

ANALYSIS OF FOSSIL HUMAN BONES BY PIXE

J. Král, Z. Nejedlý, J. Voltr, I. Čejková*, H. Kvačková*

CTU, Fac. of Nucl. Sci. & Phys. Eng., Dept. of Physical Electronics V Holešovičkách 2, 180 00 Praha 8 *National Museum, Research Chemistry Laboratory Praha

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Chemical bone composition in a living organism is extensively variable and depends on age and sex of an individual and on its living conditions. Generally, it is different in various types of bone and depends even on the bone part from which a sample is taken. On the basis of multielement analysis of fossil bones, information can be obtained on living and social conditions of ancient individuals. It should be kept in mind, however, that both inorganic and organic bone phases are affected by many physical, chemical, and biological processes, which can change their chemical composition in different ways after the burial. Foreign elements may contaminate the bone surface; heteroionic exchange and/or recrystallisation of the inorganic bone phase as well as leaching from bone to soil may take place depending on the surroundings. Knowledge of circumstances and mechanisms of the diagenetic changes is essential for the use of the bone analysis in archaeological research.

A study of chemical composition of fossil human bones from archaeological excavations has been performed by multielement analysis Proton Induced X-ray Emission – PIXE. The bone samples, spongiosa bone – rib, have been prepared in the Research Chemical Laboratory of the National Museum. Their multielement analysis has been performed by PIXE in the Ion Beam Laboratory of the Department of Physical Electronics, Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague.

First, several ways of sample preparation were tested to choose the most suitable one. It was important to homogenize the bone matter and to fix it for the analysis. After mechanical cleaning, each sample of bone was dried and then crumbled and crushed to a fine grain powder. Manual grinding with a pestle and a mortar was later on replaced with pulverization of samples frozen in liquid nitrogen with the use of the Microdismembrator. Two types of samples for analysis were tested. The powdered material was either pressed to form a tablet in a supporting dish or diluted in an acid to obtain a homogeneous solution. The pressed tablet presents a thick target ready for PIXE analysis. A dry residue thin target was prepared by depositing and evaporating 10 ul drop of the acid solution on a thin $(1.5 \,\mu\text{m})$ Mylar foil. The pressed powder targets were found to give essentially stronger signal and better sensitivity than the dry residue thin targets. As a result of the experiments with sample preparation, the following approach was chosen: first, a bone sample was dried at 550°C and then pulverised in deeply frozen state in Teflon cuvette by Microdismembrator; and finally, the resulting powder matter was pressed in a hard polystyrene dish, in which it was also analyzed.

The samples have been analyzed with an external proton beam. Original experience with the samples analyzed in a medium vacuum warned against a possible loosening of separate grains from the tablet. The ultimate way has been the analysis of the pressed powdered bone with the external proton beam in helium atmosphere kept at the ambient pressure. It has been performed in the experimental setup for liquid sample analysis described recently elsewhere [1].

For calibration, laboratory standards were prepared in a way described in [2]. To simulate the bone composition, hydroxyapatite (HAPT) was used as a matrix in which known amounts of calibrating elements were dispersed. These auxiliary standards were tested with thin film MicroMatter XRF calibration standards. The HAPT standards have been then used to calibrate the PIXE system for the bone sample analyses.

To search for minor and trace elements, it was necessary to attenuate the very strong signal of calcium. When the analysis was done with high energy protons, a thick $(965 \,\mu\text{m})$ Mylar absorber has been used. This absorber suppressed the low energy part of PIXE spectrum including peaks of Ca. On contrary, the lighter elements may be measured well without an absorber but at a low proton energy, approx. 800 keV, where the K-shell ionization efficiency for the heavier elements is low.

The study presents analyses of about 200 bone samples with the emphasis on heavier elements.

References:

- KRÁL, J. VOLTR, J. NEJEDLÝ, Z.: PIXE setup for liquid sample analysis. Nucl. Instr. and Meth. B 109/110 (1996), 167-169
- [2] HYVONEN-DABEK, M.: Proton induced prompt gamma-ray emission for determination of light elements in human bone. J. Rad. Chem. 63, No. 2 (1981), 367-378

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