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To cite this article: A Pavlenko et al 2007 J. Phys.: Conf. Ser. 93 012047

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Investigation of EPR signals on tooth enamel

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Abstract. Calcified tissues are involved in continuous metabolic process in human organism exchanging a number of chemical elements with environment. The rate of biochemical reactions is tissue dependent and the slowest one at the tooth enamel, the most mineralized tissue of human organism. The long time stability and unique chemical composition make tooth enamel suitable for number of application. The assessment of individual radiation dose by Electron Paramagnetic Resonance (EPR) and evaluations of elemental composition by Instrumentation Neutron Activation Analysis (INAA) are the well known procedures where properties of tooth enamel intensively used. The current work is focused on investigation of EPR signals and determination of chemical composition on several teeth samples having different origin. The EPR spectra and INAA element content of milk tooth, caries tooth, and paradantose tooth have been compared to each other. The results showed that the intensity of EPR signal is much higher for the caries tooth than the for paradantose tooth that is in agreement with depleted Ca content.

1. Introduction
Tooth enamel is the most mineralized and the most stable tissue of human organism. After enamel being formed and calcified it can be changed only by external chemical or physical environment and not subjected to internal biochemical processes. Since tooth is a part of human calcified tissues it makes it an appropriate cumulative indicator of whole skeleton condition. This promotes tooth enamel for different applications on assessment of external environment affection to the human organism. One of the well known applications is reconstruction of individual radiation dose on teeth enamel based on EPR measurement. The other one is assessment of macro, micro and trace elements content by INAA for identifying tooth status and level of exposure of human organism to toxic elements [1].

The EPR spectroscopy of human tooth enamel is becoming recognized technique for individual accumulated doses determination in accidental and retrospective dosimetry [2]. The method is based on measurement of stable radiation induced radicals at g = 1.997 at tooth enamel. This signal originates from carbonate impurities located at the mineral component of enamel, crystals of hydroxyapatite Ca₁₀(PO₄)₆(OH)₂. Radiation induced signal in tooth enamel is overlapped by broad radiation-insensitive signal at g = 2.005 called native or background signal. It is likely that this signal originates from group of radicals located at different sites of organic part of enamel.

The given work is focused on measurement of radiation induced and native EPR signals on tooth enamel and their dependence on Ca content determined by INAA.
2. Materials and Methods

Teeth extracted for medical purposes have been investigated. Generally teeth were extracted from patients affected by caries and one tooth from patient suffered from paradantose. All donors were adults in the range 40-50 years.

Enamel was separated from the dentine by hard alloy dental drill (minimizing the influence of dentine on the EPR signal from enamel). Care was taken not to induce additional mechanically induced signals close to \( g = 2.003 \) interfering with the radiation signal. According to [3] these radicals appear due to rupture of C-C bonds in polypeptide chains are R-NH-CO, R-CH\(_2\) and R-O\(_2\). At room temperature these radicals are oxidized and form R-O\(_2\) radicals. Additionally, mechanical treatment generates radicals at the mineral-organic interface as well, due to sliding of collagen fibers relative to surrounding crystal.

For decreasing of angular dependency of EPR signal enamel was crushed by an agate mortar to small chips with a linear size of 0.1-1 mm. To achieve good repeatability of EPR spectra, the average weight of the sample was kept close to 100 mg. The enamel samples were washed in ethanol and dried in exsiccator with annealed silicagel for several weeks to decrease spectral noise caused by water impurities.

The EPR spectra were measured by BRUKER X-band spectrometer. The obtained spectra were compared with standard \( \text{Al}_2\text{O}_3 \) crystal doped \( \text{Cr}^{3+} \) ions. The use of reference spectra of standards such as \( \text{Al}_2\text{O}_3 \) is useful for both magnetic field verification as well as amplitude normalization. The line of the \( \text{Cr}^{3+} \) spectra at \( g \)-value 1.9800 was collected for every sample.

INAA was used to determine concentration of macro element Ca. The samples (\( m = 200 \text{mg} \)) were irradiated in the vertical reactor channel with water cooling. The thermal neutron flux density in the channel was \( 2 \times 10^{13} \text{ neutrons cm}^{-2} \text{ s}^{-1} \). Teeth samples together with International Atomic Energetic Agency reference materials SOIL-5, 5D-M were wrapped in aluminum foil and placed in aluminum transport container. The irradiation time was 72 hours. The high purity Germanium detector (volume 80.8 cm\(^3\)) and multichannel analyzer LP-4900 were used for gamma spectrometry. The INAA conditions (time of irradiation, decay and measurement as well as a sample - detector distance) were optimized for the minimal statistical uncertainty of the measurement.

3. Results and discussion

The tooth is composed from different chemical elements presented in macro and micro quantities. Calcium, oxygen, phosphorus represents more than 95% of tooth dry weight [2]. The rest is covered by potassium, carbon, hydrogen, magnesium, chlorine, and nitrogen. The tooth contains variety of trace elements absorbed by the body and then transferred to the calcified tissues.

The number of extracted teeth did not allow to study level of environmental contamination by different trace elements or making supposition on element competition at tooth metabolism. In this work the attention was focused on determination of Ca content in the teeth. The selected teeth with different level of caries demonstrated Ca content variation in the range 22-36 percent. The paradantose tooth had highest Ca level 40%.

The parts of the teeth investigated previously by INAA have been studied by EPR. Figure 1 represents EPR spectra of tooth enamel non-irradiated and cumulatively irradiated with two doses. The native signal completely overlaps the dosimetric signal up to significant doses. After the irradiation up to 1 Gy the intensity of native signal and dosimetric one are comparable to each other. Close \( g \)-values and similar intensities of native and dosimetric signal making separation of these two signals an important issue of individual retrospective dosimetry [4].

There are several approaches used for subdividing two signals and purification of dosimetric signal. The first is an individual calibration of enamel radiation sensitivity for each enamel sample (additive dose method). The samples are cumulatively irradiated with known doses and the initial radiation dose is estimated from intersection of regression line with abscissa.

The second method utilizes an average sensitivity of enamel to radiation and called spectrum subtraction method. The spectrum subtraction method for dose reconstruction is non-destructive and
much less time consuming. It originates from the assumption that the variation of radiation sensitivity of enamel from different individuals is moderate. One spectrum of native EPR signal is calculated for all specimens and is excluded from the whole EPR spectra applying different approaches. The intensity of purified EPR dosimetric signal is used for future estimation of individual dose.

Figure 1. EPR spectra of tooth enamel without irradiation and exposed up to 0.1 and 1 Gy.

In way to select proper method for EPR signals purification the influence of native signal on EPR spectra has been investigated. The enamel samples from three different teeth with different Ca content have been selected: caries tooth, milk tooth and paradantose tooth. The typical EPR curves are presented on Figure 2.

Figure 2. EPR spectra of different non irradiated teeth: Paradantose, milk tooth and caries affected. The resonance line at 352 mT is Cr$^{3+}$ standard for spectrometer calibration.
One can notice the highest intensity and different shape of native EPR signal by caries affected enamel. This result is in agreement with [5] where the same results reported. The shape of EPR signal of enamel from milk tooth differ from enamel of paradantose and caries teeth: the maximum of EPR intensity for milk tooth corresponds to minimum intensity for disease affected teeth and vice versa. The group of tooth enamel samples previously examined by INAA was studied on influence of Ca content to EPR intensity. Figure 3 represents dependence of native EPR signal intensity on Ca content in tooth enamel.

![Figure 3. Dependence of EPR native signal intensity on Ca content in tooth enamel.](image)

The intensity of EPR native signal is least for healthy teeth with Ca content 35-36% demonstrating exponential growth with decreasing of Ca presence in enamel. In other words, the intensity of native EPR signal is closely related to health status of tooth and especially to Ca content in enamel. Unfortunately, in the frame of the given work was not possible to verify source of the native EPR signal. One of the possible explanations is presented in [6] and supposed that variation in native signal intensity is related to oscillation of “free” iron liganded with unidentified organic component. The reference [6] points that intracellular or extra cellular component of organic part of enamel are responsible for the given native signal making intracellular component more likely when the other.

4. Conclusion
The intensity of native EPR signal is much higher for the caries tooth than the for paradantose tooth that is in accordance with depleted Ca content in enamel determined by INAA. The exponential growth of the native signal has been observed while decreasing Ca level in the enamel. It was noticed that shape and intensity of native EPR signal is individually depended and has significant influence on whole EPR spectra of tooth enamel that could substantially affect the accuracy of radiation dose estimation. The individual calibration of enamel radiation sensitivity or additive dose method is preferable technique for radiation dose determination allowing to overcome specific peculiarities of tooth enamel as a biological tissue.

Acknowledgments
This work was partially supported by Latvian National Research Program in Materials Science
References