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The Effect of Acid Digestion on the Recoveries of Trace Elements: Recommended Policies for the Elimination of Losses

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Abstract: Digestion of biological materials is very important for trace element determinations. Although the microwave technique is mostly used, there are cases where open wet digestion is needed. This study examined the optimum conditions for the minimum loss of trace elements during the wet digestion of a sample. The effect of acid composition and digestion time for about 21 ions was investigated. The trace element concentrations were determined by differential pulse polarography. For the digestion procedure, a $\text{HClO}_4\text{-HNO}_3$ mixture was used for the first step and, after its evaporation (60 min), HCl was added as the second step, since in some cases it was necessary. The recoveries were high for ions like Zn, Cu, Ti(IV), Cd, Fe, Mn(II), Co(II), V(III) and Mo(VI) after digestion (60 min) with the $\text{HNO}_3\text{-HClO}_4$ mixture and the addition of HCl had no effect. However, although care was taken to minimize losses by using a long-necked (30 cm) flask, low recoveries were obtained for Pb, Se(IV), As(III), Cr(III) and Cr(VI) when HCl was used after $\text{HClO}_4\text{-HNO}_3$ digestion. Since Se(IV), As(III), Cr(III), Sn(II) and Sb(III) were oxidized by the acid mixture this fact has to be considered for some procedures and they have to be reduced before their determination. It was found that when HNO_3 was used alone or in an acid mixture for digestion, the recovery of Ni was only 40%. However, with the use of HClO_4 or H_2SO_4 , the recovery was very high.

Key Words: Open wet digestion, losses, recovery, elimination, trace elements, differential pulse polarography

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