

Health and Environmental Studies using Ion Beam Techniques

Introduction

The Biomedical and Environmental applications undertaken by the PIXE group at the ITN Van de Graaff Laboratory, are based on scientific research and development projects, although the technical support for both internal and external demands can not be disregarded. The collaborations established with University groups allow both research on Basic Sciences under the scope of Ion Beam Techniques and training activities under the faculty graduation programmes.

During the year of 1997, important developments in the PIGE (Particle Induced X-ray Emission) technique and in the Proton Microprobe facility were achieved at the Ion Beam Laboratory. Important contributions from both techniques, are expected to the PIXE Biomedical and Environmental current research activities and open interesting possibilities in Geological investigation.

The attention that is being dedicated to Biomedical Sciences, is centred both in **Environmental Health/Occupational** applications and in environmental toxicity studies. On the other hand the assiduous collaboration with medical teams favoured the settlement of PIXE as a recognised analytical technique for elemental determinations in clinical diagnosis and toxicity. Due to a recent research contract, Biomaterials studies were brought into the Van de Graaff team activities.

In the area of environmental applications the collaborative work carried out with other ITN groups as well as the participation of national or international research teams should be emphasised. Under this scope a *biomonitoring* program for air pollution survey in Portugal was accomplished. JNICT (National Research Science Foundation), the Ministry of Environment and the IAEA, also in the field of environmental assessment are financially supporting two other projects.

Research Team

Researchers –	5* (3 PhD)
Research Students –	3 (2 PhD, 1 MSc)
Undergraduated Students –	7
Technicians –	1

* 1 from FCUL and 1 from FCT/UNL.

Publications

Journals –	9 (7 in press)
Conf. Commun. –	6
Theses:	
Lic. –	3

	10 ³ PTE
Expenditure:	3.241
Missions:	567
Others Expenses:	1.313
Hardware & Software:	268
Other Equipment:	1.093

		10 ³ PTE
Funding:		3.241
OE/ITN	OF	1.133 ⁽¹⁾
External	1996	2151 ⁽²⁾
Projects:	1997	1.608
Others		349

⁽¹⁾ This cost will be covered by external funding
⁽²⁾ Funding not used in 1996

Amalgam Components Drift in Teeth - Toxicity Risks: A Preliminary Approach

*M.L. Carvalho**, *T. Pinheiro**, *M.A. Barreiros[○]*, *C. Casaca[†]*, *A.S. Cunha[†]* and *P. Chevallier***

**Centro de Física Atómica da Universidade de Lisboa, Portugal*

•*Instituto Tecnológico e Nuclear, Sacavém, Portugal*

○*INETI-ITA, Lisbon, Portugal*

†*Faculdade de Medicina Dentária - Universidade de Lisboa, Portugal*

***LURE, Université Paris XI, Orsay, France*

Abstract

The use of ion beam techniques applied to teeth studies have been extensive in what concerns the major elements distribution. However, it is not clarified whether amalgam components are absorbed and drifted through teeth material, although the toxicity of the elements used in amalgams, as Hg, are well known.

This work is an attempt to assess a possible teeth contamination originated by the amalgams. Therefore, teeth with metallic amalgam, as well as healthy ones, were studied. The teeth were longitudinally cut and each slice was scanned from the inner region to the surface enamel for elemental profiles determination purposes using Particle Induced X-ray Emission (PIXE) and Synchrotron Radiation X-ray Fluorescence (SRXRF) techniques. High levels of Zn, Ag, Sn, Hg and Pb were found along the scanned teeth restored with the metallic amalgam. The elemental distribution patterns suggest diffusion of these elements in the teeth material from amalgam constituents.

Nucl. Instr. and Meth., (in press).

Enhanced X-Ray Yields from Insulating Samples

A.P. Jesus¹, *M.A. Reis²* and *L.C. Alves²*

¹ *Nuclear Physics Center, University of Lisbon, Portugal*

² *Nuclear and Technological Institute, Sacavém, Portugal*

Abstract

Some insulating samples under proton bombardment produce a very large bremsstrahlung background and enhanced X-Ray lines.

X-Ray energy spectra of several insulating samples (fluorides of metals with valence 4, 3, 2 and 1, a fluoride glass and three fluoride free samples) are presented for proton energies from 0.175 MeV to 2.00 MeV. Also multiscaling spectra have been collected in order to relate the X-ray yield enhancement with the sample charging up process.

It may be concluded that the enhancement effect is obtained during the charged state of the samples and not during the discharge and that structural properties of the samples rather than their insulating properties play an important role in producing and keeping that charged state. Considerations of ionisation cross section values suggest high energy electrons as the main ionising agent.

It is also shown that some samples can hold the charged state, with corresponding high X-Ray yields, for very long periods, suggesting their use as X-Ray sources.

Nucl. Instr. and Meth., (in press).

Air Particulate Matter Characterisation of a Rural Area in Portugal

L.C.Alves*, **M.A.Reis***, **M.C.Freitas****

* ITN, E.N.10, Physics, Sacavém, Portugal

** ITN, E.N.10, Chemistry, Sacavém, Portugal

Abstract

An aerosol monitoring of a rural and an urban area was accomplished in the frame of an international project promoted by the IAEA. The Gent PM10 air sampler equipped with NILU (Norwegian Institute for Air Research) stacked filter units (SFU) was used for air particulate matter sampling. Filter loads were measured by gravimetry and elemental concentration analysis carried out with both PIXE and INAA techniques. In this work we report the results obtained in a rural area located at 30 km east of Oporto, near a coal fired power plant. The samples were collected from September 1995 to August 1996 on a twice a week basis. The use of complementary analytical techniques allowed to determine concentrations for 23 elements. Time variations for both total particulate mass and elemental concentrations are presented. Enrichment factors were calculated for the fine ($PM < 2 \mu m$) and the coarse ($10 > PM > 2 \mu m$) fractions. Principal Components Analysis was applied to infer particulate mass emission sources. Another Gent air sampler was installed at the same sampling site but under different working conditions. Time averages obtained with the two systems are also compared.

The results obtained with the two collectors operating under different conditions suggest that the statistical distribution of pollutants can be accounted by taking the average of a week and therefore a weekly monitoring of airborne particulate matter is proposed.

A factor related to the coal fired power plant emissions has been identified. Since the power plant is going to be converted from coal to natural gas combustion, the results now obtained are important for a future comparison.

Nucl. Instr. and Meth., (in press).

Elemental Composition of Sediments Determined by TTPIXE

J.E. Martín^a, **R. García-Tenorio^b**, **M.A. Respaldiza^a**, **F.J. Ager^a**, **M.F. da Silva^c**

^a Facultad de Física, Universidad de Sevilla, Apto. 1065, 41080 Sevilla, Spain

^b Dpto. de Física Aplicada, ETSA, Univ. de Sevilla, Avda. Reina Mercedes 2, 41012 Sevilla, Spain

^c Departamento de Física, ITN, E.N. 10, 2685 Sacavém, Portugal

Abstract

In this paper, the goodness of the Thick Target Proton Induced X-ray Emission technique for the determination of the elemental composition of sediments has been checked through its application to reference sediment samples. The results indicate the suitability of the technique in environmental studies.

Nucl Instr. and Meth. B (in press).

Influence of the mining activity on sediments from the Odiel river (sw of Spain) analyzed by TTPIXE

J.E. Martín^a, R. García-Tenorio^b, M.A. Respaldiza^a, J.P. Bolívar^c, M.F. da Silva^d

^a *Facultad de Física, Universidad de Sevilla, Apto. 1065, 41080 Sevilla, Spain*

^b *Departamento de Física Aplicada, ETS Arquitectura, Universidad de Sevilla, Avda. Reina Mercedes 2, 41012 Sevilla, Spain*

^c *Departamento de Física Aplicada, EPS La Rábida, Univ. de Huelva, 21819 Palos, Huelva, Spain*

^d *Departamento de Física, ITN, E.N. 10, 2685 Sacavém, Portugal*

Abstract

Sediments from all over the Odiel river (southwest of Spain) were studied by Thick Target Proton Induced X-ray Emission. For the analyses, 2 MeV protons from the 3 MV Van de Graaff accelerator of the ITN, Sacavém (Portugal), were used. Two runs for every sample were carried out: with and without a 1 mm plastic filter in front of the Si(Li) detector. The concentrations of 26 elements were determined, although, for this study, we considered only 14 of them. The aim was to analyse the influence on the Odiel river sediments of some pyrite mines sited in the vicinity. The results have shown not only the contamination of the Odiel riverbed downstream the mines, due to the mining activity, but also that this activity can be also partially responsible for the contamination of the estuary formed by the Odiel and Tinto rivers and the Atlantic ocean near the city of Huelva, together with a group of nearby factories.

Nucl Instr. and Meth. B (in press).

TTPIXE Analysis of Phosphate Rocks and Phosphogypsum

J.E. Martín^a, R. García-Tenorio^b, M.A. Respaldiza^a, M.A. Ontalba^a, J.P. Bolívar^c, M.F. da Silva^d

^a *Facultad de Física, Universidad de Sevilla, Apto. 1065, 41080 Sevilla, Spain*

^b *Dpto. de Física Aplicada, ETSA, Univ. de Sevilla, Avda. Reina Mercedes 2, 41012 Sevilla, Spain*

^c *Departamento de Física Aplicada, EPS La Rábida, Univ. de Huelva, Huelva, Spain*

^d *Departamento de Física, ITN, E.N. 10, 2685 Sacavém, Portugal*

Abstract

The TTPIXE technique was used for the determination of the concentration of major, minor and trace elements in phosphate rock and phosphogypsum samples. All the samples came from a fertilizer industry sited in Huelva (SW of Spain). The analyses were done using proton beams from the 3 MV Van de Graaff accelerator of the ITN, Sacavém (Portugal).

The aim was to obtain data about the distribution of the different elements in the process of P₂O₅ production in the factory. The obtained information allowed to evaluate the effectiveness of the chemical treatment employed for that, and gave a detailed information about the elemental composition of the phosphogypsum (main by-product of phosphate fertilizer production). These last data can be useful to define the phosphogypsum management and to evaluate its environmental impact.

Nucl Instr. and Meth. B (in press).

Elemental Drift in Teeth - The Influence of Amalgam Materials, Dietary Habits and Environment

T. Pinheiro[•], M.L. Carvalho, M.A. Barreiros[○], C. Casaca[†], M.M. Costa[○], A.P. Jesus** & A.S. Cunha[†]*

**Centro de Física Atómica da Universidade de Lisboa, Portugal*

•Instituto Tecnológico e Nuclear, Sacavém, Portugal

**Centro de Física Atómica da Universidade de Lisboa, Portugal*

○INETI-ITA, Lisboa, Portugal

†Faculdade de Medicina Dentária - Universidade de Lisboa, Portugal

*** Centro de Física Nuclear da Universidade de Lisboa, Portugal*

Abstract

Major and minor elemental composition of teeth have been extensively studied. However, data on the trace elements that mix or diffuse in the teeth material are scarce, if research on fluorine is excluded. These trace elements may give information about the dietary and living habits of the individual, the exposure to particular environmental conditions and about the contamination risk arriving from the dental treatment with metallic amalgam, that contains recognised toxic components, e.g., Hg and Pb. The teeth elemental apportionment relative to the referred factors, in particular the amalgam components spread and its possible adverse effects are questions not fully clarified.

Methods: In order to evaluate the influence of the living habits, saliva elemental content, existence of dental caries and amalgam types, in the teeth elemental distribution, teeth (with and without treated caries) and saliva from several persons with different professional activities (therefore exposed to particular environment conditions) were analysed. Teeth longitudinal slices were carried out with a microtome in order to expose the different teeth layers, from enamel to nerve canal and to permit the analysis from root to tip. In the overall the major, minor and trace constituents e.g., F, Na, Mg, P, S, Ca, Fe, Zn, Sr, Ag, Sn, Hg and Pb, could be determined using both Particle Induced X-ray Emission (PIXE), Proton Induced Gamma Emission (PIGE) analytical techniques. The elemental profiles can be achieved by scanning the samples with a collimated incident proton beam of 1mm diameter. Total Reflection X-ray Fluorescence (TXRF) was employed to the saliva elemental determination.

Results and Conclusions: The study shows that the elemental profile variations are related to the person particular environmental exposure, saliva composition and their dietary habits.

The drift of amalgam components in teeth suggests differences in the elemental uptake by the teeth material. The diffusion of Ti, Ag, Sn, and Pb seem to be particularly facilitated if compared to Hg. Nevertheless, all of the referred elements can reach very high concentration levels in dentine and in the pulp cavity. The results obtained permit to outline the elemental distribution in teeth for a large elemental range, from F to Pb. Moreover, the amalgam elemental trend in the teeth material, in a time series will allow predicting of risks to human health.

Communication to: *World Congress on Medical Physics and Biomedical Engineering – NICE97, 1997, Nice, France.*

Research on Biochemical Parameters Indicative of Toxicity in Two Cyprinids Exposed to Copper

A.M. Viegas Crespo*, P. Lopes*, A.M. Pires*, T. Pinheiro[•], M.M. Coelho*, M.J. Collares-Pereira*, C. Vale[†], A.M. Ferreira*, M.C. Santos*, M.G. Ramalhinho*, C. Sérgio*, M.A. Reis*, E.G. Crespo*, M.L. Mathias*

* Dept. Zoologia e Antropologia, Faculdade de Ciências, Universidade de Lisboa, Portugal

[•] Instituto Tecnológico e Nuclear, Sacavém, Portugal

[†] IPIMAR - Lisboa, Portugal

This work is part of a research project aiming at the study of heavy metals bio-accumulation and the evaluation of physiological, biochemical and genetic toxicity markers, in freshwater fish and riverside micro-mammals of the Guadiana river basin (center/south of Portugal). Reference is made to results obtained on the bio-accumulation of copper of two cyprinids, *Barbus sp.* and *Rutilus alburnoides*, in two locations showing significant differences in the water copper concentration - Xévoira river (S. Mamede) and Oeiras river (Neves Corvo – copper mines). In the monitoring which is being undertaken, the levels of heavy metals in the water and their accumulation in briophytes are established as the measure of environmental pollution. The activity of the superoxide dismutase and glutathione-S-transferase enzymes, regarded as biochemical parameters indicative of toxicity, are determined in the liver of pools of animals of the same sex and size. Levels of heavy metals are evaluated in the same pools by PIXE analysis. The preliminary results relating to Autumn and Winter show an accumulation of copper in the livers of animals exposed to high levels of that element in both genera ($p < 0.05$). In parallel a significative increase was observed in the activity of glutathione-S-transferase ($p < 0.05$) for *Rutilus alburnoides*. These results suggest that these two cyprinids are good bio-indicators of environmental copper and glutathione-S-transferase may be a good biochemical indicator for the copper toxicity.

Comunicación to: *Ninth International Congress of European Ichthyologists (CEI9) "Fish Biodiversity"* Book of Abstracts, Ed. Pier Giorgio Bianco, 1997, Napoli, Italy.

Comparison of INAA and PIXE in the Analysis of Lichens

L.C. Alves, M.A. Reis, M.C. Freitas

Instituto Tecnológico e Nuclear, Sacavém, Portugal

Abstract

INAA and PIXE reveal themselves as important complementary techniques in the elemental analysis of environmental samples. In a pollution survey of Portugal using lichens as biomonitors, it was possible to get results for 44 elements in the characterisation of main emission sources. Results were obtained for 37 elements using INAA, for 22 elements using PIXE and 15 elements were obtained by both techniques. Precision and accuracy are discussed using standard reference materials. For the 15 elements concentration values obtained by both techniques, the results are compared and discussed.

Comunicación to: "*International Workshop on Biomonitoring of Atmospheric Pollution*", Lisbon, Portugal, 21-24 September 1997.

Mean Annual Response of Lichens *Parmelia Sulcata* to Environmental Elemental Availability

M.A.Reis¹, L.C.Alves¹, M.C.Freitas¹, B. van Os², H.Th.Wolterbeek³

¹ ITN, EN10 Sacavém, 2685 Sacavém, PORTUGAL

² National Geological Survey, Harlem, The Netherlands

³ IRI-TU Delft, Mekelweg 15, 2629 JB Delft, The Netherlands

Abstract

Lichens collected in an area previously identified as unpolluted were transplanted to six different places located in polluted areas and near Power Plants (both fuel and coal powered). A total of 26 lichen samples were made for each place, each sample weighing about 2g. Two were analysed as zero or reference and the remaining 24 were hanged in nylon net bags in order to be able to collect two samples each month, out of every station. Besides the 24 lichen samples, each station was provided with two total deposition collection 10 liter buckets (with 25 cm diameter funnels) and an aerosol sampler. Concentration in both lichens and aerosols were measured by PIXE and INAA at ITN, and total deposition residues were analysed by ICP-MS at the "The Netherlands Geological Survey".

On this work we present the results obtained by looking for correlations between lichens elemental concentrations and annual averages of elemental availability variables such as concentration in suspension in the atmosphere and concentration in total deposition samples, for a total of 40 elements. In order to access both the limitations and the reliability of the results a discussion on the details of handling this data set is presented.

A mathematical function which tentatively represents the lichen up-take response to water availability is also proposed.

Communication to: "International Workshop on Biomonitoring of Atmospheric Pollution", Lisbon, Portugal, 21-24 September 1997.

Lichens *Parmelia Sulcata* Time Response Model to Environmental Elemental Availability

M.A.Reis¹, L.C.Alves¹, M.C.Freitas¹, B.van Os², H.Th.Wolterbeek³

¹ ITN, EN10 Sacavém, 2685 Sacavém, Portugal

² National Geological Survey, Harlem, The Netherlands

³ IRI-TU Delft, Mekelweg 15, 2629 JB Delft, The Netherlands

Abstract

Lichens *Parmelia sulcata* collected in an area previously identified as non polluted, were transplanted to six stations, five of which were placed near Power Plants and the other was placed in an area expected to be pollution free. Together with the lichen transplants, two total deposition collection buckets and an aerosol sampler were installed. Lichens were recollected two every month from each station. At the same time the water collection buckets were replaced by new ones. The aerosol sampler filter was replaced every week, collection being effective only for 10 minutes out of every two hours, in the remote station, aerosol filters were replaced only once a month, the collection rate being kept. Each station was run for a one-year period. Both lichens and aerosol filters were analysed by PIXE and INAA at ITN. Total deposition samples were dried by an infrared lamp, and afterwards acid digested and analysed

by ICP-MS at the National Geological Survey. Data for all the three types of samples were then produced for a total of 22 elements. In this work we used the data set thus obtained to test a model for the time response of lichen *Parmelia sulcata* to a new environment.

Comunicacion to: "*International Workshop on Biomonitoring of Atmospheric Pollution*",
Lisbon, Portugal, 21-24 September 1997.

Current Work

Study of Dispersion of Pollutants in the Sado's Estuarine Zone

M.A.Reis¹, L.C.Alves¹, M.C.Freitas¹ and A.P. Jesus²

¹ - Nuclear and Technological Institute, Sacavém, Portugal

² - Faculty of Sciences and Technology, New University of Lisbon, Lisbon, Portugal

JNICT/DGA PEAM/P/AMA/615/95 Project "Estudo de Dispersão de Poluentes na Região Industrial do Estuário do Sado Utilizando Biomonitorios"

This project has also the co-operation of T. Mikkelsen from Risø National Laboratory-Denmark, H. Wolterbeek and T. Verburg from IRI-TU Delft, The Netherlands

This project aims at the study of pollution distribution patterns in a well defined area through the use of biomonitors, and its comparison with the results obtainable by dispersion modelling. The particular area in question is partially inserted into a highly industrialised zone of Portugal, namely the Setúbal's peninsula. The area in question is centred at the Setúbal's Power Plant for which records of emissions exist, thus allowing (in principle) their use as input into the dispersion models. Nevertheless, several other high pollution sources exist in the surroundings or even inside the area under study. In order to combine as much information as possible within reasonable limits, the following set-up was implemented:

- lichens *Parmelia sulcata* were transplanted to a total of 42 places out of 60 points defined on a 2.5 × 2.5 Km grid, 13 of the spots being located over water were unusable.
- lichen transplants were placed in the field in the 10th and 12th of December, in these stage 5 grid places were left of due to several logistic problems (from winter access impossibility to lichen support equipment damage);
- three aerosol collecting stations will be running during the whole period of lichen campaign, two of which are now running for more than three years in a weekly filter replacement bases;
- two of the above stations were selected to place two identical sonic anemometer (low grade commercial) plus thermocouple systems (developed at Risø National Laboratory Denmark), for micro meteorological data recording. These systems were installed at a high of 6m above ground;
- the above data will be used to run the LINCOM and RIMPUFF Risø models to obtain the patterns which would be expected from a dispersion model approach;
- the lichen campaign is planned to have a 12 month duration, two lichen samples being recollected from each site every three month;
- a special small equipment was developed to allow for the installation of a set of lichens permanently facing the wind and another set facing the opposite way;
- elemental analysis of lichen and aerosol samples will be performed by INAA and PIXE techniques;
- elemental concentrations data thus obtained will be submitted to Monte Carlo Aided Target Transform Factor Analysis (MCATTFa, developed at IRI-TU Delft) thus allowing for source apportionment from the receptor model approach;
- the MCATTFa code allows the plotting of individual factors influence mapping, which can be directly compared to the results obtainable by dispersion modelling;
- the dispersion models data will also be compared directly to the lichen concentration mapping obtained from the lichens;
- finally, the possibility of combining measured aerosol concentration and the dispersion model results to obtain density probability fields for suspended elemental concentrations, will be inquired.

S.A.R.A - an Automatic System for Replacement of Aerosol Stacked Filter Units

R. Mateus^{1,2}, L.C. Alves¹, M.A. Reis¹

¹ Nuclear and Technological Institute, Sacavém, Portugal

² Nuclear Physics Centre, University of Lisbon, Portugal

Aerosol sampling is most of the times a problematic procedure. When remote locations are considered this becomes an even worse problem. Sometimes, even places within a city must be characterised as “remote” due to the frequency of traffic jams which may imply in hours of work loss just to replace filters.

On developing an automatic filter replacement device for aerosol studies big problems are faced relative to the assurance of the vacuum tightness characteristic of the air path. This is essential to assure the correctness of the measurements made.

The automatic system now presented was already referred in the previous report. This year several problems relating both vacuum tightness and robustness of the whole system were identified revealing themselves to be even more critical than *a priori* expected. Several tests have been made comparing SARA and a single SFU unit sampler model from Gent University (which requires manual SFU replacement). During these tests several problems were identified and corrected in SARA functioning.

In Table I a sequence of the final pressures attained for each sampling unit when the inlet of the system is close is presented. This table refers to different stages of SARA fine tuning modifications. The whole set of coupling parts had sometimes to be completely reconstructed in order to overcome leakage problems. Mechanical stress situations were frequently identified as responsible for defective working conditions. Non rigid coupling parts had to be designed to overcome the problem. Phase 1 and 2 presented in table I represent the test of materials with different elastic tension. The final solution required besides a non-rigid coupling, additional detail on the mechanical system design namely on the distribution of forces acting on the moving parts and on the precision of the machining of the coupling parts.

Table I : Results from the tests of fine tune of vacuum air tightness.

Sampling Unit	Coupling System Rigid	Coupling System Semi - rigid (phase 1)	Coupling System Semi - rigid (phase 2)	Coupling System Semi - rigid (final)
1	-0.2 bar	-0.8 bar	-0.8 bar	-0.8 bar
2	no vacuum	no vacuum	-0.8 bar	-0.8 bar
3	no vacuum	-0.8 bar	-0.8 bar	-0.8 bar
4	-0.2 bar	-0.8 bar	-0.8 bar	-0.8 bar
5	-0.2 bar	no vacuum	no vacuum	-0.8 bar
6	variable	-0.8 bar	-0.8 bar	-0.8 bar
7	variable	no vacuum	no vacuum	-0.8 bar
8	variable	-0.8 bar	-0.8 bar	-0.8 bar

Particulate Matter and Health - from Air to Human Lungs

T.Pinheiro^{1,3}, A. Bugalho de Almeida², M. C. Freitas³, L.C. Alves^{1,3}, M. Reis^{1,3}, R. Pinheiro³

¹ Nuclear Physics Centre, University of Lisbon, Portugal

² Santa Maria Hospital, University of Lisbon, Portugal

³ Nuclear and Technological Institute, Sacavém, Portugal

International Atomic Energy Agency Research Contract N^o: POR 9479/R0: Particulate Matter and Health - from Air to Human Lungs

The aim of this project is to search for respiratory system particular aggressors to which workers are submitted in their labouring activity. The working environment monitoring and workers respiratory health control is far from being routinely performed in many Portuguese industries. Data on air quality parameters are scarce as well as the workers medical exams performed periodically by the company physicians, do not account for respiratory health monitoring. Therefore, statistic data on the harmful effects of airborne particulate in human beings inferred through affections or complains of workers exposed have not been regularly produced.

The work performed under the current IAEA contract comprise the environmental evaluation (airborne particulate monitoring according to the periodicity of labouring cycles) at a steel plant, *Siderurgia Nacional* (SN), and the respiratory health status of workers so exposed. So far, a preliminary health status evaluation from 32 of the 80 workers included in this survey was achieved and data on their blood elemental content has been gathered using PIXE and NAA techniques.

The worker's health status evaluation and airborne particulate matter are not completely assessed, therefore results are still not conclusive. Although, 18% of individuals observed have an apparent respiratory affection, exposure period, smoking habits and respiratory symptoms can not be clearly associated. Furthermore, if cases and/or variables in serum are examined for their similarity using a hierarchical cluster procedure, the individuals that had respiratory complains, are aggregated in the same group, as can be observed in Fig. 1. The variables used in the statistical analysis were besides elemental data, total number of working years at the SN steel casting sector (variable "Yexp"), smoking habits (variable "SmHa") and occurrence of complains (variable "Complain"). The variables were also clustered to infer any common behaviour or identification of similar episodes, and the resulting

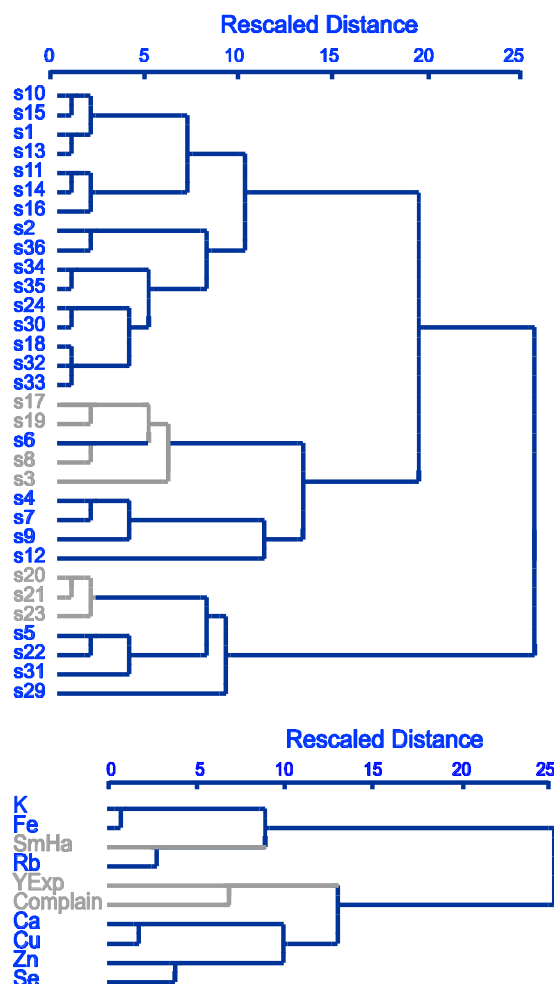


Fig. 1 – Dendrograms for Serum data set. Both cases and variables are grouped.

dendrogram are displayed below the correspondent cases agglomeration. It can then, be observed that the occurrence of complains associates with the total working years.

PIXE Analysis of Pen Paint

L. C. Alves¹, J. C. Soares^{1,2}

¹ Nuclear and Technological Institute

² Nuclear Physics Centre, University of Lisbon, Portugal

It was proposed to make a preliminary study on the possibility of identifying the pen responsible for the writing of a certain manuscript. For that purpose 4 different pens were selected. The same paper sheet was used for making ten different targets (two for each pen and two paper-blank samples) and elemental analysis carried out by PIXE. The samples were labelled accordingly to the manufacturer and the target replicate in the following manner: BIC (BIC1 and 2); Parker (Par_A1 and 2, Par_R1 and 2); Klio-Eterna (Klio1 and 2); paper sample (blank1 and 2).

In the X-ray spectra the elements Al, Si, P, S, Cl, Ca, Mn, Fe, Ni, Cu, Zn and Sr could be identified. Nevertheless only Cu, Zn, Ni and S presented significant differences from the blank spectra, and only those were retained for further analysis.

In order to take into account the different amount of paint analysed in each target, a normalisation to the Cu amount was performed. The concentration ratio of the several elements presented in the different paints is shown in fig. 1.

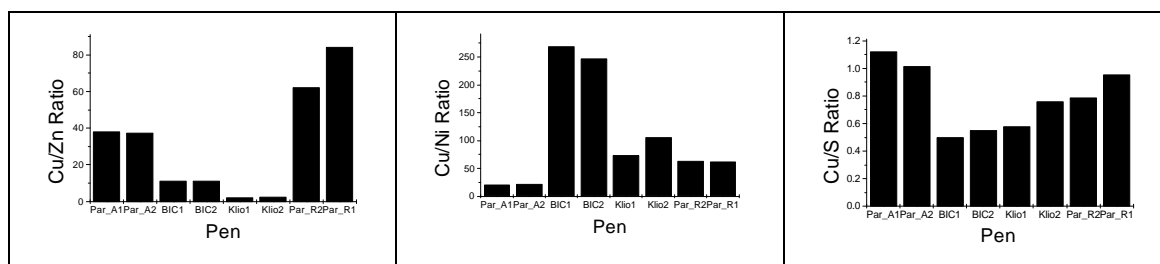


Fig. 1 - Concentration ratio for the different elements determined in the pen paint analysis.

From the graphics it can be concluded that: *i*) the ratio Cu/Ni identifies the BIC pen; *ii*) the ratio Cu/Zn differentiates the Parker_R and the Klio; *iii*) the Parker_A has the lowest value for the Cu/Ni ratio and the highest for the Cu/S one, enabling to identify it among the others. Apart from the graphical analysis, cluster analysis was also performed to establish relationship between the concentration ratios obtained. The quadratic Euclidean distances between the several samples are plotted in fig.2.

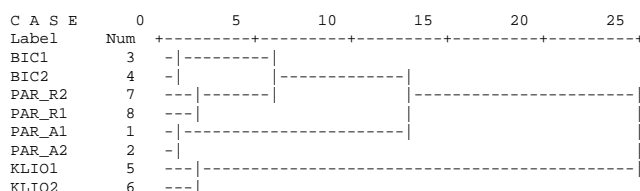


Fig. 2 - Cluster analysis for the four different pen paints. The concentration ratio between Zn, Ni, S and the amount of Cu were the considered variables.

The four different pens are clearly identified by cluster analysis. Nevertheless it is interesting to notice that the distance between the Parker_R and the BIC pen is shorter than the one between the two Parker pens. Although statistically not significant, the results now obtained suggest that it may be possible to clearly identify the pen that wrote a certain document.

X-Ray Yields from Insulating Samples

A. P. Jesus¹, M. A. Reis², L. C. Alves²

¹ - Nuclear Physics Centre, University of Lisbon, Portugal

² - Nuclear and Technological Institute

Results concerning previous results of X-ray yields obtained from insulating samples bombarded by protons were accepted for publication in Nucl. Instr. and Methods. In this previous work we concluded that in some insulating samples, powder pressed pellets namely of fluorine compounds, there is a strong enhancement of the yields of X-ray lines, besides the large bremsstrahlung characteristic of charged samples. Enhancement factors and multiscaling time-dependent spectra are consistent with the existence of high potential states during which high energy electrons make an important contribution to the ionization of atomic inner-shells. Still, there is a lot to understand: the mechanism of charge trapping leading to high potential states, and its relation with the composition and the structure of the sample and the effect causing a preferential enhancement of K X-ray lines. We have been studying this last effect thoroughly. Powder pressed pellets of some insulating compounds were bombarded by protons and alpha-particles. The effect is induced by both projectiles, suggesting that it is related to the charge-up of the sample and not to the primary interaction with the ion beam. Besides X-ray energy spectra, gamma-ray energy spectra and multiscaling time-dependent spectra set at three different windows (low energy gamma-rays, L-lines and K-lines) have been collected. Analysis of the data is still going on.

Nuclear Reactions in ¹⁹F with Low Energy Protons

J. P. Ribeiro¹, Bruno Braizinha¹, A.P. Jesus²

¹ FC/UL, Lisbon, Portugal

² Nuclear Physics Centre

The first measurements of nuclear reaction cross sections with low energy ion beams have been made between 1950 and 1960. Absolute values depended on the knowledge of the beam charge collected on the sample, of the sample thickness and of the detector absolute efficiency. An accurate measurement of the beam charge is difficult due to the possible loss of secondary electrons freed by the beam; the determination of the sample thickness (~1000 Å) depended on the stopping power values existing then. It is a reasonable estimate an inaccuracy of more than 20% arising from these two factors. Concerning the absolute detector efficiency, things were not very simple either. No calibrated radioactive sources were available for energies superior to 3.5 MeV, so for high energy gamma-rays some kind of model had to be developed to extrapolate efficiency curves. Simulation programs based on Monte Carlo methods were not available either.

After the first measurements of the largest and easiest cross sections to measure, next measurements were done relative to the first ones or at least the gamma detector calibration at high energies relied on a few of the first reactions studied yielding high energy gamma rays. So it can be stated that the nuclear reaction cross section “building” is based upon a few “bricks” only. And no effort has been made to verify, with better and most accurate methods, these basic values.

We begun a program to measure some of these basic cross sections starting with reactions on ¹⁹F induced by protons. Actually, by our method, we measure simultaneously the reactions, ¹⁹F(p,αγ) ¹⁶O, ¹⁹F(p,p'γ) ¹⁹F and ¹⁹F(p,p') ¹⁹F. Our target was made of GdF₃ evaporated on a thin carbon backing and the stoichiometry of the target was verified by RBS of 1.8 MeV alpha

particles. Then the number of protons scattered by Gd could be used to overcome the need for absolute values of the beam charge and target thickness. About the determination of the gamma detector absolute efficiency see below. Gamma-ray (with a 25% efficient Ge(HP) detector) and scattered proton spectra were collected for energies from 0.75 to 2.7 MeV. Data analysis is still going on.

Determination of the Optimal Experimental Conditions for F Detection in Thick Samples by PIGE

A.P. Pinto¹, T. Pinheiro², L. C. Alves², A. P. Jesus¹

¹ - Nuclear Physics Centre, University of Lisbon, Portugal

² - Nuclear and Technological Institute

A facility to make PIGE analysis of light elements is being developed at ITN accelerator laboratory. At present the facility is based on an adapted chamber (a new one is being designed) provided with polarizable Faraday cage and entrance collimators and a system of gamma detector positioning and shielding. In order to optimise F detection and quantification in thick samples we made a series of tests with samples of known fluorine concentrations at different accelerator conditions related to extraction current, beam current and proton energy.

The results obtained led to the following conclusions:

- i) The background at low energies, under the peaks of 110 keV and 197keV coming from the reaction $^{19}\text{F}(p,p'\gamma)^{19}\text{F}$, is partially related to the fluorine concentration in the samples and almost independent of the composition of the samples.
- ii) There is a small contribution from fixed environmental conditions, i. e., natural radioactive sources of the walls and shielding. But most of the background is beam related, being for the same beam current on the target, dependent on the extraction current used on the accelerator ion source. As the detector is shielded from direct radiation coming from the accelerator, this means that there is an important contribution to the background coming from multiple scattered radiation in the accelerator room that enters the detector from the unshielded region connected to the liquid nitrogen dewar.
- iii) Being the major part of the background independent of the effects of the beam on the target, any developments leading to the improvement of the absolute efficiency of the detector system will result in a better sensitivity of the method. Presently with a solid angle of about 0.03 sr, the sensitivity to fluorine is of the order of 100 ppm, but can be improved to less than 10ppm by approaching the detector to the sample at a distance of 3 cm.

