

DETERMINATION OF TOXIC ELEMENTS IN TITANIUM USED IN DENTAL IMPLANTS BY NUCLEAR TECHNIQUES

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ABSTRACT

Nuclear techniques have been employed to investigate the purity of metallic titanium used in dental implants. This study consists of the irradiation of cylindrical pieces of Ti with neutrons for qualitative identification of the nuclides as well as its quantification. Samples of titanium, from different origin, were analyzed and the concentration of the toxic elements present in them was determined.

Key Words: titanium, toxic elements, neutron activation, nuclear spectroscopy

I. INTRODUCTION

In recent years titanium has been used as a biomaterial, in dental implants, due to its chemical and physical properties [1,2]. For this application it is very important to assure its high purity, mainly concerning the presence of toxic elements like: Hg, As, Cd, Th,... To make it possible, two nuclear techniques: gamma spectroscopy and neutron activation analyses were employed to the characterization and quality control of titanium. The application of these techniques is appropriate due to nuclear properties of Ti to be favorable to neutron activation.

II. METHODS

To the development of these studies singles measurement of radioactive titanium were made. Two independent gamma-spectrometer systems with 35cm³ Ge detectors of high resolution (FWHM < 1.9 keV at 1.32 MeV of ⁶⁰Co) were used. Both detectors were operated with 671 ORTEC amplifier, in pile-up rejection mode. The two gamma-spectrometer systems were mounted inside a lead

shielded hood in order to reduce the background radiation. The detectors were calibrated for energy and efficiency through the measurements of standard sources (⁵⁷Co, ⁶⁰Co, ¹³³Ba, ¹³⁷Cs and ¹⁵²Eu) [3].

Various cylindrical pieces of titanium (commercialized biomaterial), with 20mm x 5mm dimensions, were irradiated in thermal neutron flux of the 10¹³n.cm²/s, in the IEA-R1 reactor at IPEN, for different periods (from minutes to hours). This procedure was made in order to identify short and long lived nuclides present in the samples. As natural titanium has an isotopic distribution of 8.2% ⁴⁶Ti, 7.4% ⁴⁷Ti, 73.7% ⁴⁸Ti, 5.4% ⁴⁹Ti and 5.2% ⁵⁰Ti [4] when it is irradiated with neutrons, the identification of radioactive products provides information about its chemical composition.

Gamma Spectroscopy. In the gamma spectroscopy experiment, the cylindrical samples of Ti were studied by means of (n,γ), (n,2n), (n,p) and (n,d) nuclear reactions. Each sample was irradiated for periods of 1 minute, 1 hour and 1 day, in the nuclear reactor. The singles spectra were taken with a Ge detector, in 4096 channels.

The identification of the radioisotopes produced was made in two ways: determining the energies of gamma-rays and their disintegration periods ($T_{1/2}$).

Various measurements of five minute each were taken within a period of 2 hours. An example of a gamma

ray spectrum for short-lived isotopes, recorded during one minute, using two samples from different origins, classified by A and B, is shown in Fig. 1.

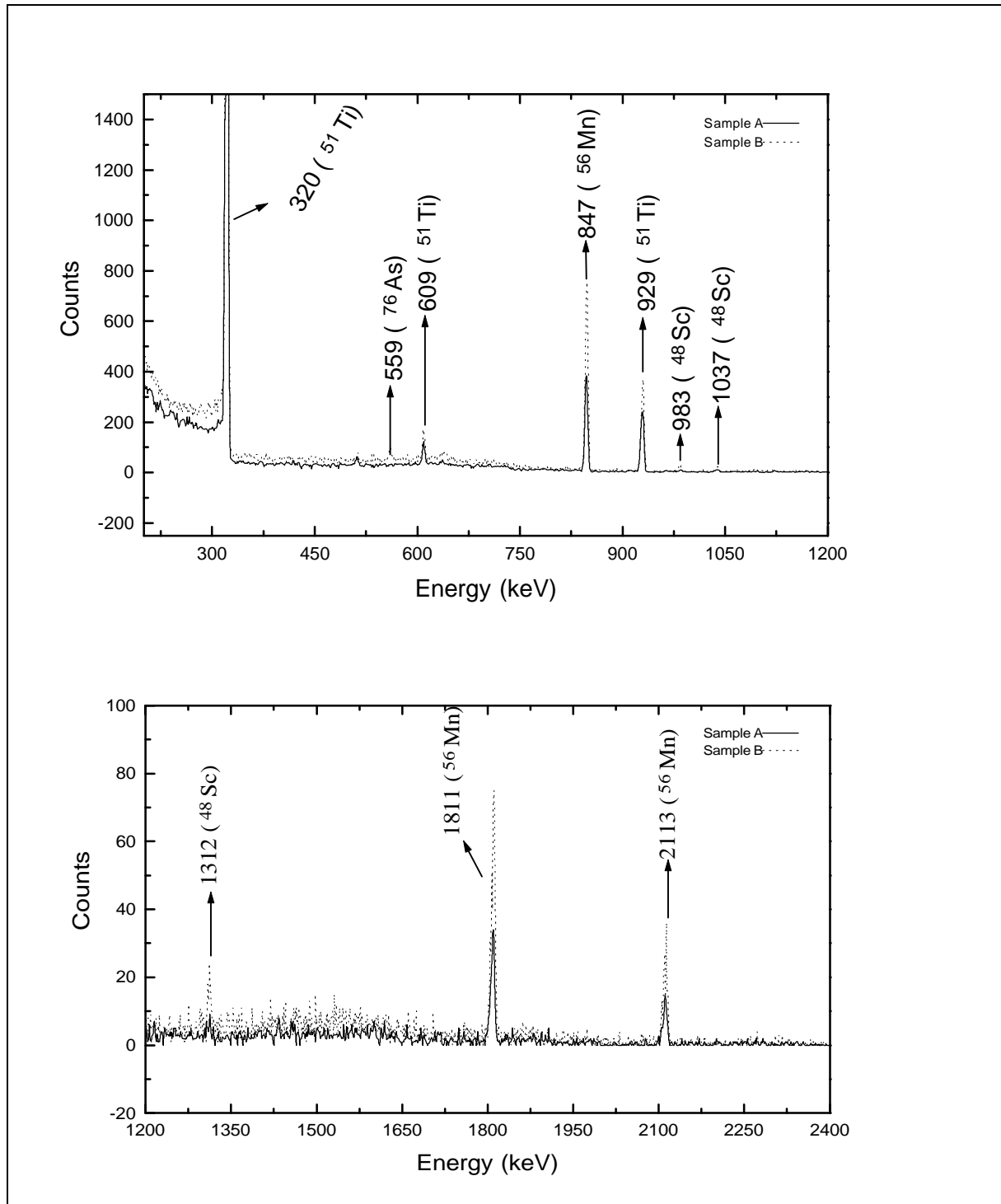


Figure 1. Spectrum of Short-Lived Isotopes.

The analysis of these data revealed the presence of Ti and other isotopes like as Na, Al, Cl, K, Mn and the toxic element As, in all the samples.

Gamma-ray spectrum was taken for long irradiation, too. Various measurements of one hour were

carried out within a period of hours as well as days. Systematically, this procedure was repeated during two months. An example of a gamma-ray spectrum, recorded during one hour of live counting time, using the same samples (A and B), are shown in Fig. 2.

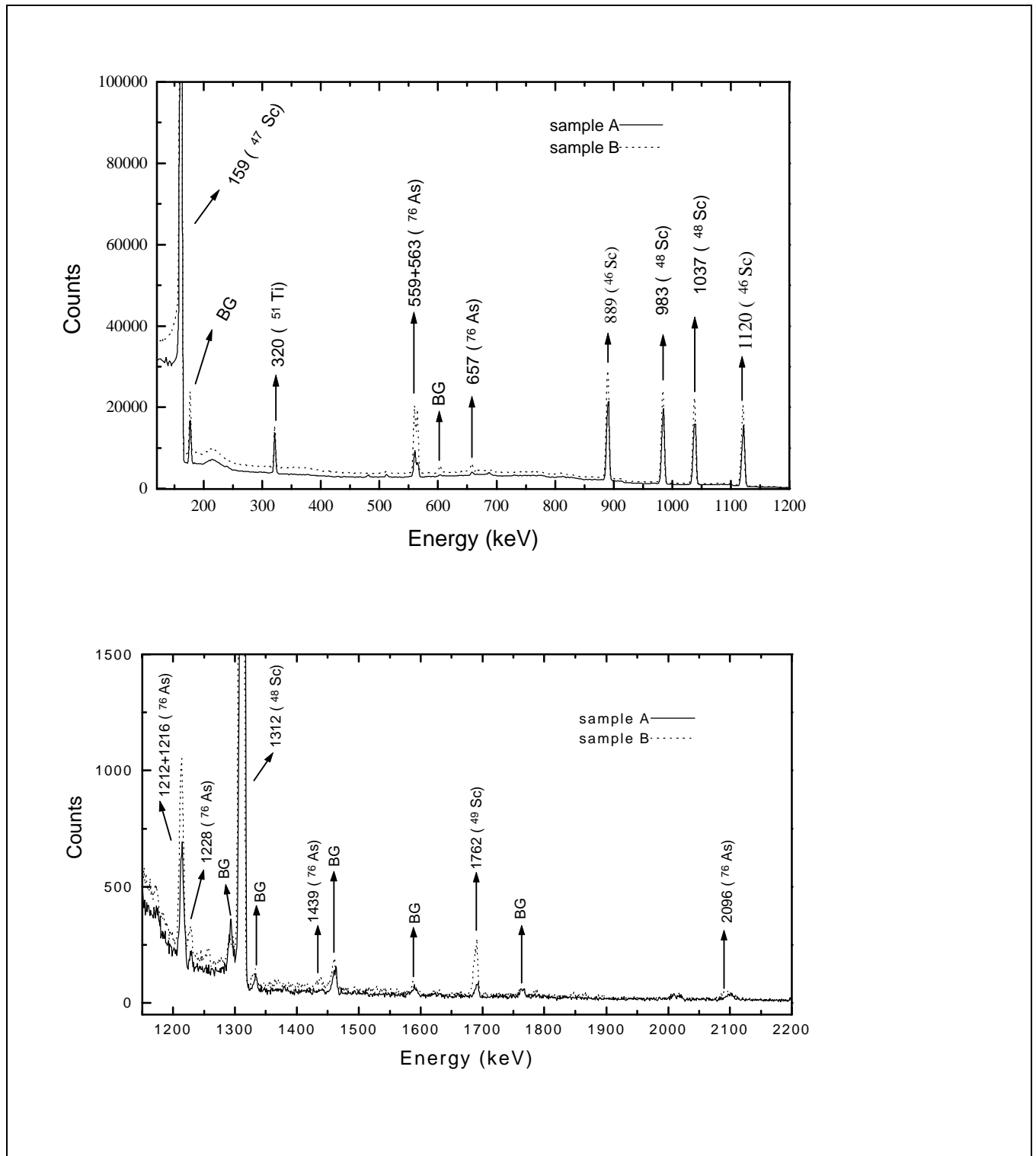


Figure.2. Spectrum of Long-Lived Isotopes.

In this case, the radioactive isotope of Scandium ($A = 46, 47, 48$ and 49) confirmed the presence of Titanium in the samples. Contributions from Arsenic, Na and K were also identified. Finally, after three months, the two samples were gamma-counted again and we noticed only the presence of ^{46}Sc ($T_{1/2}=83\text{d}$).

The measured energies of short and long lived isotopes, present in the samples, (including Ti isotopes) are shown in Table 1.

The time dependence of the number of counts of all the transitions identified was analyzed using the PANORAMIX program [5].

TABLE 1. Gamma-ray Energy of the Isotopes Identified in the Samples of Ti by Gamma-Spectroscopy.

Radioisotope ($T_{1/2}$)	Isotopic Composition (%) [4]	Production	Gamma-ray energy (keV) [4]
^{51}Ti (5.8 min)	5.4	^{50}Ti (n, γ) ^{51}Ti	320, 609, 929
^{46}Sc (83.8 d)	8.0 7.3	^{46}Ti (n,p) ^{46}Sc and ^{47}Ti (n,d) ^{46}Sc	889, 1120
^{47}Sc (3.3 d)	73.8	^{48}Ti (n,d) ^{47}Sc	159
^{48}Sc (43.7 h)	73.8	^{48}Ti (n,p) ^{48}Sc	983, 1037, 1312
^{49}Sc (57.3 min)	5.4	^{50}Ti (n,d) ^{49}Sc and ^{49}Ti (n,p) ^{49}Sc	1762
^{24}Na (15 h)	100	^{23}Na (n, γ) ^{24}Na	1368
^{28}Al (2.3 min)	100	^{27}Al (n, γ) ^{28}Al	1779
^{38}Cl (37 min)	24.23	^{37}Cl (n, γ) ^{38}Cl	1642, 2167
^{42}K (12.2 h)	6.7	^{41}K (n, γ) ^{42}K	1525
^{56}Mn (2.6 h)	100	^{55}Mn (n, γ) ^{56}Mn	847, 1811, 2113
^{76}As (26.3 h)	100	^{75}As (n, γ) ^{76}As	559, 563, 657, 1212, 1216, 1228, 1439, 2096

Instrumental Neutron Activation Analysis. Instrumental neutron activation analysis (INAA) was used to determine the concentration of the Arsenic in the samples of Titanium.

The samples of Titanium were weighed and sealed in plastic envelopes. Aliquots of standard solutions with well known concentration of As was pipetted onto 1 cm² pieces of Whatman N^o.40 filter paper, evaporated to dryness under an infrared lamp, and also sealed in plastic envelopes. Samples and standards were irradiated for one minute at a thermal neutron flux of about $5 \cdot 10^{11}$ n /cm².s at the nuclear reactor. The measurements of the induced gamma - ray activity were performed 1 hour after irradiation, after the decay of the radioisotope ^{51}Ti ($T_{1/2} = 5.8$ min). The gamma - ray spectra were processed by using the VISPECT program [6]. The radioisotope used to calculate the concentrations of the analyzed elements was ^{76}As ($T_{1/2} = 26.3\text{h}$).

To evaluate the concentrations of the As impurity present in the samples, the counting rates (area of photopeaks) corresponding to the gamma rays of 559 keV from samples and standards were compared. The results are presented in Table 2.

TABLE 2. Concentrations of As by INAA.

Sample	Mass (mg)	Concentration of As ($\mu\text{g g}^{-1}$)
A	0.42514	3.8 ± 0.4
B	0.36515	6.4 ± 0.6
C	0.26410	13.2 ± 0.4
D	0.22390	17.9 ± 0.4
E	0.07030	17.1 ± 0.7

III. CONCLUSIONS

In γ - spectroscopy experiment were analyzed five kind of Ti samples classified by: A, B, C, D and E. This classification was made considering the differences in the production process of the cylindrical piece of metallic Ti, like as: origin, dimension, mass and texture. The nuclides identified, presented in table 1, show only the presence of toxic element Arsenic, produced through $^{75}\text{As}(n,\gamma)^{76}\text{As}$ nuclear reaction. Impurities of Na, Al, Cl, K and Mn were also identified by gamma-spectroscopy and according to reference [2] these contaminations are due its production process and surface treatment [7,8] , but no indication about impurities of As was made.

The measurements of the induced γ -ray activity performed, presented in table 2, show the high concentration of As in all the samples and consequently the necessity of toxicological analysis in this commercialized biomaterial.

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