SYSTEM FOR PIXE TRACE ELEMENT ANALYSIS AT MTF

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Abstract

This paper presents the system for PIXE trace element analysis at a new Ion Beam Laboratory in Trnava. The PIXE system was extended to a new application for analysis of trace elements in aerosol samples. Sample holder was modified with aspect to the dimensions of the aerosol filters and a new sample holder plus Faraday cup (FC) was made, which was placed behind the sample holder. For our experiments the air aerosols were sampled in various MTF laboratories.

1. Introduction

The new 6MV Tandem accelerator at MTF STU in Trnava, at the Advanced Technologies Research Institute, is a compact, multipurpose electrostatic tandem accelerator system produced by High Voltage Engineering Europa B.V. was commissioned in December 2015. The system is based on a 6.0 MV coaxial High-Current TandetronTM Cockroft-Walton type accelerator [1]. High voltage generator provides the terminal voltage varying from 300 kV to 6 MV and is equipped with two ion sources and two end-stations (Fig. 1).

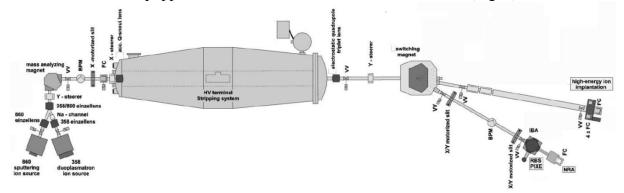


Fig. 1: System arrangement of the 6 MV tandem accelerator [2]

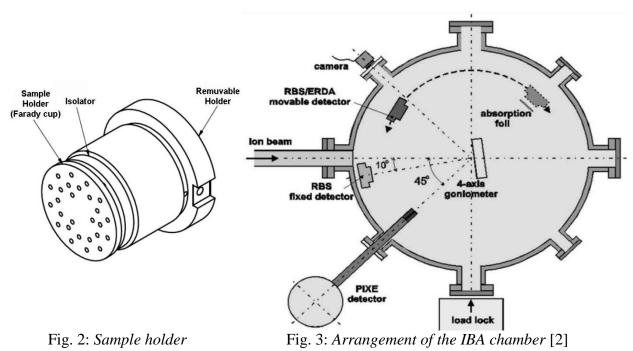
The IBA system (Fig. 1) is equipped with two ion implanted silicon charged particle detectors, one fixed at 170° with respect to the ion beam axis, used for RBS/channelling, the other movable used for RBS/ERDA, which with an absorption foil placed in front of it is used for hydrogen concentration measurements, and finally the PIXE HPGe X-ray detector. For sample positioning, the end-station is equipped with a four-axis goniometer with a 0.01° positioning precision in the three rotational axes and 0.01 mm in vertical translation [2].

The advantage of PIXE technique is its non-destructive nature and simultaneous multielemental character of analysis. The PIXE analysis can be performed for a large range of elements (Na $\leq Z \leq U$) of the periodic table. PIXE is fast, highly sensitive (in limits ppm), precise, accurate and is better for analysis of both matrix and trace elements in thick sample as compared to other ion beam analytical techniques [3, 4]. PIXE has a wide range of applications in biology, medicine, atmospheric aerosol analysis, earth science and archeology, etc. [5]. Many trace elements are emitted into the atmosphere both by human activity and as natural background [7]. Natural sources include soil particles from ground surfaces, gases from volcanoes, forest fires, biological respiration, whereas anthropogenic sources are actually man made sources and include gases from industries, automobiles, coal thermal plants, refuse burning, etc. [8].

2. Experimental setup

A proton beam with energy from 1 to 3 MeV are preferentially used for PIXE analysis. In the case of thick samples the intensity of the proton beam, varying from 1 to 50 nA, is measured using a sample holder (Fig. 2) and it is removable from four-axis goniometer. The proton beam is collimated to the required spot sizes using two motorized slits placed in beam line in front of the IBA chamber.

The HPGe (GL0055) X-ray detector is characterized by a 50 mm² nominal active area, a 5 mm nominal Ge-crystal thickness and a 25 μ m-thick Be window. Its energy resolution at 5.9 keV is 145 eV guaranteed by Canberra. A 100 μ m-thick Melinex absorber can be placed in front of the detector in order to attenuate the characteristic X-rays from light elements, which contribute to pulse pile-up increase of the spectra. The detector is situate at an angle of 45° to the incident beam as can be seen in schematic picture of IBA chamber (Fig. 3). The advantage of the 45° orientation is the reduction of electron bremsstrahlung or background radiations.



The PIXE experimental setup was extended to optimize measurement of aerosols. The front site of the original sample holder was modified so that analyzing protons passing the aerosol filter continue freely to the new FC situated at the backside of IBA chamber (Fig. 4). The beam current measurement by the new Faraday cup was tested.

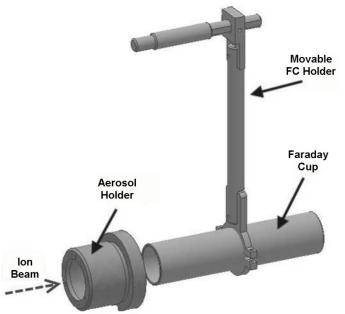


Fig. 4: *PIXE aerosol setup*

The PIXE setup was designed for normal incidence of the primary beam to the aerosol filter.

3. Results

The obtaining spectra were evaluated by GUPIXWIN software. Before using GUPIXWIN many parameters characterizing the detection setup must be determined. With the suitable parameters, we can analyze both elemental composition and concentration present in the target [6].

In our experiment of aerosol sample the incident beam 2.5 MeV, current beam 2 nA and dose 30 μ C were used. The proton beam was collimated to diameter of 5 mm spot. The X-rays were detected by a High purity germanium HPGe (GL0055) detector, Be window thickness 25 μ m, FWHM of 150 eV at 5.9 keV (obtained experimentally, using Fe-55 radionuclide), without absorber foil placed at 45° to the beam axis. Aerosol sample was clamped on our modified PIXE setup for aerosols.

Aerosol samples were collected in laboratories at MTF STU in Trnava, for example laboratory of foundry. The collection parameters were the following: collection time 6 hours, flow rate 2 L/min, distance 1 meter in front of a melting furnace. Polycarbonate membrane filter of 25 mm diameter and 0.8 μ m pore size by SKC standard 244-44MTX personal pump with IOM sampler were used. The filter collected air aerosols with dimensions below 10 μ m, what is typical for air quality monitoring.

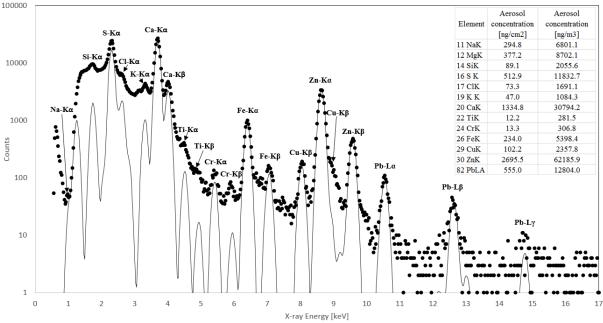


Fig. 5: PIXE logarithmic spectrum of aerosol sample (1 meter from melting furnace)

On the Fig. 5 dots represents measured spectrum, solid line is simulated spectrum by GUPIXWIN. X-ray peaks from appropriate elements are indicated and measured concentrations of impurities in ng/cm^2 of filter, ng/m^3 – impurity per m³ of sampled air, are shown in the table (Fig. 5).

4. Conclusion

The PIXE system at MTF in Trnava was extended to a new modified aerosol holder and Faraday Cup situated behind the holder. Using the new setup the aerosol filters can be analyzed. The GUPIXWIN software with appropriate configuration of detection system can be utilized for quantitative air aerosol analysis with high sensitivity.

Acknowledgement

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References:

- [1] Gottdang, A., Mous, D.J.W. and Haitsma, R.G.: *Nucl. Instrum. Meth. Phys. Res.* B **190** (2002) 177.
- [2] P. Noga. J. Dobrovodský, D. Vaňa, M. Beňo, A. Závacká, M. Muška, R. Halgaš, S. Minárik, R. Riedlmajer, A new ion-beam laboratory for materials research at the Slovak University of Technology, *Nucl. Instr. Meth. Phys. Res.* B, in press, DOI: 10.1016/j.nimb.2017.04.051 [IF: 1.389]
- [3] Johansson S. A. E. and Johansson T. B.: *Nuclear Instrument and Methods* **137** (1976) 473
- [4] Ene A., Popescu I. V., Stihi C., Gheboianu A., Pantelica A., Petre C.: *Rom. Jour. Phy* 55 (2009) 806

- [5] Hoansson S. A. E., Campbell J. L., Mjalmqvist K. G.: *Particle Induced X-ray Emission Spectroscopy* (New York: John Willey & Sons) pp. 147-412
- [6] Zeb J., Ali S., Ahad A., Ahmad I., Akbar J.: Standardization of Proton Induced X-Ray Emission for Analysis of Trace Elements, Cornell University Library, arXiv:1703.04532 2017
- [7] Ogulei D., Hopke P. K., Zhou L., Paatero P., Park S. S., Ondov J. M.: Atmospheric Enviroment, **39** (2005) 3751-3762
- [8] Oswal M., Kaur R., Kumar A., Singh K. P.: *International Journal of PIXE*, Vol. 22, Nos. 3&4 (2012) 271-285