QUANTITATIVE ANALYSIS OF THE TRACE ELEMENTS ZINC, STRONTIUM AND LEAD IN HUMAN BONE BY ENERGY DISPERSIVE X-RAY FLUORESCENCE ANALYSIS

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In the human body trace elements are present in minute quantities and while some of them, for instance zinc (Zn) or strontium (Sr), play an important role in human health, there is a growing number of toxic trace elements, among them lead (Pb), involved in development of numerous diseases.
In bone metabolism the concentrations of the vital trace elements Zn and Sr and the toxic element lead Pb are of great interest.
In this study the concentrations of Zn, Sr, and Pb in four human osteoporotic fractured femoral neck samples, previously analyzed by synchrotron induced micro x-ray fluorescence analysis (SR-µXRF), were determined by quantitative x-ray fluorescence analysis.
For the quantification an approach using external standards with matrix very similar to human bone was chosen. Two materials suitable for standards were tested: calcium sulfate dihydrate ($CaSO_4 \bullet 2H_2O$), commonly known as Gypsum, and milled pig bone.
The measurements were performed with the EDXRF-Spectrometer Epsilon 5 (PANalytical, Almelo, The Netherlands) featuring a three-dimensional polarizing optical geometry. For ideal excitation conditions Germanium (Ge), Zirconium (Zr) and Molybdenum (Mo) were used as secondary targets. The spectrometer has a variable tube voltage of 25-100kV with a maximal power of 600W and is equipped with a HPGe detector.
The organic parts (bone marrow, fat, periosteum) of the bone samples was removed and after dehydration and drying the samples were grinded in a disc mill. The pig bone meal was separated in terms of particle size to a <80 µm and 80-200 µm fraction. Both particle sizes were analyzed and no significant difference with respect to the major and trace elements present in the sample matrix was found.
For each matrix (pig bone meal <80 µm particle size, pig bone meal 80-200 µm particle size and $CaSO_4 \bullet 2H_2O$) a sets of 11 standards with equal amounts of Zn, Sr and Pb was prepared. The standards were pelletized and cover a range of 0-125 µg/g added concentration.
An error analysis of the calibration curves using confidence and prediction bands was performed. The calibrations were validated with the IAEA H-5 Animal Bone Standard. The results obtained were in excellent agreement with the certified values. The Detection Limits (LOD) for Zn and Pb in pig bones and calcium sulfate dehydrate are 1.5 µg/g and for Sr 0.5 µg/g. The Quantification Limits (LOQ) for Zn and Pb were 5 µg/g and for Sr 2 µg/g.
Sample preparation of the human bones was carried out similarly to the pig bones. Human bone meal was separated to <200 µm and 200-1000 µm particle size. The quantification of the human femoral necks shows a very high relative uncertainty for lead due to the concentrations being near the quantification limit of about 5 µg/g. The concentration of Zinc varies in the range of 90 µg/g to 150 µg/g whereas the concentration of Strontium varies in the range of 30 µg/g to 70 µg/g.