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Department:Chemistry

Degree:PhD

Title:Determination of total selenium and seleno-amino acids in yeast and aquatic organisms by liquid chromatography and inductively coupled plasma mass spectrometry

Selenium-laden agricultural drainwater in the Imperial Valley of California may be affecting the survival of endangered desert pupfish, which inhabit the waters of that region. The primary goal of this dissertation research was to develop methods for routine speciation analysis of selenium in tissues, which has been identified as a critical data need in the derivation and interpretation of selenium residues in fish and waterfowl.

Initial experiments were conducted to optimize the selenium determination for dynamic reaction cell (DRC) ICP-MS using methane as the reaction gas. In the DRC mode, the Ar-Ar background signals could be reduced greatly but at the price of a large loss of net selenium signal intensities. Therefore, a method for total selenium using on-line stable isotope dilution analysis with conventional ICP-MS (SIDA-ICP-MS) was developed. Masses at 77, 78, 79, 81, and 82 were monitored and quantitation of Se was determined based on both 78Se and 82Se. SIDA-ICP-MS was successfully applied to the determination of total selenium in biological materials, such as selenized yeasts, certified reference materials, lab-cultured oligochaetes and desert pupfish.

The separation and quantitation of seleno-amino acids was accomplished by ion-pairing reversed-phase liquid chromatography (RPLC) and detected by ICP-MS at mass 82 using the standard mode. The efficiencies of different extraction methods were evaluated and it was found that methanesulfonic acidic hydrolysis demonstrated a higher extraction efficiency of selenomethionine than enzymatic digestion. Selenomethionine (SeMet) was the only significant Se-containing species detected in the biological samples that were examined.