

## ABSTRACT

The selectivity of the free metal speciation methods, the ion selective electrode potentiometry (ISE) and the ion exchange column equilibration (IEX), was examined at low free metal concentrations using copper metal ion buffers and compared with the speciation predicted by the COMICS program.

An enzyme assay method, based on the inhibition of  $\beta$ -glucuronidase by copper, was developed as an independent monitor to test the accuracy of metal speciation methods. The assay procedure appears to be very sensitive and selective to the free cupric ion. The results of the assay study indicated that the ISE response in metal ion buffers could be altered by the free ligand present in the buffer.

The utility of Nafion ionomer coated glassy carbon electrode (GCE) in metal analysis and speciation was investigated. The Nafion coated mercury coated GCE provides a 75-100 fold preconcentration of Cu, Pb, Cd, and Zn ions, but the sensitivity of the electrode was less than that of conventional ASV apparently due to the presence of a "strong" site and the slow diffusion of metal ions in the membrane. The Nafion membrane achieves metal preconcentration by ion exchange, therefore, the electrode shows more promise in the analysis of ASV insensitive metal ions such as iron. The membrane apparently exclude anionic complexes and is permselective to cationic species. A metal speciation method was developed by incorporating a Nafion coated GCE in a flow cell design. The cupric ion selectivity of this method appears to be comparable with that of the ISE. The potential application of the glassy carbon electrodes coated with cellulose acetate in metal speciation was also examined.

Column experiments were performed to examine the interaction of Cu, Pb, Cd, Zn, Ni, Co and Mn with peat under continuous flow conditions. The effect of proton and major cation concentrations on the stability of metal/peat complexes was also studied. The order of stability of di-valent metal/peat complexes was found to be  $Pb > Cu > Cd > Zn = Ni > Co > Mn$ .