

ESTABLISHING ADEQUATE CONDITIONS FOR MERCURY DETERMINATION IN ENVIRONMENTAL SAMPLES BY INAA

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Mercury (Hg) is a toxic element released into the environment by various atrophic activities. Consequently, the improvement of analytical methods for Hg determination in environmental samples is of great interest. Instrumental Neutron Activation Analysis (INAA) is considered an adequate technique to determine several elements. However, Hg determination by INAA is often hampered by its volatility, which causes losses. The aim of this study was to establish adequate conditions for Hg determination in environmental samples by INAA. The following parameters were evaluated: the irradiation time, polyethylene capsule or envelope for irradiation and spectral interferences caused by other elements present in the samples. For the study, four certified reference materials (CRMs) were analyzed: INCT M-4 CormTis Comorant Tissue, IAEA-085 Methylmercury, Total Mercury and Other Trace Elements in Human Hair, NRC DOLT-3 Dogfish liver certified reference material for trace metals and BCR 186 Trace elements in lyophilized pig kidney. Aliquots of these materials were irradiated together with Hg synthetic standard in the IEA-R1 nuclear research reactor. The Hg synthetic standard was prepared by pipetting aliquots of Hg standard solution and thioacetamide solution into filter paper sheets or into polyethylene capsules. The thermal neutron flux utilized was about $4.6 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ and the irradiation times were of 1 and 8h. The gamma activities of the samples and standard were measured using a Ge detector coupled to a digital spectrum analyzer. Hg was identified by the photopeaks of 77.34 keV of ¹⁹⁷Hg and 279.20 keV of ²⁰³Hg. Results obtained in this study indicated that the Hg impurities present in polyethylene envelopes are negligible. The gamma ray activities of Hg standards measured for different decay times indicated that there is no Hg loss after irradiation. Results obtained in the analysis of CRMs indicated that the adequate irradiation time is of 1h, since long irradiations of 8h indicated the increase of spectral interferences such as ¹⁹⁸Au, ⁷⁵Se and ²⁴Na. The quality control of Hg results, obtained in the CRMs using 1h of irradiation, indicated good precision and accuracy with $|Z \text{ score}| < 2$. The experimental conditions established in this study were applied to tree bark samples. Detection limits for Hg of these analyses were about $0.14 \mu\text{g g}^{-1}$ and $1.9 \mu\text{g g}^{-1}$, for the photopeaks of ¹⁹⁷Hg and ²⁰³Hg, respectively.

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