emission. Salt Basin playas (dry lake deposits) are a major source of mineral aerosols in arid Southwest North America. These dust storms can be visible from satellite imagery, extend for large distances, and be pervasive for several days. The basin is immediately west of the Guadalupe and Delaware Mountains, which enclose a national park and an Interagency Monitoring of Protected Visual Environments (IMPROVE) aerosol monitoring station. Understanding the chemistry of Salt Basin aerosols and the influences that mineralogy, sedimentology, and hydrological cycles have on dust emissions from the Salt Basin would greatly improve our understanding of aeolian processes at work in this area and aerosol transport to protected ecosystems in the mountains. In this study we monitor seasonal, spatial, and temporal variations in the composition of the aerosols and wind-erodible sediments on the playa surface. Aeolian dust and sand sampling stations were positioned on the Salt Basin playa surface, nearby gypsum dunes, and at the crest of the Delaware Mountains, and have been actively collecting since June of 2005. Elemental analysis of the dust and source sediment samples was performed using Proton Induced X-ray Emission (PIXE), and additional compositional information was obtained through ion chromatography (IC) and X-ray diffraction (XRD). This study elucidates the effects of Salt Basin dunes and playas on regional dust storms and mineral aerosol transport to the adjacent uplands, and the impact of locally-generated dusts on aerosol measurements in the Guadalupe Mountains.

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PI-35 **Earth sciences**

Trace metal distribution in humic substances of wastewater irrigated soils of central Mexico.

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The Mezquital Valley in central Mexico has received wastewater from Mexico City for nearly 100 years. This practice brings in organic matter and nutrients but also trace metals that may represent a risk to consumers of products cultivated in these soils. There is little information about the fate of trace metals accumulated in these soils irrigated with wastewater for long time periods. Humic substances, the organic soil matter main components, are the responsible to retain and regulate the mobility of trace metals in soils. In this work, humic substances were extracted from soil and separated into its distinct fractions (humic acids, fulvic acids and humines). PIXE was applied to determine the metal association to each one of these fractions. In order to asses if the long term input of organic matter and metals modifies the metal association to the humic substances, parcels irrigated for three time periods (5, 47 and 89 years) were selected. It was observed that metals such as Zn, Cu and Pb are strongly associated to the humic acids. Humines retain mainly Rb, Zr and Pb while Ti is preferred by fulvic acids. Iron is present in all fractions. It was also observed that in general, humic substances form soil irrigated for long periods accumulate more trace metals.

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PI-36 **Earth sciences**

The Aznalcollar disaster: An in-depth PIXE study of the pirite mine spill of 1998

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In 1998 a contention wall from a pirite mine broke in Aznalcóllar (Sevilla, Spain). As a result, tons of toxic heavy metal enriched tailings and acidic water were spilled through agricultural soils and reached the Doñana Natural Park, covering a surface of 4.402 Ha and affecting the rivers Agrio, Guadiamar and Guadalquivir. The fact has been known since then as the "Aznalcollar Disaster".

Despite the remotion of the mud and other soil decontamination processes, residuous pollutants from the spill were still found in 2002, and the following of the Natural Park's recovery and its surroundings is generating many information in different science fields up to date. Many independent studies have been carried out to valutate the extent of the disaster since 1998. PIXE studies in precedent works have been done with scarce number of samples and/or extended to little areas.

In this paper we present the PIXE analysis of the samples that horticultural exploiting companies FRUTANSA and AFREXPORT had analysed after the disaster during 1998 to 1999 with techniques such as SEM/EDS, INAA or IPC covering the areas of the Finca de Guadiamar, Finca de Coto and Finca de Quema. The main pollutants found with PIXE have been lead and arsenic. There are some differences between the results obtained in the 1998-1999 analyses and the current PIXE results.

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PI-37 **Earth sciences**

Space weathering of extraterrestrial silicates simulated by nanosecond pulse UV excimer laser and PIXE checking of chemical modifications

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Laser irradiation experiments have been performed on powdered silicates (othopyroxene, clinopyroxene, and olivine) using a nanosecond pulse UV excimer laser (193 and 248 nm) to simulate the effects of space weathering induced on minor bodies of the Solar System by micrometeorite bombardment. We have used different fluences (from 0.05 to 2 J/cm2) to weather the samples, experimenting below and above the ablation threshold. All the irradiated materials have shown reddening and darkening of their UV–VIS-NIR reflectance spectra. In addition we have found that: (1) below ablation threshold, weathering effects increase with increasing number of laser pulses, and with increasing fluence, confirming that a thermal process is active; (2) above ablation threshold, weathering is much stronger and efficient than in the previous case, and is independent on the number of pulses. Some authors refer the modifications of the UV-VIS-NIR to the possible iron enrichment of the asteroidal surfaces.

All the sample have been analyzed by PIXE to check a possible chemical modification that could cause the reddening and darkening of the UV-VIS-NIR spectra. In our work we discuss two different way to explain space weathering: chemical modifications induced by micrometeorite collision and crystallographic defects induced by micrometeorite collision and solar wind.

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PI-38 **Earth sciences**

Matrix composition contribution to elemental concentrations measured by PIXE

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PIXE analysis of trace elements is widely applied in several fields of science and technology, like the enviromental sciences. To calculate the concentrations of elements by using GUELPH like codes it is necessary the knowledge of both, the matriz composition and its structure. PIXE analysis is not self consistent, in the sense that it does not give full information about the matrix composition, and consequently the structure, because it is not possible to measure light elements like carbon, oxigen, etc. In this work an additional use of X-ray diffraction was carried out to get information on the matrix structure related to its crystalline phases. Concentrations of elements of ocean sediment samples calculated with several matrices are shown.

PI-39 **Material science**

Traces elements present in Ecomaterial by PIXE

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A great problem faced by constantly developing cities is in the industrial sector because the different processes generate an important amount of inorganic solid waste. It is important to highlight the possibility of applying the ceramic process as a new alternative to incorporate inorganic industrial waste, which due to their metal content could be highly toxic in inert materials. Currently, many countries have prioritized matrix (cements, ceramics, glasses, glassceramics) development research that with proper processing can guarantee the blocking of inorganic industrial residues, which have to present chemical and thermodynamic stability, and have to be easily produced and manipulated. The feasibility of obtaining ceramic materials in the form of tiles out of sludge ashes from a treatment plant and residual sludge from the anodized process of water treatment has been studied as a way to reuse both residues and prevent their disposal. The studied systems SiO2-Al2O3-CaO, SiO2-Al2O3-MgO, and SiO2-Al2O3-Li2O using 30% of ashes in the three systems. Two series of tiles are prepared, the individual pieces of these materials were submitted to a thermal process of 1000 – 1100 °C/120 min. (sinterization) and 900 °C/240 min. (cristallization). In the first system, industrial grade Al2O3 is used along with aluminum salts residues. The starting ceramic has been studied by means of X Ray Diffraction Analysis (XRD), microstructural characterization by Scanning Electron Microscopy (SEM/EDX) and chemical composition applied Particle Induced X-Ray Emission (PIXE). Our results suggest that this industrial waste, can be used in the elaboration of ceramic material (tile) or Ecomaterials and is possible to applied PIXE to know the chemical composition of traces elements present in the inorganic solid waste.

PI-40 **Material science**

PIXE depth profile Ge in Si-Ge film using algorithm of maximum likelihood

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Silicon-germanium alloys concern to the best high-temperature materials of transformation of thermal energy in electrical. But their electrical and thermal characteristics strongly depend on a method of production. Method CVD for making of Si-Ge alloys is very perspective. It is known, that for such alloys high uniformity of allocation of germanium in silicon is characteristic. The film of alloy thickness more than 30 microns on a substrate from carbon has been made by this technique.

Method PIXE and the modified algorithm of maximum likelihood have been used for study of uniformity of allocation Ge in film of Si-Ge alloy. Examination was carried out on an analytical complex "Sokol" ISSPMST NSC KIPT. The beam of protons impinged on a normal line to a target. The detecting system consisted from Si(Li) the detector under a angle 135º in a direction of beam of protons. Energy of protons changed from 300 keV to 1.7 MeV that was equaled 3.3-35 μm for range of protons. The requirement of equality of thickness of layers between ranges of protons was satisfied at each change of energy. The current of beam achieved 300 nA. For verification of the program of calculation of a profile the measurements on the sample from pure germanium were made also. In the researched sample concentration of germanium was equaled 20.3 % that is close to settlement 20 % from technology of preparation of the sample. Such concentration was constant up to depth about 12 microns, and then decrease to concentration on a limit of detection follows.

PI-41 **Arts and archaeology**

PIXE analysis of artefacts from radiocarbon dated archaeological contexts

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The recent installation of a new, external beam PIXE-PIGE beam line on the 3 MV Tandetron accelerator of CEDAD, University of Salento, Lecce, Italy originally equipped with beam lines for in vacuum RBS-Channeling, ion implantation and AMS (Accelerator Mass Spectrometry) 14C dating, has significantly enhanced the experimental potentialities of the centre in particular in the fields of cultural heritage diagnostics. The new experimental set up, formed by an extraction nozzle with a 1.5 mm graphite collimator, a 8 μm thick Kapton® extraction window, an x-y-z sample handling and positioning system and the radiation detection system formed by two Canberra Si(Li) X-Ray detectors (30 and 80 mm2 active area) and a Ge detector for gamma rays, is described. In this paper we describe the results obtained in the non-destructive compositional characterisation of artefacts recovered from AMS-14C dated archaeological contexts. In particular we present the PIXE analyses of obsidian tools recovered in Neolithic sites in Southern Italy from contexts 14C dated to the 5th millennium BC, allowing to obtain information about the provenance of the raw material and to shade new light on the chronological evolution of the trading routes of raw materials in southern Italy prehistory. We also present the results of the PIXE compositional study performed on the wooden parts of the exceptional, and to some extent unique, wand attributed to the Roman emperor Massenzio, discovered in 2005, during an archaeological excavation in the centre of Rome carried out by the University of Rome, La Sapienza. The analyses allowed to obtain information about the presence of iron corrosion products on the wood and were used to select the portion of the wood samples more suitable to be submitted to 14C dating.

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PI-42 **Arts and archaeology**

Analysis of 19th century Mexican postage stamps by PIXE

Th. E. Gill

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Subtle variations in ink color and paper of postage stamps can affect their value and desirability to collectors and investors. PIXE has been utilized since the mid-1980s in studies of the ink and paper used for printing of postage stamps. Almquist (1985) utilized PIXE to analyze the color of blue to green lines on 1887 Mexican Numerals stamps, revealing color variations related to metal content of the inks. The 15-centavo Mexico Mail Transportation stamps of 1895- 1898 ("Mulitas") were printed in similarly varying colors from blue to green, and believed to be from the same ink type. External beam PIXE milliprobe was used to analyze the paper and ink of examples of the Mulitas stamps to investigate whether the same printing variations would be revealed. Strong differences between the two stamp designs and color shades were noted. The Mulitas papers were relatively devoid of the inorganic fillers and binders used in the Numerals paper. Unlike the Numerals stamps, the Mulitas ink was shown to be primarily organic (indicating an extremely early use of non-metallic ink in postage stamp printing) with minor amounts of metals including titanium, zinc, vanadium and lead. Color variations between greenish and bluish are related to zinc and titanium content, with more zinc in the first, bluish printing and more titanium in the last, greenish printing. Different batches of pigment appear to have been used for each printing, with color differences related to the presence of sulfur compounds and the addition of whitening agents.

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PI-43 **Arts and archaeology**

In situ PIXE analysis of pigments used in a Leonardesque painting

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During intensive scientific and art historical studies of a Leonardesque painting depicting the infants Christ and St. John, PIXE was used to explore two specific questions. The painting has been subject of speculation as to its relationship to Leonardo and his studio and the main aim of the research was to establish whether the picture could credibly be dated to the supposed period and, further, associated with Leonardo's workshop methods. While other analysis such as radiocarbon dating and studies of sampled pigments indicated that the painting could in fact be reasonably placed at the turn of the fifteenth to sixteenth centuries, two outstanding issues remained that were felt appropriate for application of PIXE, using the external microbeam to avoid the need for further sampling.

First, PIXE was used to study the trace element content of the lead white pigment present both as part of the painting and in an inscription on the reverse. The inscription was of special interest since it ties the painting to a key early seventeenth century collection inventory. Resulting data was compared to previous studies of trace elements in lead white using other techniques.

Second, it was noted that there were numerous traces on the painting conjectured to be highly degraded remains of a formerly green copper-based pigment. It was possible to show from PIXE analysis that this was probably the case and thereby propose a reconstruction of the likely original appearance of the painting.

This paper will summarise the outcome of this study and discuss the role of PIXE analysis in the project, concluding that non-invasive elemental analysis is only one aspect of the characterisation of pigment type and morphology and that the technique is most effective when used to answer specific questions as part of a suite of other investigations.

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PI-44 **Arts and archaeology**

External beam analysis of Roman glasses

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Ion beam analytical techniques can provide valuable information on structure and elemental composition of art or archaeological objects giving clues to conservation-restoration procedures, about provenance or manufacturing. An external beam facility is under development at ITN as an extension to the existing Nuclear Microbeam line, based on the OM 50 triplet quadrupole system. An extraction nozzle was designed and mounted downstream of the nuclear microprobe vacuum chamber and fitted with a 100 nm thick Si_3N_4 membrane of 1 mm^2 window area. A laser beam and a video camera are used for sample positioning at ~3 mm from the exit window. Although the working distance of the quadrupole triplet setup increases from the usual 16 cm to 43 cm, initial tests reveal that a beam spatial resolution of 70×75 μm^2 is easily attainable for external analysis, with a 1-3 nA proton beam current. At present, the beam spatial resolution improves to 65×60 μm^2 by flooding the region in front of the target and detectors with He. This provides microbeam capabilities to the external beam line as well as the ability to perform scans up to 800×800 μm^2 under control of the OMDAQ acquisition system. PIXE and RBS spectra are simultaneously collected using a Roentec SDD X-ray detector and a surface barrier particle detector.

The setup was used for the elemental composition analysis of a collection of Roman glasses. These glasses were recovered from an archaeological site of a Roman *villa*, in Amadora, that is believed to have suffered two different occupations between the 3rd and the 4th centuries AD. The analysis of fragments from contexts of both occupations intends to materially define these two moments. Due to lamination of the glass fragments' surface, these cannot be analysed in vacuum making the external beam analysis a better option for their study.

PI-45 **Arts and archaeology**

Scanning-PIXE analysis of ancient embroideries

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"Gold lace embroideries"of Middle Ages and Renaissance (using silk threads, spiral-wrapped by a sort of miniature golden tape), are unusual targets for PIXE analysis. Indeed, a major analytical problem derives from the spatial inhomogeneity of the material and from the "roughness" of the surface to be analysed. However, there exist interesting archaeometrical problems related to these fabrics: learning about the production techniques and the materials of the golden tape, discriminating original embroideries from later mends, taking decisions about restorations. A good approach to try and solve these problems - in a non-destructive manner - is that of using scanning PIXE in an external set-up. Indeed, we had recently the opportunity of investigating, with the scanning PIXE external set-up of the LABEC Laboratory in Florence, the materials of two embroideries produced with the above described technique: an embroidery after a cartoon by Raffaellino del Garbo (XV-XVI century), and a pillow-case, used after the death of St. Francis of Assisi to line a pillow believed to have belonged to him. For each of the two, several areas of the order of two square millimetres were scanned with a 3 MeV proton external beam of 20 micron size on target. List-mode acquisition was used and elemental maps were reconstructed in the examined areas. Selected sub-regions within each of the maps, once acknowledged as representative - also on the basis of a visual comparison with optical microscopy images - allowed us to extract the quantitative composition of the materials and to obtain some answers to the problems posed by the historians and by the restorers.

PI-46 **Arts and archaeology**

Provenance of Belgian Merovingian garnets By PIXE on IPNAS Cyclotron

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The archaeometry dedicated line of the CGR MeV cyclotron of the Institute of Nuclear and Atomic Physics and of Spectrometry (IPNAS) of the University of Liege has been improved in order to make matrix and trace analysis of cultural heritage artefacts.

Two HpGe Low Energy detectors have been set up around the extracted beam, one dedicated to the matrix analysis with helium flow between the target and the detector in order to detect low energy elements, one dedicated to trace analysis with appropriated filter. The extraction of the 3.1 MeV proton beam to atmospheric pressure is done with a nickel foil of 2.5 µm thick [1, 2] and the beam spot size on the sample is about 1 mm.

This set up has been used for the study of provenance of Belgian Merovingian garnets coming from the recent excavation of the Grez-Doiceau necropolis. This cemetery was constituted of 436 tombs with a period of occupation of almost two centuries (470-660 AD). About 450 garnets distributed on 60 objects have been found.

This paper aims to present these results in comparison with the same type of study made in France [3] in order to compare the experimental results and the archaeological conclusion, and in particular to determinate if commercial ways, concerning garnets, were identical in Merovingian Europe.

1. G.Weber et al., *Nucl. Instrum. Meth B* 139 (1998) 196-201.
2. G.Weber et al., *Nucl. Instrum. Meth B* 189 (2002) 350-355.
3. T. Calligaro et al. *Nucl. Instrum. Meth B* 189 (2002) 320-327.

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PI-47 **Arts and archaeology**

Non destructive study of gilded tumbaga artifacts from the Chichén-Itza cenote

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The studied artifacts come from the sacred Chichen-Itzá Cenote, one of the major Mayan archaeological zones. These artifacts are soles of sandals and other pieces of the costumes dressed by the ones sacrificed in this. The constituent material of the artifacts is tumbaga; a Cu-Au_Ag alloy, with a golden platting. In Center and South America the work of tumbaga reached a notable development, obtaining not only complex alloys like this one, but also surface finishing techniques like depletion gilding which, as it is known, makes possible to obtain a layer of almost pure gold. Since this technology was not developed in the Yucatan area, it is thought that these pieces were obtained by means of commercial interchange with Panama. When arriving at the conservation laboratory the artifacts showed heterogeneous surface; polished and rough, in different colors: copper, golden and black. This alteration was due not only to the long burial period, but to an inadequate method of extraction that included the use of a dragger, and an acid and mechanical "cleaning" method done in the 60's. Instead of it the artifacts conserved a large amount of gilding, atypical for pieces obtained by means of depletion gilding.

In order to identify the artifacts' constituent materials and production process, the pieces were analyzed by means of portable X-ray fluorescence (XRF), Particle Induce X-rays emission (PIXE) and Rutherford Backscattering (RBS), and those fragments isolated before the conservation processes were kept for its analysis with Scanning Electron Microscope with an electron microprobe (SEM-EDS). Thus, XRF was used at the conservation laboratory to determine the homogeneity of the gilding of all the artifacts and to get a mean surface composition. From this analysis, few artifacts were selected to carry out a more fine analysis by PIXE and RBS in order to measure the gilding thickness, the elemental depth profile of Au, Ag and Cu and to identify the gilding procedure. On the other hand, the microscopic studies were performed to refine the gilding thickness measurements, its homogeneity, the surface morphology and the crystalline structures of the corrosion layers. From the results, it could be verified that the constituent alloy of the support was a tumbaga, nevertheless the platting is a rich Au-Ag alloy. The measurements pointed out to an electrochemical deposition gilding technique instead of a depletion gilding method. The methodology applied was suitable for the characterization of the materials and the manufacturing of the artifacts. This research has been partically supported by CONACyT Mexico grant U49834-R.

PI-48 **Arts and archaeology**

PIXE and Ionoluminiscence for Mesoamerican jadeite characterization

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Green stone was highly appreciated in pre-Hispanic Mesoamerica. Most of the finest green artefacts were worked in jadeite and other minerals such as serpentine and nephrite. Jadeite is perhaps the most precious stone of the jade family in ancient Mesoamerica and it was widely used and traded from the Preclassic Horizon (1500 B.C.). In fact, there is only one known (and geologically possible) region source of jadeite in the Mesoamerican area. The main bed is located in the Maya region, in in the Motagua area of Guatemala and Honduras. In this work, some pieces of a collier of green stones from an offering discovered in an extensive excavation carried out in the palatial structure of Xalla (a project directed by L. Manzanilla) at Teotihuacan site, in the central highlands of Mexico, were studied using an external beam PIXE. The aim of this study is to determine the elemental composition of the green stones and to establish the sourcing, trade and relationship between these regions. The offering corresponds to the early Miccaotli period (around 155 A.D.) The collier is composed by eleven round pieces of green colors ranging from light to dark green. The analysis of three pieces was compared with several samples from Motagua sources of Guatemala and other green minerals commonly used in Mesoamerica. During the irradiation a green luminescence of high intensity was observed in the archaeological pieces but only in one case the luminescence was violet. When comparing with the mineral samples, only the jadeite source from Motagua region presented the green and violet luminescence. The corresponding spectra were obtained from an analysis of proton induced luminescence in vacuum on the Motagua samples. In this work, the elemental composition comparison of the archaeological items and the known mineral samples is presented. The luminiscence spectra, corresponding mainly to Mn emissions, are discussed. This research has been partically supported by CONACyT Mexico grant U49834-R.

PI-49 **Arts and archaeology**

Characterization of archaeological obsidians from Lagartero, Chiapas Mexico by PIXE

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Twenty obsidian samples collected at the Maya archaeological site of Lagartero, Chiapas, Mexico, were analyzed by PIXE and the data obtained was statistically treated to obtain principal component diagrams and dendrograms. The majority of obsidians came from Guatemala.

The archaeological site of Lagartero (Chiapas, Mexico) is a classic Maya site and it is the only one with particular ecological environment in the Upper Grijalva River Basin, presumably because it is surrounded by swamps. These cover an area of 8.6 km^2 of swiftly flowing streams and lakes (Lagos de Colon), fed by springs and the backed-up water of the Lagartero and San Lucas Rivers diverted by natural travertine barriers. The ceremonial architecture is mainly found at the Limonar Island, whereas the habitation area is located on the other smaller islands. The site has four principal pyramids that form a big plaza, a ball game, several interconnected platforms with structures on them and 160 archaeological mounds of different sizes. The twenty studied obsidians were collected in unit VII of the Limonar Island. Sixteen are size waste, four are fragments prismatic knife, 3 fragments of medial part and others polished fragment. Due to the geographical localization, close to the border between Mexico and Guatemala, this site is ideal for studying economical and social aspects of this historical time.

The study of obsidians provide information on the subsistence, population, social organization, cultural boundaries, trade networks, alliances and vision of the world of ancient civilizations and these information is useful for the basic tasks of reconstructing archaeological formation processes and for establishing chronological sequences.

PI-50 **Arts and archaeology**

PIXE analysis of obsidians from Teotihuacan

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A collection of about 50 archaeological obsidians studied in the framework of the *Ciudadela project* (Teotihuacan, Mexico) has been analysed using PIXE in external beam at the AGLAE facility (C2RMF, Paris) and at the Instituto de Física (UNAM, Mexico). To determine the provenance of these obsidian samples, the elemental composition derived from the PIXE spectra have been compared with data published in the literature [1] on the basis of instrumental neutron activation analysis (INAA). By looking at the concentrations of key elements (Na, K, Mn, Fe, Zn, Rb, Sr, Zr, Ba), it is possible to unambiguously assign the provenance of most samples. Many of them are originating from two major sources, namely Sierra de Pachuca I also known as Sierra de las Navajas (Hidalgo state) and Otumba (Mexico state), the main suppliers of obsidian exploited by Teotihuacans. However, some samples exhibit a compositional fingerprint matching other provenances, such as Zacualtipan (Hidalgo state) and Paredón (Puebla state). Special emphasis will be put on the data reduction strategy and statistical tools used to derive the provenance.

1. R. H. Cobean, *A world of obsidian*, INAH and Pittsburg University, 2002.

POSTER SESSION II

PII-1 Advances in experimental devices

DT2 a PIXE spectra simulation and fitting program

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Recently, a new PIXE code, LibCPIXE, was produced as a variant to the old DATTPIXE code but following a normally unused approach to PIXE data handling. In LibCPIXE, in opposition to most PIXE codes, PIXE set-up yields are simulated from first principles and detailed (including experimental) measurement of set-up facilities. This is the approach required to merge PIXE data handling code to other IBA data handling procedures, which many times require a knowledgeable user interacting with the computer in order to build a good model for the sample, mainly when facing complex samples. The merging of LibCPIXE and NDF (a well known code for handling RBS and other IBA methods data) showed afterwards that complex structures in the samples can also dramatically change the observed PIXE yields. A full stepwise process was then undertaken in order to produce a new PIXE data handling code that uses this approach not only to simulate the K and L alpha yields of the elements, in structured samples, but to fit the entire PIXE spectra. This approach is of particular importance for both the new high energy and high resolution PIXE set-up being installed at ITN (presented in another communication at this conference), and for the precise interpretation of PIXE spectra from complex targets. In this communication we present the new code and a set of examples where the program capacities are clarified.

PII-2 Advances in experimental devices

New high energy and high resolution Lisbon PIXE set-up

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Nowadays the PIXE technique is a well established method and few improvements have been reported during the last years. Recent improvements are related with technical developments rather than the technique itself. Among these there can be found the use of microbeams, higher solid angle detectors, Si(Li) detectors which are more closer to the physical ideal, some advances in data handling software and attempts to reach tomography analysis of inhomogeneous samples. Still, cross sections work has been nearly standing on the ECPSSR theory with small step improvements being made, as well as on the limitations of Si(Li) detectors, which lead to a limited exploitation of the electromagnetic spectra available. At ITN, a new approach was taken relative to this and a new PIXE set-up is being installed, which foresees important changes by extending the useful energy range of x-rays up to 120 keV and reducing the resolution at low and intermediate energies down to 1 and 0.5% respectively. This is achieved by making use of a CdTe detector that presents an energy efficiency platô up to roughly 70 keV, and a VeriCold Technologies GmbH POLARIS detector that presents a resolution better than 15 eV for the 1.486 keV of Al-K_α and better than 40 eV for the 10.550 keV of Pb-$L_\alpha 1$. In what concerns the first part of this two new approaches, the system is already working and applications are presented in other communications at this conference. Relative to the second part, natural delays have made that, at the time of the conference, the detector is still at the production site. Relative to this component, simulated PIXE spectra will be presented in this comunication which were produced by using the new DT2 code, also being presented in another comunication at this conference.

PII-3 Advances in experimental devices

Status report of Sasaki Taro memorial PIXE Center

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A PIXE center was established at Hakodate in 1995. Although the 3 MeV-cyclotron installed at the PIXE center had successfully been used for the PIXE analyses, we unfortunately met the cyclotron trouble due to not only an rf fault but also the deflector trouble to interrupt the research programs. In 2006 the PIXE center could restart the PIXE analyses as Sasaki Taro memorial PIXE Center (STPC). We are preparing to provide the beam from the cyclotron for multi-purpose use in various fields of research or commercial use in the next phase of STPC.

Design of the cyclotron has been described in [1]. The beam intensity of a 3 MeV proton is currently about 100 nA at the cyclotron exit. The beam transport system of the cyclotron consists of two beam lines for horizontal and vertical beam irradiations. A vacuum chamber for conventional PIXE analyses is installed at the end of the horizontal beam line while the vertical beam line is equipped with a two-dimensional scanning stage. The beam extracted from the vertical beam line into air through a piece of Kapton foil is delivered to the sample on the stage.

Details of the present status and new research programs of STPC will be presented at the PIXE 2007 Conference.

1. S. Wakasa et al., *Int. J. PIXE* 3 (1993) 329.

PII-4 Advances in experimental devices

External beam PIXE setup in Aarhus, Denmark

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A PIXE setup at a 5 MV van de Graaff accelerator in Aarhus has been supplemented by an external beam in air with diameter up to 2 mm. A Faraday cup in vacuum measures the intensity of a 4 mm diameter beam, which is further collimated to 2 or 1 mm diameter. The energy of protons was normally between 2 and 3 MeV, and with a 2.5 MeV beam the energy declined to 2.2 MeV after passing a thin Kapton foil (Polyimide) before entering the air region. For thick external targets the elemental composition is determined by the program GUPIX. As external targets 8 mm from the exit window we used thin foils for efficiency calibration and thick solid state targets for analysis. Application is made to geological and archaeological samples, including coins.

PII-5 Advances in experimental devices

Radiographic technique for densitometric studies using heavy ion microbeams

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Different analytical techniques are typically used to perform multi-elemental and densitometric analysis by means of particle beams with micrometric space resolution. Usually, those analyses are respectively performed by PIXE and STIM. Traditionally, to characterize the trace element concentrations in a specimen two different experiments are required with differences in setups and types of detectors employed, as well as in the necessary ion current intensities. In this work, we discuss the latest results in the development of a new technique that synthesizes both analyses in just a single one, by means of heavy ion induced x-ray emission. This technique, implemented for the first time at the Tandar Laboratory, employs a second target in addition to the sample under study. The multi-elemental information of the specimen is provided by its PIXE signal and its densitometric information is supplied by the PIXE signal of the secondary target, which is placed immediately behind the sample under analysis. These PIXE signals are produced and acquired during the same experiment, allowing the analysis of both features (composition and density) at the same time. The X-rays originated in the secondary target are attenuated when traversing the specimen in the direction of the detector and consequently a radiographic image of the specimen is obtained. In this case, the characteristic X-rays of the secondary target act like a monochromatic secondary source. In the present work, a method to estimate the thickness of specimens is introduced and compared with estimations performed by the STIM method.

PII-6 Complementary analytical techniques

Phase stability study of Y and Gd stabilized zirconia by luminescence methods

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Recent investigations on zirconia-based oxides for thermal insulating applications indicate significant thermal conductivity reductions with the combined addition of $YO_{1.5}$ and $GdO_{1.5}$. The problem of phase stability is important for these ceramics because of the phase de-stabilization of the initial metastable tetragonal phase, upon long-term high temperature exposure. In this study, the phase evolution of four selected compositions are analyzed with conventional microscopy methods and novel luminescence techniques, as a way to test the application of luminescent techniques on the phase characterization of zirconia compositions.

The ZrO_2-7.6mol%$YO_{1.5}$ (7YSZ) composition is the preferred material for thermal barrier coatings due to its long thermal cyclic life, but is limited to operating temperatures below 1200°C. Previous comparative studies of Y+Gd doped zirconia compositions have shown that relatively small additions of Gd to 7YSZ may improve the thermal stability of the single-doped composition. However, a better understanding on the underlying mechanisms for de-stabilization is needed to guide the design of these new compositions. Two selected compositions with Y and Gd are contrasted to their counterparts with Eu as a third dopant, to test the differences in the spectra with the phase changes. These samples are produced by reverse co-precipitation of precursor solutions, followed by pyrolisis, synthesis and subsequent heat treatments at 1350°C. The phase evolution is monitored by X-ray diffraction, microscopy, proton luminescence, PIXE and photoluminescence. A clear correlation between luminescence and phase changes is obtained from this work.

PII-7 Complementary analytical techniques

Characterization of natural and syntetic zeolites using ion beam analysis techniques

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Zeolites are very important materials in catalyst and in industrial processes. Natural and synthetic zeolites have a wide range of uses because of their good adsorption and their ion exchange capacity and catalytic properties. One of these materials, TAFF, a natural bentonitic material had been used successfully In the last 25 years, as catalyst in the preparation of organic molecules with biological activity. Mexico is an import source of natural zeolites, however their utilization in the natural form is limited due to the presence of impurity trace metals. For example metals such as vanadium and chromium inhibit the elimination of sulfur in hydrocarbons. Therefore it is important to know the composition of the zeolites and natural clays. In this work, we report the elemental characterization of synthetic and natural zeolites as well as clays using different ion beam techniques. A ^{3}He and ^{2}H beam were used to measure the major element concentrations (Si/Al, O, C) by RBS and NRA. PIXE was use to measure the total trace element content (V, Cr, Fe, Co, Ni, Zn Sr, As, Pb, etc). XRD and thermal analysis of these materials were also performed. Comparison of their composition and structure as well as their potential use is discussed.

PII-8 Complementary analytical techniques

Cleaning wastewater from ammonium with the mineral vermiculite –Using PIGE for nitrogen monitoring

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Solid samples were irradiated with a 4.2 MeV proton beam in a specially built chamber. The ion beam was extracted out of the cyclotron vacuum system through a 4 μm thick nickel foil. A helium gas flow of 10 cm3/min was used to avoid interference from the atmospheric nitrogen and oxygen. The emitted gamma radiation was measured with an HPGe coaxial detector located behind the chamber. The reaction 14N(p,p γ)14N was employed for the determination of nitrogen. The peak from the nickel foil at 1445 keV was used for normalization of the peak areas and a pressed pellet of KNO3 was used for nitrogen calibration. The method was evaluated using six different biological certified reference materials. A synthetic tobelite mineral was also used in the evaluation. The limit of detection for nitrogen in the mineral samples was about 1 mg/g.

This set-up was applied for the determination of the total amount of nitrogen in vermiculite. The mineral vermiculite was treated in different ways to optimise for the nitrogen uptake. The experiments were performed by doping samples in ammonium solutions and by analysing the dried and ground samples with PIGE. The aim of the study was to find a product that could be used for the removal of ammonium from wastewater.

PII-9 **Biology and biomedical sciences**

Tin (Sn) metal transport and its influence on essential elements in the yeast *Saccharomyces cerevisiae*

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Tin is not considered an essential metal micronutrient but cells do take up Sn^{2+} and suffer its potentially toxic effects. Moreover, homeostatic mechanisms are required to regulate its intracellular levels. Tin toxicity is ascribed to disturbances in the homeostasis of indispensable elements such as iron, zinc and copper. Therefore, the effects of dietary tin on S, P, Mg, Zn, Fe, Cu, K, Na, Cl and Ca metabolism were investigated by PIXE in the unicellular eukaryotic model *Saccharomyces cerevisiae.* Yeast cells were treated with 25 mM $SnCl_2$ for 1h and 2h and, 50 mM for 1h. The PIXE results show that tin induces changes in the metabolism of endogenous elements such as Zn, Mg, K and S (their levels were found to be significantly lower in Sn^{2+}-treated cells). For P, a significant intracellular increase were observed while the results for Ca, Fe and Cu were inconclusive. In particular, the quantification of Ca for Sn-treated cells was not possible because the K-lines of this element were shadowed by the Sn L-lines present in the cells. Possible oscillation of Cu could not be measured as Cu content was below the detection limit of PIXE (< LOD). In addition, we analysed some proteins that mediate the uptake of Zn, Cu and Fe and their putative involvement in the uptake of Sn. The possible influence of the metallo-regulatory transcription factors Zap and Aft1 on Sn-stimulated changes in Zn and Fe accumulation was also analysed. Yeast mutants *Δzrt2* and *Δfet4* that lack the low-affinity zinc and iron transporters Zrt2p and Fet4p, respectively, and the mutants *Δcrt2* and *Δcrt3* lacking the low-affinity copper transport mediating Crt2p and Crt3p, were more resistant to Sn^{2+}, indicating a possible involvement of these proteins in Sn^{2+} transport. Absence of zinc-responsive transcription activator, encoded by yeast gene *ZAP1* and of iron-responsive transcription activator, encoded by gene *AFT1* resulted in increased sensitivity to Sn^{2+}.

PII-10 **Biology and biomedical sciences**

An investigation of metal ions in the exoskeletons of wasps using nuclear microscopy

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Many insect exoskeletons are hardened at points subjected to wear by the presence of high concentrations of metal ions in the chitin polymer matrix. In some cases, such as the mandibles of leaf cutter ants [1], this can result in an organic material with a hardness comparable with that of iron, and for this reason the properties and synthetic pathways of these materials are being actively studied. Nuclear Microscopy (NM) using a scanned micrometre focused beam of 2 – 3 MeV protons is an ideal method for studying the localisation and concentration of metals in insects since the range of MeV ions is such that they will pass through the entire body of a dried insect of 'normal' size. In addition, the emitted x-rays from metallic elements are not strongly absorbed by the organic body matrix, allowing x-ray analysis coordinated with STIM imaging of internal structures.

The subjects of this study are various species of ichneumonid wasps (Hymenoptera) that are parasitoids which lay eggs on the larvae of other wood-boring insects which are then consumed. The female wasps have a long ovipositors which are used to penetrate several millimetres through the wood to reach their host larvae, using a variety of mechanisms, some of which involve breaking the wood fibres with consequent wear on the ovipositor's 'cutting' teeth. Electron probe studies indicate that manganese is present at the tip of the ovipositor, which may result in hardening of the tip to facilitate boring into the wood. This has been confirmed by NM analysis, which also reveals the 3-D elemental distributions within the ovipositor tip. We have also observed for the first time high levels of Zn in the tips of the claws, particularly in taxa known or suspected to have to penetrate hard wood including Rodrigama, Rhysella and Perithous.

1. Grime, G. W., E. Palsgard, et al. *International Journal of PIXE* 9 No. 3&4 (1999)199-216.

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PII-11 **Biology and biomedical sciences**

Application of micro-PIXE and dynamic analysis for the characterization of human hard tissues

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In recent years applications of PIXE in the bio-medical field have opened important areas of research with a significant sensitivity to compete with techniques such as XRF, EDXRF, EMP and/or SEM. The micro-PIXE technique is used to investigate the morphology as well as the elemental composition of human tissues on a microscopic scale. In addition, complementary ion beam techniques are used to provide information on the major and minor components. This paper will deal with an overview of the bio-medical projects at iThemba LABS particularly in relation to spatial distribution of trace metals in hard human tissues such as kidney stone concretions, teeth and hair, undertaken at the Nuclear Microprobe (NMP) facility. Relevant information about ion beam techniques used for material characterization will be discussed. Mapping correlation between different trace metals to extract information related to micro-regions composition will be illustrated with several examples using proton energies of 1.5 and 3.0 MeV and applied to: 1) Teeth erosion mechanisms; 2) Human kidney stone concretions nucleation region analysis; 3) Mapping of human hair cross sections from two different population groups.

PII-12 **Biology and biomedical sciences**

PIXE and PIGE identification of a dental composite from a dental filling and assessment of elemental changes associated to its oral use

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Dental composites brought substantial innovation in dentistry. However, they convey foreign elements in organism, they face chemical and mechanical degradation, and formation of secondary caries arises around the composite tooth filling. To reduce the latter, the new restorative materials release F^- ions. All these processes imply changes in elemental composition. PIXE and PIGE are well suited to analyze the dental composites and their changes. We applied these methods to identify and study such a biomaterial extracted surgically from an old dental filling of a patient's tooth. The removed composite was sectioned centrally, and its outer and inner surfaces were examined comparatively. Flat surface samples of the composites Ariston and Tetric Ceram (Ivoclar-Vivadent, Liechtensein), Valux Plus and F2000 (3M Dental, USA) and of five Romanian Restacril products were prepared and used for identification. The specimens were irradiated at 45° with 3.0 MeV protons from a tandem van de Graaff accelerator and PIXE spectra were collected with a HP Ge detector. PIGE spectra were recorded with the same proton energy and detector between 0 and 300 keV. The unknown composite evidenced Ca, Zr, Ba and Yb in proportions close to Tetric Ceram, with whom it was identified. However, Ba and Yb were partly lost, and Cl and K were accumulated from saliva. The PIGE spectrum showed a dramatic decrease of F. The results suggest the partial dissolution of composite's particles of YbF_3 and of Ba aluminofluorosilicate glass. The inner section was much alike to the outer surface, pointing to the diffusion of F^-, Cl^-, K^+, Yb^{3+} and Ba^{2+} ions deep inside the filling. Thus PIXE and PIGE demonstrated that changes occurred in the dental composite during its use not only at the surface but also in the bulk, providing thus valuable insight of the *in vivo* behavior of these biomaterials.

PII-13 **Biology and biomedical sciences**

An elemental analysis of Periphyton: A natural source of phosphorus in the wetlands of the Mayan region of Quintana Roo, Mexico

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Periphyton is a complex ecosystem composed by filamentous algae and an assemblage of heterotrophic microbes and other microorganisms that live attached to great submerged substrata in almost all aquatic ecosystems. The site where periphyton was collected corresponds to an ancient Mayan settlement recently discovered at the El Eden Ecological Reserve, located at the Northeast of the Yucatan peninsula in the State of Quintana Roo, Mexico. The elemental analysis of periphyton was carried out by the IBA techniques PIXE and NRA. Pellets of periphyton were bombarded with protons at 3 MeV in vacuum and non-vacuum conditions. The NRA technique was also used in order to determine the ^{16}O and ^{12}C composition, using a deuterium beam at 1.2 MeV. The results obtained from the periphyton analysis show the presence of 25 elements and give a similar patron in the concentration of the total macronutrients (Ca>P>S>K). Related to micronutrients, Cu and Zn were detected, but only Mn and Fe present a similar patron of concentration According to these results, we can conclude that periphyton plays a very important role in the biogeochemical cycling, due to the fact that it acts as a natural source of nutrients in the ecosystem, as well as an important indicator of water quality.

PII-14 **Biology and biomedical sciences**

PIXE analysis of some Nigerian pharmacological plants

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PIXE analyses of some Nigerian pharmacological plants were carried out. Two groups of plants were analyzed. These groups of plants are commonly used as medicinal plants in Nigeria for which pharmacopoeia standards are being established to enable their use for pharmaceutical purposes. These are *Jatropha curcas* Linn (Euphorbiaceae) – antimicrobial, *Bridelia ferruginea* Benth (Euphorbiaceae) – astringent and hypoglycemic agent, Mormordica charantia Linn (Cucurbitaceae), *Cassia occidentalis* Linn (Caesalpinaceae)- laxative, and *Chromoleana odorata* King and Robinson Linn (Compositae) antimicrobial. PIXE measurements were carried out using collimated proton beams produced by the 2. 5 MV AN 2000 accelerator at Istituto Nazionale di Fisica Nucleare (INFN), Laboratori Nazionali di Legnaro (LNL), Padova, Italy. Twenty elements were detected in the samples. However, *Jatropha curcas* (Ife) and *Cassia occidentalis* (Nsukka) contained traces of gold with concentrations of 33.7 ± 10.8 ppm and 76.3 ± 4.6 ppm respectively. Furthermore, the results showed that all the plants do not contain any toxic heavy metals such as Pb, As, Cd and Hg. Three plants *Jatropha curcas* (Nsukka) (4.6 ± 3.1 ppm), *Chromoleana odorata* (Ife) (11.6 ± 7.4 ppm) and *Cassia occidentalis* (Ife) (5.7 ± 4.7) showed detectable levels of selenium. The plant samples obtained from Zaria (Sudan Savannah) showed higher concentrations of elements compared to the other three locations Nsukka (Derived Savannah), Ile-Ife (Rain forest) and Jos (Guinea Savannah) from which plants were collected. The elements were more highly concentrated in the leaves than the stem and bark. The results give a good picture of the distribution of elements in the plants from different locations, with evidence that environment has effect on the constituents of the plants.

PII-15 **Biology and biomedical sciences**

Characterization of wines from Rio Grande do Sul, Brazil

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We are studying the elemental concentration of wines from different regions of Rio Grande do Sul. This state is the most important wine producer in Brazil and we are particulary focused in the production steming from Vale dos Vinhedos, a little mountain region of this state. Wines from others regions and others countries as Chile and Argentina were also analysed for comparison. For this study we chose Cabernet Sauvignon (vintage 2002). Soil, grape, leaves, bottle and cork were analysed as well. The analytical technique used in this work was Particle- Induced X-Ray Emission (PIXE). The respective concentrations were obtained by GUPIX software. For the matrix composition, we made use of the Rutherford Backscattering Spectrometry (RBS). Samples of wine were obtained by thermal treatment and the residues were compressed into pellets. Preliminary results for wines from a single vineyard showed that the concentration of iron in white wine is higher than in red wine, while other elemental concentrations did not present any substantial difference. The PIXE spectra consisted tipically of seventeen elements between sodium (Z = 11) and strontium (Z= 38). The matrix composition analysis revealed an amount of 32% of oxygen and 68% of carbon approximately. The analysis of the internal side of the cork showed a salient Si peak, while this result was not observed in wine spectra.

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PII-16 **Environmental sciences**

Micro-PIXE study of heavy metal uptake and transport of aquatic plant species

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In aquatic ecosystems water contamination by trace metals is one of the main types of pollution that may stress the biotic community. Although some metals are needed as micronutrients for autotrophic organisms, they can have toxic effects at higher concentration. Aquatic plants can take up large quantities of nutrients and metals from the environment, they can live under extreme environmental conditions therefore they are being increasingly used in phytoremediation processes.

Concentrations and distributions of major, micro and trace elements were determined within and along the roots of reed (Phragmaties australis), bulrush (Typha angustifolia) and sea club-rush (Bolboschoemus maritimus) using off-axis STIM and PIXE-PIXE ion beam analytical techniques on the Debrecen Ion Microprobe in order to reach a better understanding of the heavy metal uptake, transport and detoxification mechanisms of the plants,

The plants originated from the dried units of the wastewater sedimentation pond system of the tannery of Kunszentmárton, where 1500 m3 waste water containing lime, sodium-salts, ammonium-salts, chromium-salts, sodium, chlorine and magnesium ions, sulphur and organic material was released every day till 1988. The chosen species are the dominant species of the area, composing 85-90% of the green plant covering.

The essential macro-elements were found to be equally distributed along and within the roots, while most of the metals (Fe, Mn, Cu, Zn, Al, Ni, As, Ti, Sc, Hg and Cr) were found in much higher concentrations in the epidermis than in the inner tissues. Furthermore the uptake of the non-essential and the potentially toxic elements seems to be bound to the presence of iron plaques.

Ion microscopy study, providing elemental concentrations in a micrometer scale, proved to be a useful complementation to the usually applied bulk analytical techniques in understanding the elemental uptake and transport processes, and heavy metal resistance of these plant species.

PII-17 **Environmental sciences**

Trace metals in the sea grass *Thalassia Testudinum* from Mexican Caribbean coasts

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The impact of human activities on Mexican Caribbean zones is reflected in the partial or total destruction of these habitats and the increasing settlement of urban and tourist resorts. As a consequence, wastewater discharged to sea increase the levels of chemical elements and foreign substances present in the marine environment. In order to accomplish an adequate management and conservation of the coastal ecosystems is necessary to make and adequate diagnosis of the situation through factors that may monitor the vulnerability as well as the level of damage of the aquatic communities. In this work we present results on the use of the seagrass *Thalassia testudinum*, as a potential bioindicator of metal pollution in Caribbean Sea water. Seagrasses were collected in Holbox (considered as mildly influenced by human activities) and Puerto Morelos (considered as heavily influenced by human activities) in the State of Quintana Roo. Trace metals were determined by PIXE and ICP-MS. In order to get an insight of the trace metal levels of the seagrasses environment, coastal water was also analyzed by PIXE. Results are reported for metals such as Fe, Mn, Cu, Zn, Pb and Cd. The metal distribution on the different parts of the plant, the differences between two sites and the effect of season are discussed.

PII-18 **Environmental sciences**

Application of the PIXE technique to the study of marine coastal environments using fishes as bioindicators

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The coastal regions reflect, in its own characteristics, particular effects due to the convergence of material flow stemming from oceanic, atmospheric and earthly systems. Besides, these regions may suffer from additional anthropogenic influence depending on the human activities carried out in these places. If not controlled, human pressure gives rise to numerous negative impacts on natural resources of coastal ecosystems, like water quality, habitats lost in mangroves, sandy and rocky beaches, coral reefs and seaweed blooming, leading to a reduction of fishery resources that depend on coastal environments. The primary motivation of this work is obtain data in order to allow an evaluation of the anthropogenic influence in different coastal environments of São Paulo State, Brazil. The areas of study (continental shelf, Santos Bay, Santos esturary and a small esturarine system in the north coast of São Paulo) are exposed to different degrees of urbanization and other anthropogenic influences. Initially, this study was focused on two coastal and estuarine fish species (*Stellifer rastrifer* and *Atherinella brasiliensis*) and a crab (*Callinectes danae*) chosen as bioindicators of such ecosystems. Individual musculature was pooled, according to season and location, dried, pulverized and pressed into pellets. The samples were analyzed using RBS and PIXE techniques. Preliminary results show that significant metal concentrations were not observed in *Stellifer rastrifer* fish specimens of Santos Bay, reinforcing the role of the mangroves and sediment to retain them up river. Crabs from both Santos estuary and continental shelf presented elements like arsenic, although the role of this metal in the metabolism of these invertebrates is not well understood. The *Atherinella brasiliensis* specimens also presented some metals, but it is necessary to investigate their source since the area is far from major industries.

PII-19 **Environmental sciences**

Zinc profiles in archaeological and modern teeth

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Zinc elemental maps were made in archaeological tooth enamel to determine if zinc was affected by the post-mortem environment. Zinc is an important dietary trace element and deficiency is common in malnutrition and chronic illness. A permanent premolar from an Australian child who died in the second half of the 19th century was compared with contemporary premolar. Teeth were cut in the mid sagittal plane and were analysed using the Australian Nuclear Science and Technology Organisation (ANSTO) High Energy Heavy Ion Microprobe. Maps were produced showing the distribution of zinc across the enamel as well as linear profiles from the outside enamel to inside enamel. Results show the distribution of zinc in the archaeological tooth is quite different to the contemporary tooth, raising the suggestion that zinc has been significantly altered by the post mortem environment.

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PII-20 **Environmental sciences**

The role of PIXE within PATOS project, the first extensive field campaign for the aerosol characterisation in Tuscany (Italy)

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In most of the Tuscany communities the limit values for PM_{10} set by the EU are not fulfilled. As a consequence the Regional Government entrusted the extensive investigation PATOS (Particolato Atmosferico in TOScana), involving the University of Florence, the INFN and other research institutes.

PM_{10} daily samples have been collected in six sampling sites, representative of areas of different typology, from September 2005 to September 2006, by low volume sequential samplers; each sampler was equipped with two inlets so that aerosol can be simultaneously collected on Teflon and Quartz fibre filters. Samples collected on Teflon filters have been analysed by different techniques. Particle Induced X-ray Emission was used to measure the concentrations of all the elements with atomic number Z>10. The soluble component of inorganic ions was assessed by Ion Chromatography, while Atomic Absorption and Ion Coupled Plasma Mass Spectrometry were used to obtain the soluble component of several metals. Samples collected on Quartz fibre filters were analysed by Gas Chromatography and Gas Chromatography Mass Spectrometry to determine n-alkanes and PAHs concentrations. These samples were also used for total Carbon assessment. PM_{10} mass concentrations have been obtained gravimetrically.

During shorter periods, the fine and coarse fractions of the aerosol were also collected by streaker samplers. PIXE analysis of these samples gave the concentrations of all the elements with atomic number Z>10 with hourly resolution.

We will present some results which highlight the role of PIXE within the project.

PII-21 **Environmental sciences**

Aerosol source apportionment by Positive Matrix Factorisation applied to daily and hourly concentration datasets obtained by PIXE.

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Since aerosol particles retain elemental compositions characteristic of their origin, the simultaneous detection of groups of elements by multi-elemental techniques, like PIXE, can be of great help in the study of aerosol sources by receptor models. Among PIXE detectable elements there are markers of specific components such as marine aerosol (Na,Cl), mineral dust (Al, Si, Ca, Ti, Sr), sulphates (S), biomass burning products or biogenic emissions (K, Zn, Rb), heavy oil combustion (V, Ni), incinerator emissions (K, Zn, Pb), traffic and industrial emission (Mn, Ni, Cu, Zn, Pb).

Many studies have been devoted to aerosol source apportionment by receptor models applied to 24-hr averaged data. However, the impact of many aerosol sources can vary on a time scale of few hours or less. As a consequence, the use of hourly concentration datasets can be of great help for at least two reasons: the high resolved time patterns can help in source identification and more accurate source profiles can be obtained. Only few analytical techniques, like PIXE, can produce elemental concentration with hourly resolution.

In the framework of the PATOS project, the first extensive field campaign for the aerosol characterisation in Tuscany (Italy), PM_{10} daily samples have been collected in six sampling sites for one year (Sept.2005 – Sept.2006), simultaneously on Teflon and Quartz fibre filters, thus allowing the application of different analytical techniques (PIXE, IC, Total Carbon measurements, etc.). During shorter periods we also collected the fine and coarse fractions of the aerosol by a streaker sampler; PIXE analysis of these samples produced elemental concentrations with hourly resolution.

Results concerning the application of Positive Matrix Factorisation (PMF), a least squares formulation of factor analysis, to daily and hourly concentrations in the most polluted of the sampling sites will be shown.

PII-22 **Environmental sciences**

High airborne Chlorine in micro or sub-micrometer particles

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During standard analysis of airborne particle samples, a strange condition of extremely high deadtime was observed in a PM2.5 sample. The reason was promptly determined as an overwhelming amount of chlorine. Corresponding airborne concentration (which refers to a week average) was calculated to be of 14000 ng/m^3 of chlorine in particles of aerodynamic diameter below 2.5 μm. Since then, care has been taken to properly identify cases such as this. The study of a 12 years series of airborne samples shows that several similar chlorine concentrations above 1000 ng/m^3 did occur since 1995 in the Lisbon area or nearby, and maxima values show an average tendency for growing since 1995 up to present. Additional studies, including nuclear microprobe studies showed strange stuctures in the samples, namely strong inhomogeneities between chlorine containing and chlorine free areas. These analysis also showed that chlorine containing particles have micrometer or even sub-micrometer sizes, which makes that relative to human health impact, these particles should be seen as potencially more dangerous than chlorine. Elemental correlation studies nevertheless point to a marine signature that is not compatible to the estimated size of the particles, at least if sea salt spray is assumed to be the origin. XRD studies showed no NaCl signature, which corroborates the non-sea salt spray possibility. In this work, the results obtained by studing these samples in various ways will be presented and a few hypothesis for the particles origin will be discussed.

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PII-23 **Environmental sciences**

Seasonal variability in atmospheric aerosol levels and elemental composition during 2006 at Uccle, Belgium

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During 2006, a study was undertaken at Uccle, Belgium, to examine the relationship between the vertical column-integrated Aerosol Optical Depth (AOD) and the boundary layer aerosol characteristics. As part of this study, PM2.5 and PM10 aerosol samples were collected on 0.4 µm pore size Nuclepore polycarbonate filters. The collections were done during the daytime only and on days with no or few clouds when 50% or more valid AOD data were to be expected. A total of 109 collections were performed with each sampler. The Nuclepore filters were analyzed for the particulate mass (PM) by weighing, for black carbon (BC) with a reflectance technique, and for up to 29 elements (from Na to Pb) by PIXE [1]. Seasonal median atmospheric concentrations (and ranges) and seasonally averaged crustal enrichment factors (EFs, with Si as crustal reference element) were calculated. From examining these data, it appeared that (for both the PM2.5 and PM10 aerosol) the median atmospheric levels of the PM and BC were about a factor of two higher in winter than in the other three seasons. The medians for the crustal elements (Al, Si, Ca, Ti) were similar in all seasons; for the primary biogenic element P and the essentially anthropogenic element V the highest medians were noted in summer; and still other (mostly anthropogenic) elements, including K, Mn, Fe, Cu, Zn, and Pb, had highest medians in winter, followed by fall; the medians for Zn and Pb in fall were about a factor of 2 higher than those in summer. The crustal EFs showed the same seasonal patterns as the concentrations. Several variables were highly ($r > 0.75$) correlated with each other. This was for example the case between PM2.5 Pb and the PM2.5 levels of BC, K, Mn, Fe, Cu, and Zn.

1. W. Maenhaut, N. Raes, X. Chi, J. Cafmeyer, W. Wang, I. Salma, *X-Ray Spectrom*. 34 (2005) 290.

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PII-24 **Environmental sciences**

Detailed aerosol and elemental mass size distributions during winter and summer campaigns in Ghent, Belgium

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During 2004 winter, 2004 summer, and 2005 winter campaigns in Ghent, Belgium, 24-hour size-fractionated samples were collected with a 10-stage MOUDI and a 12-stage small deposit area low pressure impactor (SDI). The two devices were operated in parallel and between 30 and 35 samplings were made in each campaign. As impaction surfaces, aluminium foils were used in the MOUDI and Kimfol polycarbonate films in the SDI. The MOUDI samples were analysed for the particulate mass (PM) by weighing and the SDI samples were analysed for 27 elements (from Na to Pb) by PIXE. The average size distributions for the PM of winter and summer differed substantially from each other. In both seasons, two modes were present, but they had different relative intensities and the fine mode peaked at a different diameter. For the crustal elements (Al, Si, Ca, Ti, Fe), similar monomodal size distributions, which peaked at 3.3 μm aerodynamic diameter (AD), were obtained in both seasons. For several typical anthropogenic elements (e.g., S, V, Ni, Pb) or elements with an important anthropogenic contribution (K), two submicrometer modes (i.e., a condensation and droplet mode) and one coarse mode could be discerned. The condensation and droplet modes peaked at 0.3 and 0.7 μm AD. For V and Ni, which are attributable to residual oil burning, the condensation mode was by far the major mode in both seasons. For K, the size distributions of winter and summer were very different. Whereas during summer most of the K mass was present in the coarse mode, during winter, the condensation mode was the major mode, followed by the droplet mode. The large contribution of fine K during winter is attributed to biomass (wood) burning. In an earlier study, it was found that biomass burning can be quite important at Ghent during winter [1].

1. Z. Zdráhal, J. Oliveira, R. Vermeylen, M. Claeys, W. Maenhaut, *Environ. Sci. Technol*. 36 (2002) 747.

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PII-25 **Environmental sciences**

Comparison of Debrecen fine fraction aerosol data with others collected in some European collaboration

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Airborne particulate mass and its elemental composition are usually used for the qualification of local air at a given region. Atmospheric aerosol samples were collected in the yard of the Institute of Nuclear Research of the Hungarian Academy of Sciences from November 1996 twice a week with a two-stage "GENT" SFU (stacked filter unit) sampler. Coarse particles are between 2,5 and 10 micrometers in diameter and fine particles are less 2,5 micrometer. Airborne particulate masses (coarse and fine) were measured using a Sartorius microbalance. Elemental concentration of Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Br, Pb elements was determined by PIXE method. The black carbon (BC) content of fine aerosol, which is a particulate pollutant of aerosol emitted from the combustion of carbonaceous material, was determined by a Smokestain reflectometer.

Our data of the time interval from June 2000 to December 2001 were compared with others collected in some western- and middle European laboratories. Similarities and differences were found in the regional features, respectively, in the yearly average concentrations for silicon and sulphur elemental constituents and PM2.5. Silicon, sulphur and black carbon are considered as main representatives of the crustal material, traffic and long-range background pollution components, respectively. PM2.5 is a parameter accepted for measuring overall air quality from the point of view of human health conditions in a site.

PII-26 **Environmental sciences**

Time resolved elemetal component study of urban aerosol in Debrecen, Hungary

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With the use of accelerator based PIXE elemental analysis technique systematic investigation of aerosol samples have been performed in the Institute of Nuclear Research of the Hungarian Academy of Sciences for 15 years determining the elemental composition, size distribution, seasonal and long term time variation, sources and lung deposition probabilities of atmospheric aerosol characteristic to the east-Hungary region.

As a complementation of this research we observe the short-term time variation of the inorganic elemental components of fine (PM2.5) and coarse (PM10-2.5) urban aerosol at the end and the beginning of the heating season. Aerosol sampling campaigns are carried out with a PIXE International bimodal continuous streaker sampler, 2-stage samplers and PI cascade impactors in a downtown and an outskirt site of Debrecen. Parallel to the aerosol sampling meteorological parameters are also recorded with a micrometeorological station. PIXE analysis of the samples is made at the ATOMKI 5 MV VdG accelerator.

The short-time variation of urban aerosol combined with meteorological data serves as a basis to reach a better understanding of the aerosol sources and receptor areas, to select single episodes (like the appearance of Saharan dust in the region), to follow the evolution of aerosol, and to make a better estimate on the health impact.

PII-27 **Environmental sciences**

Elementary composition of particles $PM_{2.5}$ present urban areas of Baja California, Mexico

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The atmospheric dispersion in the state of Baja California can be conditional of factors as the proximity to the zone coast, Santa Ana winds on the Pacific Ocean in front of the Peninsula and type of climates in the state: characterized as dry and extremists with little annual precipitation, in this sense; the atmospheric contamination in Baja California at this moment the particle PM_{10} are being monitored by the government, nevertheless in the state has little knowledge referring to the Particulate Material smaller to 2,5 micrometers ($PM_{2.5}$), that according to diverse authors report adverse effects on the public health, mainly negative deficiencies in the pulmonary function and cardiovascular, as well as impacts in the environmental quality. The objective of this study is to know information relative to chemical, mineralogical and morphologic the composition of material $PM_{2.5}$ generated by diverse sources of emission: natural and anthropogenic, originating in urban zones of the state using a portable equipment of low volume. The morphology and elementary composition of the obtained samples were analyzed by Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM) and Particle Induced X-Ray Emission (PIXE).

PII-28 **Environmental sciences**

PIXE technique used for characterization of human exposure to mineral sand dust particles

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The aim of this study is to characterize the human exposure to airborne particles in a Brazilian region with natural high concentration of mineral sands. In this study, Proton Induced X ray Emission (PIXE) technique was used to characterize mineral dust particles in respirable fraction of aerosols, Plasma Desorption Mass Spectrometry (PDMS) was used to identify the Thorium compound in aerosols and excreta (urine) samples from inhabitant of village. Alpha spectrometry was used to determine concentration of radionuclides from ^{232}Th and ^{238}U series in aerosols and lichen samples.

The inhabitants were exposed to airborne particles containing Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Br, Sr, Zr, and Pb in fine fraction of aerosols, particles size were in the range of 0.6 to 2.0 μm. Ce, La, Y, ^{232}Th, ^{228}Th, ^{228}Ra, ^{238}U and ^{234}U were not identified in aerosol samples, showing that the inhabitant were not exposed to monazite particles during the sampling period and that there is another source of ^{226}Ra and ^{210}Pb, besides the mineral sands processing plant, in the village.

The main anthropogenic sources of particles are the mineral sands processing plant and the truck traffic; natural sources are the sea, soil and swamp. The results of lichen samples showed that during the last 15 years the inhabitants have been exposed to monazite particles. Excreta sample (urine) from an inhabitant living more then 40 years in the village was analyzed by PDMS technique, the mass spectra suggest that the inhabitant of the village has been exposure to mineral dust particles and they incorporate the metals present in mineral sands. In urine sample, Thorium was observed as NH_4Th. The Thorium and Rare earth compounds in urine sample from inhabitant indicate that there was a systemic incorporation of monazite.

PII-29 **Environmental sciences**

Analysis by PIXE of underground water from Ixtacxochitla, Puebla

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The *Sierra Negra* mountain range is located in the Southeast of Puebla, Mexico. This area is part of the Papaloapan basin, the third in importance in Mexico due to its water volume. According to some estimation the local annual precipitation is around 4000 mm. At the same time *Sierra Negra* is a very rich karstic zone. In the present work the contents of heavy metals in water in karst streams in the site of Ixtacxochitla, was investigated for its potential use as an indicator of pollution. In a preliminary study, four points were chosen for sampling; they were selected at different stratigraphic levels in the cave. Concentrations of the elements Fe, Ni, Cu, Zn, and Zr were determined using particle induced X-ray emission (PIXE). The amount of trace elements bound to each sampling point may give insight of its availability and geochemical dependence. Metal amounts quantified in this study remained below the critical values established by Mexican drinking water regulations, although Cu concentration appears to be high in one of the sites. The most relevant result of the analysis is that toxic elements in water cave samples were below detection limits.

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PII-30 **Environmental sciences**

Heavy metal, radionuclides, ben/z/ahyrene accumulation in soil of inhabited settlement nearby NSC KIPT

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The National Science Center "Kharkiv Institute of Physics & Technology" (NSC KIPT) is the largest research establishment of Ukraine. The structure of NSC KIPT includes five institutes, two scientific and technical complexes and a number of other organizations. The subjects of researches of the center cover practically all basic directions in solid-state physics, physics of plasma, nuclear physics, physics of accelerators etc. The researches are connected with the manufacturing of materials (beryllium, uranium, zirconium, GaAs, CdTe etc.) and products from them (a foil, hire, a wire, detectors). There are executed some works in which the radioactive installations are used and radioisotope are produced (accelerators, radioactive sources). The various methods are used for creation of coverings and products connected with formations waste products of benz/a/pyrene etc. It makes NSC KIPT by ecologically potentially dangerous object. The Center is located on industrial area in immediate neighborhood with which is placed inhabited settlement. This complex is surrounded from three sides by wood and from the fourth side it is limited to district road of Kharkiv.

Long-term observations of the content in soil of toxic elements, radionuclides, benz/a/pyrene are conducted in territory of settlement. The PIXE, PIGE and other methods of the analysis of a matter are used for the researches of ecological objects, first of all soil. Besides on the beam of protons in an atmosphere the element content of annual rings of a linden by age of 30 years was explored. In work the description of performance of examinations from sampling to analyses is presented.

Obtained data are analyzed in temporary gauge and by means of GIS technologies are developed maps of the content of various substances in soil inhabited settlement. Conclusion made about possible influence of functioning NSC KIPT on ecology of settlement.

PII-31 **Environmental sciences**

Development of sample preparation method for engine lubricating oil analysis using in-air PIXE

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Elemental particles (particle size: <10 nm) contained in automobile exhaust particles originated from the elements in the engine lubricating oil, and may become a nuclear of atmospheric particles. Therefore, investigation of elements in engine lubricating oil became our major consideration relating to chemical speciation of particles in the atmosphere. Since there was no adequate analytical method, we originally developed a preparation method of target sample for automobile engine lubricating oil (liquid sample) specifically designed for 5.1 MeV Helium in-air PIXE analysis. In the developed preparation method, target samples were fixed by making the oil sample sandwiches with 1% collodion solution based ethyl alcohol. With this analytical method, elements such as Mg Al, Si, P, S, Cl, Ca and Zn can be detected from the oil samples, where Mg, Si, P, S, Ca and Zn were the elemental components of the oil additives.

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PII-32 **Earth sciences**

Particle size/composition relationships of wind-eroding sediments, Owens Lake, California, USA

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Major (Na, Mg, Al, Si, S, K, Ca, Fe), minor (Cl, Ti, Mn, Sr), and trace (Ni, Cu, Zn, Ga, As, Br, and Rb) element concentrations were determined by PIXE in 119 bulk aeolian dust samples deposited at six heights in seven locations along a 1.2 km long transect during three sequential windstorms at Owens Lake, California, USA. All elements except Ni, Ga, and Br were detected in each sample. Na and S concentrations covaried with each other (and inversely with Si and Ca), increased with height, and decreased with distance downwind and time. Mg, Al, Si, K, Mn, Fe, and Sr concentrations in dust from northerly sites varied with height and location as opposed to nearly constant concentrations at southerly locations. Volumetric particle- size distribution (PSD) for each sample was determined via laser diffraction. PSDs reflected a trimodal distribution: 63% of the samples peaked at 20- 50 µm (silt), 11% at 50-100 µm (very fine sand) and 26% at 100-250 µm (fine sand). Most silty samples occurred during the first two events. Significant differences in element concentrations existed in relation to the volumetric percentage of particles in a given size range. For example, Na and S concentrations were proportional to the submicron to silt particle fraction during each event. Al, Ti, Mn, Fe, and Rb concentrations correlated positively to 100-500µm (fine / medium sand) particles in the first two events and a wider PSD range 250-1000µm (coarse sand) in the third event. The results suggest sodium sulfate aerosol emission during the first windstorm, while subsequent saltation-dominated events released more aluminosilicate minerals containing higher trace metal concentrations. These combined techniques reveal particle size/ chemical fractioning and small-scale spatial variability of sediments during resuspension at aeolian "hotspots," with implications to geochemical cycling and aerosol source/ receptor relationships.

PII-33 **Earth sciences**

PIXE Based Geochemical Characterization of the Pluvial Lake Palomas – Samalayuca Dunes Corridor System, Chihuahua, México

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Pleistocene Pluvial Lake Palomas is connected to the Samalayuca Dunes Field by an aeolian sediment corridor extending over 45 kilometers in an easterly direction in the central Chihuahuan Desert in northern Mexico. Mineral aerosols emplaced from these areas are responsible for constant air quality violations in northern Chihuahua, western Texas and far beyond, making it one of the principal sources of aeolian sediments in the Chihuahuan Desert. It elongates in a north to south direction for 200 kilometers, with an average of 25 to 30 kilometers in width. The Samalayuca Dunes are one of the most prominent ergs in North America. Excluding a large region of coppice dunes and a sand sheet, the Samalayuca dunes field extends in a northwest direction for 30 kilometers in length, reaching 13 kilometers at its widest point. The dune field is dominated by a north striking ridge of Draa dunes, with a height exceeding 100 meters above the surrounding dune forms: also present are combinations of barchanoid and transverse dunes. The connecting corridor system exhibits a complex and progressional series of geomorphic features. Following the sediment genesis from source to deposition sites, these features include: sediments deposited in the shoreline of the lake, barchanoid and transverse dunes, compound parabolic dunes and coppice dunes, all of these seated on, or surrounded by a large sand sheets. These sedimentomorphic progression and individual geomorphic features are analyzed on the basis of geochemical signatures. PIXE analysis for elemental compositions and ratios and X-ray diffraction for mineralogical composition are used as key techniques for documenting the geochemical evolution of the sediments traversing the corridor from Lake Palomas to the Samalayuca Dunes.

PII-34 **Earth sciences**

PIXE Based Geochemical Characterization of the Pluvial Lake Palomas System, Chihuahua, México

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The Pluvial Lake Palomas System is located in the Chihuahuan desert in northern Chihuahua, Mexico. This Pleistocene lake (12-15 Ka) inundated a surface close to 7800 Km^2 during the last glacial age. Pluvial Lake Palomas is presently composed of several perennial open and closed hydrologic basins. In the Mexican side these basins include: The Bolson de Los Muertos (largest), Laguna Santa Maria, Laguna El Fresnal, Laguna Guzman and Lake Palomas. Areas in the US side include the Indian Basin of Luna County, New Mexico. Mineral aerosols emplaced from these basins are responsible for constant air quality violations in northern Chihuahua, western Texas and far beyond, making it one of the principal sources of aeolian sediments in the Chihuahuan Desert. The sediment load deposited in these basins range from gravel and sand size sediments at the basins shorelines, to mixtures of finer silt, clay and sand particles towards the center of the basins. Pluvial Lake Palomas was originally inundated by the ancestral Rio Grande/Bravo, suggesting a semi-homogeneous sediment chemical composition. Later in time the Mimbres River joined the Rio Grande/Bravo in the inundation of the system. At present time the system (in individual basins) is inundated by the perennial Casas Grandes River (NW), the Santa Maria River (SW) and the Del Carmen River (S–SW). The input from these rivers and the presence of the flanking ranges contribute to create a distinct chemical composition of each basin's sediments in the system. Each basin is geochemically characterized using elemental compositions of lacustrine sediment samples determined by PIXE analysis. X-ray diffraction analyses are used to determine the mineralogical affinity of the samples to the surrounding lithologies. The resulting geochemical signatures will provide comparison parameters to aerosol samples collected downwind in northern Chihuahua, Southwest Texas and possibly as far as Canada.

PII-35 **Arts and archaeology**

Determination of elemental distributions in cherts using μ-PIXE

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In this work, a proton microbeam at energy of 3 MeV and spatial resolution of 10 μm was used to map the elemental distribution across a sample of the mineral chert obtained from a formation of archeological interest (Tecovis Flint, Texas, USA). The elements mapped in different areas of the sample were Al, Si, S, Cl, K, Ca, Ti, Mn, Fe and Cu. Other impurities were also detected: O, P, Zn and Sr.

The authors performed the mapping to ascertain the lateral distribution of impurities in a typical chert used by archaic and historical Native Americans in the southern plains of the United States of America to fabricate projectile points. The impurities present in the sample are thought to be responsible for quenching the fluorescence and absorbing the UV and fluorescent emission in localized spots. The distribution of impurities detected does correlate with local quenching and absorption of the fluorescence emission.

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PII-36 **Arts and archaeology**

PIXE and the anthropic circulation of obsidian during the Neolithic of central and western Mediterranean

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Obsidian, a glassy volcanic rock used as a raw material of the prehistoric lithic industry is often found in Neolithic sites far away from its geological occurrences. Coupled studies of 'archaeological' obsidians provenances -based on elemental composition- and of their typo-technology constitute a powerful tool in view of the reconstitution of '*chaînes opératoires*' and of past 'obsidian roads'.

In the frame of a regional project, more than 300 geological and Neolithic obsidians from central and western Mediterranean were analysed by PIXE, either at C2RMF with the AGLAE extracted beam facility or at CENBG with a 4 MV Van de Graaff and with the new AIFIRA facility. Elements Na, Al, Si, K, Ca, Ti, Mn, Fe, Zn, Ga, Rb, Sr, Zr, were always quantitatively determined as Y and Nb on AGLAE and AIFIRA. Data consistency was secured by the use in each sample batch of one reference obsidian (ARC-URS, a Monte Arci type SA Sardinian obsidian). Element contents were calculated from the 2000 version of the GUPIX software.

The geological samples were prepared as polished section. The agreement on element contents determinations between the three PIXE facilities used is better than 10%. Major element contents are usually in agreement with electron microprobe (EMP-WDS) data to better than 5% for Na, Al, Si, 10% for Ca and 20% for K, Ti, Mn, and Fe. For trace elements the agreement with conventional (solution) ICP-MS measurements is generally better than 15%. It is shown that obsidians from the source-islands of Lipari, Palmarola, Pantelleria and Sardinia can easily be distinguished, as obsidians from the several sources of Pantelleria and Sardinia.

The archaeological obsidians were treated non-destructively. All could be sourced from their elemental compositions. Combined provenance/typo-technological studies show that from the early Neolithic ancient men selected specific obsidian types according to the expected final product.

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PII-37 **Arts and archaeology**

Provenance Studies by PIXE of Obsidians from Laguna de los Cerros, Veracruz, Mexico.

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Obsidian is a natural volcanic glass widely used to produce artifacts in pre-Hispanic cultures of Mexico since Early Preclassic period (1500-1000 B.C.) up to the first part of the Spanish colony in XVI century. This material was highly appreciated and finely worked. Obsidians were extracted from various volcanic sources, then worked to obtain smaller fragments before trading. On the other hand, Laguna de los Cerros is one of the most important sites of the region of the Gulf of Mexico (today Veracruz state) during the Preclassic and Classic Horizons. In several archaeological excavations corresponding to different periods of Laguna de los Cerros, obsidians artifacts and fragments have been found. PIXE is an outsantding and quick method for non destructive studies and provenance of obsidians. The aim of this research is to establish the volcanic source exploitation in the region and the exchange pattern during the Preclassic and Classic horizons in Laguna de los Cerros. Then, after a selection by archaeologists, about 100 fragments and small artifacts of obsidians from the Laguna de los Cerros excavations and samples from known volcanic sources were analyzed without sampling by a 3 MeV proton beam using the external beam setup of the Pelletron accelerator of the IFUNAM. The PIXE results of major and trace elements were compared with data obtained by X-ray fluorescence and neutron activation analysis for various obsidian sources of the Veracruz and Puebla regions and central Mexico highlands. From principal components statistical analysis, a correlation was established between the excavated items and the obsidian sources of the region and other foreing provenances, being Mn contents a discriminant element for the local sourcing.

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PII-38 **Arts and archaeology**

Long distance transport of Neolithic variscite ornaments along the European Atlantic arc demonstrated by PIXE analysis

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The large grave mounds from the Carnac region in Brittany are among the most impressive funeral architecture of the Neolithic period in France. The exceptional quality of the adornments deposited in burials, such as polished stones axes and beads necklaces probably reflects the high status of the buried persons. Despite the high importance of these artefacts, already pointed out by archaeologists during the early excavations of the 19th century, their origin has never been really established on the basis of objective criteria. This study focus on the beads, green in majority, similar to the turquoise colour. A combination of techno-typological study and chemical analysis by external beam PIXE has been performed on these archaeological artefacts. The composition in major elements gives the mineralogical nature of the material which turns out to be variscite, a hydrated aluminium phosphate. By comparison with the composition of variscite reference samples from various geological sources of Western Europe, the concentrations in trace elements allowed us to establish that the beads were originating from the Iberian Peninsula. The presence of these Iberian ornamental elements in archaeological sites in Western France thus reveals long distance relations between the Neolithic human groups during the fifth to the second millennium BC.

PII-39 **Arts and archaeology**

TTPIXE analysis of roman coins from Ilipa (II-I B.C.)

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The non destructive analysis given by TTPIXE has shown to be very useful for the multielemental characterisation of valuable objects. A set of roman bronze coins from Ilipa (Alcalá del Río, Sevilla, Spain) and dated on II-I B.C., has been analysed in the Centro Nacional de Aceleradores de Sevilla, Spain, using 3 MeV protons in our external beam line [1]. Ilipa was an important city-mint from the South of the Iberic Peninsula and its numismatic interrelation with other coetaneous cities-mint of this area (Carmo (Carmona, Sevilla), Obulco (Porcuna, Jaén) or Castulo (Linares, Jaén)) is of great historic interest.

In spite of the surface concentration alterations by corroded patinas, that can be advoided in some cases by combining the PIXE results with measurements of gamma-ray transmission [2], we have compared the present results with analysis of other coins coming from relevant contemporary mints. This can allow us to find representative »fingerprint« of the different mints. Moreover, some systematic differences found in the composition of both sides of most of the analysed coins could also give us some information about the coinage procedure.

1. M. A. Ontalba, et al., *Nucl. Instr. and Meth.* B 181 (2001) 664-669.
2. M. A. Respaldiza, et al., *Nucl. Instr. and Meth.* B 50 (1990) 226.

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PII-40 **Arts and archaeology**

Micro-SR-XRF and micro-PIXE studies for archaeological gold identification – The case of Carpathian (Transylvanian) gold

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Trace elements are more significant for provenancing archaeological metallic artifacts than the main components. For gold, the most promising elements are Platinum Group Elements (PGE), Sn, Te, Sb, Hg, Pb, but also high melting point elements, such as Ta and Nb. Several small fragments of natural Carpathian gold - placer and primary - were studied using Synchrotron Radiation X-Ray Fluorescence (SR-XRF) at BESSY synchrotron. The goal of the study was to identify the trace elements, especially Sn, Sb and Te. The measurements were performed in air by using a 34 keV beam to excite the characteristic X-lines in Sn-Sb-Te region. We found Sn to be present in placers from Valea Arieşului and Valea Pianului, Sb in primary gold from Zlatna, Ruda-Brad, Valea Morii, Runculeţ-Straja and Pb in primary gold from Brădişor-Brad, Zlatna, Runculeţ-Straja, Valea Morii, Muşariu-Brad. These results are consistent with the geological data for Brad-Zlatna region. Micro-PIXE is a similar method, but uses protons instead of photons for characteristic X-rays excitation. Two native gold nuggets and several fragments of objects coming from Visigothic Pietroasa "The Golden Brood Hen with Its Chickens" hoard were analyzed using micro-PIXE technique at the AGLAE accelerator, Louvre Museum, Paris and at the Legnaro AN2000 microbeam facility. Using a 75 microns Cu filter to minimize the Au lines, we found Te in primary gold from Brădişor-Brad, Muşariu-Brad (different samples from BESSY analyzed ones), and Roşia Montană, in primary gold at Bucium-Izbiţa. For Pietroasa hoard, we found Sn in the Oenochoe cup and small fibula, indicating that alluvial gold - probably from Anatolia (Pactolus river) - was used. We also detected Ta inclusions in the large fibula, indicating that Ural Mountains (the only region where Ta and Au minerals are together) gold was (at least partially) used. No Te, Sb or Pb traces were detected, indicating that Carpathian gold was not used to manufacture these artifacts.

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PII-41 **Arts and archaeology**

PIXE analysis of a 12th century stained-glass window

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A 12th-century stained-glass window panel removed in 1847 from the Saint-Denis basilica near Paris, France, has been analysed using a combination of PIXE, PIGE and RBS methods carried out *in situ* with external beams of protons and He ions. The aim of studying such a well preserved panel is to give reference composition of stained glass and *grisaille* of the 12th century period in the Paris region, in view to address three issues: 1 - to understand the alteration process dramatically affecting the conservation of the other panels removed only recently from the choir of the basilica, 2 - to improve our knowledge in art history and ancient technology and 3 - to inter-calibrate complementary analytical techniques such as X-ray fluorescence or Raman spectrometry. The results obtained allowed us to define alteration markers, notably affecting the alkali elements (sodium and potassium) and lead. By combining the identification of various chemical types of glass yielded by PIXE with a visual examination of their surface under the guidance on the conservator, we have been able to refine the question of authenticity of the panel in discriminating the genuine parts from the restored ones. Our data complete those published in the past by the *Corpus Vitrearum Medii Aevi*. Technological markers have been evidenced such as, for instance, a specific chemical fingerprint Sb/Fe/Cu/Co for the blue soda-lime glass that can be compared with the famous blue stained-glass from the Chartres cathedral.

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PII-42 **Arts and archaeology**

Non destructive analysis of daguerrotypes by simultaneous PIXE-RBS

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Daguerotypes were invented by J. Daguerre in early XIX century to produce a photographic image on metallic plates. This work presents a first characterization of four anonymous daguerrotypes on copper plates produced between 1839 until the early 1860's. They are part of the collection of The Detroit Institute of Arts (DIA). The aim of this work is to analyze the surface composition and get reliable information to understand the chemical processes that lead to image fading. The analysis was carried out using the external beam setup of the Pelletron Laboratory of the IFUNAM. Particle Induced X-ray Emission (PIXE) and Rutherford Backscattering spectrometry were performed simultaneously by a 3 MeV proton beam using and external beam setup.

Results of the PIXE analysis showed expected major elements such as copper and silver while gold, lead and mercury were detected as minor elements. Concerning the RBS results, it was possible to measure the silver photographic layer thicknesses. The measured thicknesses ranged from 7 to 14 μm. Two of the plates had similar thicknesses and they may have a common origin. Daguerreotypes were irradiated in visually distinctive areas; for example in the dark shadows, blue tints and highlights, however, no significant differences in the elemental composition were found.

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PII-43 **Arts and archaeology**

The Grolier Codex: A PIXE & RBS study of the possible Maya document

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The Grolier Codex has been a controverted document ever since its late discovery in 1965. Because of its rare iconographical content and its provenience from non-authorized archaeology, specialists are not keen to assure its authenticity that would set it amongst the other three known Maya codes in the world (Dresden Codex, Paris Codex and Madrid Codex).

The document that has been kept in the Biblioteca Nacional de Antropología e Historia in Mexico City, ever since its exposure in 1971 at the Grolier Club of New York, is being now analysed by a set of non-destructive techniques in order to characterise its forming materials including paper, preparation layer for painting and pigments. PIXE and RBS have been the first techniques applied for this purpose. Measurements have been carried out at the 3MV Pelletron Particle Accelerator of the Instituto de Física (Universidad Nacional Autónoma de México).

This paper discusses the results obtained for PIXE and RBS, the aim of which is to ensure the materials in the Grolier Codex match those found for other prehispanic documents and discard the presence of modern pigments that would appoint the document as a fake.

The preparation layer for painting shows the presence of $CaSO_4$, colour red is due to red hematite (Fe_2O_3) and black is a carbon-based ink (C). However, the presence of Maya Blue in the blue pigment cannot be assured.

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PII-44 **Arts and archaeology**

Study of damage induced by ion beam in white pigments

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Pigments and paint layers are known to be sensitive to particle irradiation. Occasionally, the analysis of painting by PIXE can induce a slight or dark stain according to the irradiation conditions. In order to understand the damage formation, we have irradiated white pigments (lead white, basic lead sulphate, calcium sulphate, gypsum, calcite, zinc oxide, titanium oxide and lithopone) with the external 3 MeV proton micro-beam used for PIXE experiments. We have observed various sensitivities according to the pigment; for the majority of compounds, the damage is proportional to the beam current and the charge, but for other compounds the behaviour is different. For example, no visible change occurs for calcite but, on the other hand, lead white pigments seem to be very sensitive. Damages induced by the proton beam were studied by micro-Raman spectrometry. Structure modifications, such as dehydration, were detected, specially for lead compounds. The damage recovery was investigated by heating and UV-light irradiation. Damage has disappeared under UV after one week of illumination, showing that PIXE experiment can be safely undertaken for pigments.

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PII-45 **Arts and archaeology**

PIXE analysis of trace elements in middle age human and animal bones

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Human and animal bones from the deserted medieval village of Apigliano, southern Italy were submitted to in air PIXE analyses at the external beam IBA line at CEDAD, University of Salento, Lecce, Italy. The samples were recovered in two dinstict contexts: the first one dated, on the base of archaeological considerations, to the 8th-10th century AD and the second to the 13th-14th century AD. All the studied samples were characterise by FTIR (Fourier Transform Infrered) analyses both in ATR (Attenuated Total Refletion) and transmission mode in order to check the preservation status of both the organic and the inorganic bone fraction. In particular, only the samples attributed to the first occupation phase showed a preservation status of the collagen good enough to allow AMS 14C measurements, which confirmed the archaeological dating to the 8th-10th century AD, while the samples attributed to the second phase showed a high degree of diagenesis. With the aim to try to identify the possible origin of this difference between the two sets of samples, recovered from very close areas, they were crushed to powder, pressed in pellets and analysed by in-air PIXE. The PIXE analyses confirmed the different feautures of the two set of samples in terms of both major (Ca and Sr) and trace elements.

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PII-46 **Arts and archaeology**

Proton Beam Characterization of Bone Remains from the Middle Mesoamerican Formative

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In this work, bone remains of ten individuals from a multiple burial discovered in Tixtla, Guerrero, Mexico, were studied using ion beam techniques. The chronology of the burial corresponds to the Middle Formative Horizon (1200 to 400 BC). The spatial distribution of the bones and their conservation conditions indicated that the bone remains were buried at different times in the tomb.

Since collagen contents decrease as a function of the age, the bone antiquity may be determined from the N/Ca ratios from Ca and N measurements by simultaneous Proton Induced X-rays Emission (PIXE) and Rutherford Backscattering Spectrometry (RBS) using the $^{14}N(p,p)^{14}$ N resonance at 4 MeV. Al and Fe concentrations, among others, were measured by PIXE using an external beam setup to determine bone alterations and possible diagenesis. For these purposes, complementary analyses by luminiscence induced by 3 MeV protons beam were carried out as well. The aim of this study is to measure the bone elemental composition and the effects of the deterioration processes on the dating results.

PIXE and RBS results pointed out to three groups of N/Ca contents with an estimated antiquity of 3050 to 3380 years. This result agrees with the archaeological age (3005-2409 years). Besides heterogeneous alterations have been observed in the bone remains from metallic elements contents, it was possible to estimate the bone antiquity from this residual collagen approach. Bone deteriorations are discussed.

Notes

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