

HOMOGENEITY STUDY ON BIOLOGICAL CANDIDATE REFERENCE MATERIALS: THE ROLE OF NEUTRON ACTIVATION ANALYSIS

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Instrumental Neutron activation Analysis (INAA) is a mature nuclear analytical technique able to accurately determine chemical elements without the need of sample digestion and, hence, without the associated problems of analyte loss or contamination. This feature, along with its potentiality use as a primary method of analysis, makes it an important tool for the characterization of new references materials and in the assessment of their homogeneity status. In this study, the ability of the comparative method of INAA for the within-bottle homogeneity of K, Mg, Mn and V in a mussel reference material was investigated. Method parameters, such as irradiation time, sample decay time and distance from sample to the detector were varied in order to allow element determination in subsamples of different sample masses in duplicate. Sample masses were in the range of 1 to 250 mg and the limitations of the detection limit for small sample masses and dead time distortions for large sample masses were investigated.

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TRACE ELEMENTS DETERMINED IN MINERAL WATERS USING INAA FROM PARQUE DAS ÁGUAS DE LAMبارI AND CONTENTAS

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Mineral water, according to the Brazilian Law N 7.841 of August 8, 1945, "are those from natural springs or springs artificially captured that have chemical composition or physical or physicochemical properties other than ordinary waters, with characteristics that give them a drug action". In this same law, mineral waters can be classified according to their chemical composition in: oligomineral, radiferous, alkaline-bicarbonated, alkalineearthy, sulfated, sulphurous, nitrated, chlorinated, ferruginous, radioactive, thorioactive and carbogasous. In several Brazilian hydromineral resorts, springs of radioactive mineral water are commercially exploited and are consumed by the population that believes this practice is beneficial. For example, in thermal parks like Caxambu, Cambuquira, São Lourenço and Lambari (MG), the waters of the various springs are used for human consumption and are often associated with medicinal use, like diuretic and cathartic waters (with properties of cleaning, purification, facilitating hepato-biliary functions and stimulating intestinal function directly or indirectly), and waters with antiphlogistic properties (anti-inflammatory). Therefore, due to the recommendation of these waters as a form of treatment spent

in spas, the present work was developed in Centro de Radiometria Ambiental – IPEN, with the aid of those responsible for the spas of Lambari and Águas de Contendas and, with the objective to perform an inorganic chemical characterization of the mineral waters of Parque das Águas of Lambari and Águas de Contendas. Samples of mineral waters were analyzed in duplicates and concentrated from 500 mL. The samples were irradiated at the IPEN Research Reactor IEA-R1, for a period of 6h under a thermal neutron flux of 10^{12} n cm⁻²s⁻¹. In the samples of mineral waters of the Parque das Águas of Águas de Contendas the elements Ca, Co, Fe, Hf, K, Na, Rb, Sb, Sc, Th, Zn, La and Sm were found. Similarly, in the Parque das Águas of Lambari the elements Ca, Co, Fe, K, Na, Rb, Sb, Sc, Zn, La and Sm were found. The analysis of the samples of these water parks evidenced that the elements of greatest occurrence were La and Co.

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PREPARATION OF BIOLOGICAL REFERENCE MATERIALS AT LAN – IPEN – CNEN/SP

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Three biological reference materials were prepared to be used in interlaboratory programs and/or proficiency tests, as well as, for calibration of analytical instruments and validation of analytical methods for environmental trace elements: A Perna perna mussel reference material was produced and certified by means of an interlaboratory comparison. The material was designed as a quality assurance tool for element biomonitoring studies along the Brazilian seashore. For the preparation of the Brazilian mussel reference material, 164kg of Perna perna (Linnaeus, 1758) mussels were purchased from a single producer, from Cocanha Beach in Caraguatatuba City, São Paulo State North Shore where mussels are cultivated by the longline system. After cleaning, freeze-drying and homogenization, the mussel samples were packed in 171 bottles and, the material was irradiated with a gamma ray dose of 5 kGy to enhance its stability. The certified mass fraction values and associated expanded uncertainties were obtained for Al, As, Ca, Cd, Cl, Co, Cu, Fe, K, Mg, Mn, Na, Ni, Se, Th and Zn at the certification campaign. A fish tissue reference material was produced in the context of an International Atomic Energy Agency (IAEA) project aiming analytical quality improvement for laboratories of the Latin American and Caribbean region. Whitemouth croake (Micropogonias furnieri), known as corvina, was the chosen species due to its low cost and high consumption in Latin America countries. For preparation, about 300kg of fish was collected and only the edible parts were used. After adequate processing, the fish material was bottled in 534 bottles with approximately 25g each. The reference values and associated expanded uncertainty ($k = 2$) were established for As, Cd, Cu, Fe, K, Mn, Na, Se and Zn; Finally, a bovine kidney reference material to be used in the quality control of meat products was prepared using 35 kg of fresh bovine kidney from cattle reared under controlled feeding conditions. The preparation resulted in a final batch of 175 flasks.