

Elemental Characterization and Speciation CEEFI

Miguel A. Reis

The Elemental Characterization and Speciation Group of the IBL (CEEFI), carries out R&D work on ion beam based nuclear analytical techniques aiming at elemental composition characterization, as well as the establishing of ion beam based instrumental speciation methodologies.

So far, the main focus was put on particle induced x-ray emission (PIXE) applied to the characterization of airborne material. A tuning of this working line is presently being undertaken in order to cover a broader but more concise focus on small mass samples. Still, the recently installed aerosol characterization set, that includes a DOAS system (operational since 2007), a meteorological station (operational since January 2008), online in the Portuguese Meteorological Institute Urban Stations Network and a PM10 and PM2.5 sampling station is kept operational.

Nanoparticles and other particulate matter (airborne or not), as well as macromolecules and thin film samples, do now come into the scope of the group application work. This orientation is also connected to recent results on potential health impact issues identified by the group, which fit into a new research context of study of physical mechanisms behind airborne nanoparticles toxicology.

Strategically, the group assumes that it is important not to depend exclusively on collaborations neither for sampling nor for data handling processes. Therefore, effort is put on airborne particle sampling and data handling methods.

Given that PIXE is a matured analytical technique, services are provided to the community in general, and to the scientific community in particular.

Within the organics of the Ion Beam Laboratory (IBL), CEEFI is responsible for the maintenance and improvement of PIXE facilities, and assures that, at least, no losses on the installed capacity occur.

In 2008, the groups' activity was focused on the implementation of the Laboratory for Characterization and Speciation of Aerosols (LCEA), a major equipment upgrade based on a large re-equipment project. Equipment installation and test tasks used a fair chair of the available time. Following the acquisition of the ITN-IBL Tandetron accelerator in 2007, a new High Resolution and High Energy PIXE (HRHE-PIXE) setup was made operational in 2008 and the first high resolution EDS X-ray spectra were obtained using the POLARIS microcalorimeter detector produced by Vericold Tech. GmbH.

The present lines of work involve: (1) ion induced X-ray lines Relative Yield Ion Energy Dependence (RYIED) studies, an effect shown to contain information on both emitters speciation as well as physical dimensions (size) of the emitters container, which is now the subject of two ongoing PhD programs; (2) improvements on the existing spectra data handling software, including holistic approaches to data from other IBA techniques; (3) calculations of ion impact ionization cross-sections, a recovery of work started on the 1990 decade, which regained major importance in face of the latest developments in the field, in particular the new capacities of the 15eV resolution POLARIS X-ray EDS detector; (4) the maintenance of the ITN PM10 airborne particles sampling station, which already lead to the identification of potential important public health impact conditions

Researchers

M.A. REIS, Aux. (Group Leader)
O. MORALES, Aux. (Jul. to Nov. 2008)

Students

P.C. CHAVES, Ph.D. Student, FCT Grant
A. TABORDA, Ph.D. Student, FCT Grant

Technical Personnel

R. PINHEIRO

Collaborators

A. CARVALHO, CFMC-UL, ESTeSL
V. PRIOR, IM
S. CORREIA, IM
A. FERRAZ, Aux. Prof., CFMC-UL, IST
L. CARVALHO, Ass. Prof., CFA-UL, FC-UL
M. MOTA, Graduation Student (from Jul. 2008)
R. TEIXEIRA, Graduation Student (from Dec. 2008).

High resolution EDS and Characterization and Speciation of Micro and Nano-particles

M. A. Reis, A. Carvalho¹, A. Taborda, N.P. Barradas, P. C. Chaves

The characterization and speciation of small mass samples like airborne particles, is the basis and central issue of the CEEFI group work. Each of the specific activities of the group therefore converges towards this aim, or emerges from it. During 2008, the main activities besides the operation of the airborne particle sampling unit at the ITN campus, were: (a) the installation of the Laboratory for the Characterization and Speciation of Aerosols (LCEA), including the POLARIS microcalorimeter detector; (b) the development of software for spectrum data handling; (c) the study of the Gd₂O₃ nanoparticles RYIED ; and (d) bibliographic research aiming at understanding the state of the art regarding mechanisms for nanoparticles toxicology. All of the tasks converging to the same goal, namely using particle induced X-ray for determining chemical and/or sample size effects, and eventual consequences and/or uses of that information. This work being partially a process to deepening the level of data handling of preliminary works presented at the PIXE conference held in Mexico in 2007, showed to be a task leading to greater developments than expected. Combining this fact with the commissioning of all LCEA equipments resulted on delays on reaching the final level of the works, and therefore to delays on publication of results.

Nevertheless, several important results were obtained like the commissioning of the POLARIS detector in which it was possible to achieve a 14 eV resolution at 2.345 keV of Pb-M_{α1} line, as shown in the Pb M-shell X-ray spectrum in Fig. 1, and 26 eV at 17.479 keV of Mo-K_{α1} line, 0.6% and 0.15% respectively.

Besides this important instrumental and operational results, it was also possible to identify plausible mechanisms for a cause/effect relation between high PM_{2.5} chlorine measurements at ITN and a surge in diabetes incidence in Portugal in 2004/2005, as well as plausible explanations for the physics behind the similarity between Gd₂O₃ 5nm particles RYIED pattern and that for Gd-DOTA. An *a priori* strange result, since it is different from the RYIED pattern found for a powder pellet of Gd₂O₃. This raw data result presented at the International PIXE Conference held in 2007, and mentioned in the 2007 ITN Report, has lead to some very important conclusions that will be made public in the beginning of 2009, and have strong implications regarding the potential of the PIXE-RYIED method proposed in 2005 [Reis et al., NIM B229 (2005) 413-424].

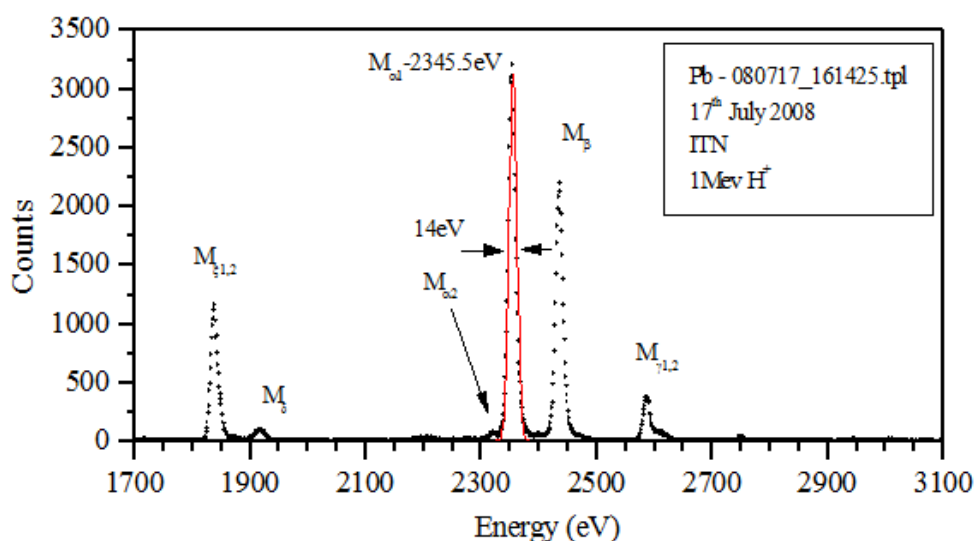


Figure: Detail on the spectrum of Pb M lines obtained using the POLARIS detector at ITN and the Python digital ADC code specifically written for the purpose at ITN.

¹ Escola Superior de Tecnologias da Saúde de Lisboa, Portugal