BIO-RAD

Capillary Electrophoresis
Application Note 53

Analysis of Lead in Foods Using Capillary Electrophoresis

Introduction

The occurance of residues of lead salts in exotic foods is of concern since these materials are often obtained outside normally-regulated sources and distribution channels, and the consumer may unknowingly be at risk in consuming such foodstuffs. Two examples are illustrated in this study. Wild mushrooms are sometimes gathered from locales adjoining motorways, where lead deposited in the soil can be incorporated into the edible mushroom tissues. Limestone-preserved ("thousand year old") eggs are a delicacy prepared in China by burying duck eggs in limestone-containing clay for 1–2 months. If the clay contains significant amounts of lead, the egg tissue may be contaminated during the curing process.

Capillary electrophoresis, a versatile analytical tool for separation of ionic species, can be used for analysis of inorganic ions using indirect UV detection. In this technique, termed capillary ion analysis, a UV-absorbing ion is incorporated into the analysis buffer. Displacement of the chromophore ion by the UV-transparent analyte ion forms a negative peak in the electropherogram with amplitude proportional to analyte concentration. Capillary ion analysis with indirect detection was used for quantitative analysis of lead in extracts of wild mushrooms and preserved eggs.

Sample Preparation

Wild mushrooms: 376 g of mushroom caps from wild cepes were blended with 1 liter of 0.1 N HCl for about 2 min. An aliquot was centrifuged for 5 min in a microcentrifuge, then filtered through a 0.45 µm filter. The filtrate was analyzed directly.

Chinese preserved egg: A single peeled limestone-preserved egg imported from China weighing 47 g was blended with 500 ml 0.1 N HCl, then centrifuged and filtered as described above.

Standards

Solutions of neutral salts of metal cations were prepared in deionized water; concentrations are expressed in ppm of free metal cation.

Analysis Conditions

Capillary	65 cm x 75 µm ID, uncoated
Analysis Buffer	11 mM lactic acid + 7.5 mM 4-methyl-benzylamine + 2.5 mM 18-crown-6, pH 4.2
Purge Protocol	40 sec with analysis buffer
Injection	pressure, 3 psi * sec
Polarity	positive to negative
Voltage	20 kV
Capillary temperature	20 °C
Autosampler temperature	20 °C
Detection	214 nm, indirect

Results

Selectivity

Lead is well-resolved from six other common metal cations, migrating after zinc (Figure 1).

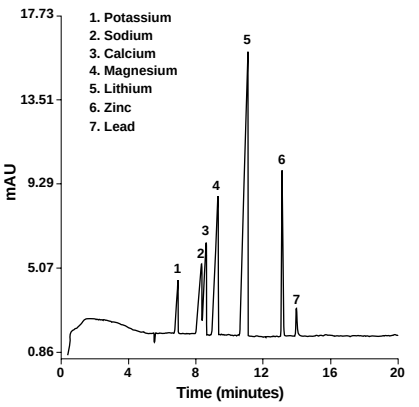

Fig. 1. Separation of inorganic cation standards by capillary zone electrophoresis using indirect detection. All standards were analyzed as their chloride salts except lead, which was analyzed as the nitrate salt.

Sensitivity

A minimum detectable concentration (MDC) at S/N = 3 of 2.6 ppm was calculated from the peak-to-peak noise value of 67 µAU and a response of 385 µAU for a 5 ppm lead solution.

Linearity

Response at 214 nm using indirect detection was linear from the MDC to at least 100 ppm (Figure 2).

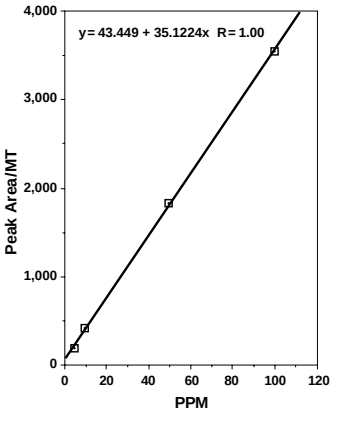

Fig. 2. Response linearity at 214 nm for lead.

Analysis of Wild Mushroom Tissue

A small peak migrating at 13.3 min (Figure 3) was identified as lead by spiking the extract to a final concentration of 25 ppm (Figure 4). Slight differences in peak patterns observed in Figures 3 and 4 are probably due to differences in sample concentration, since the spiked extract was diluted 1:1 with a 50 ppm lead solution. The weight/weight lead concentration in the mushroom tissue was calculated to be 2.3 ppm.

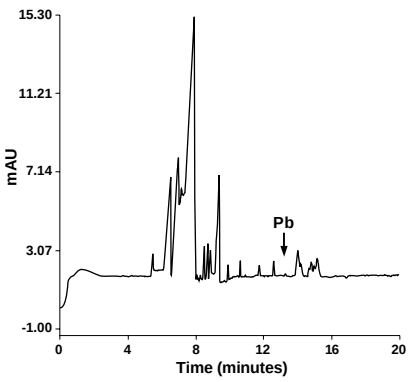

Fig. 3. Analysis of acid extract of wild mushroom tissue for lead content.

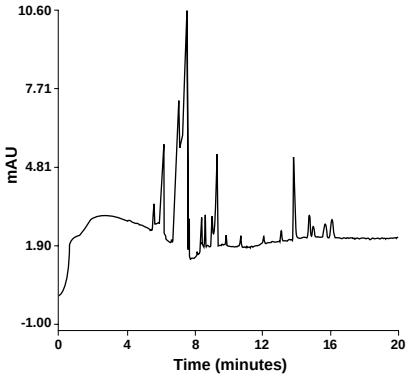

Fig. 4. Analysis of acid extract of wild mushroom tissue spiked with 25 ppm of lead.

Analysis of Chinese Preserved Egg

No response at the migration time of lead was observed in the egg analysis (Figure 5) which was confirmed by spiking the extract to a final concentration of 25 ppm (Figure 6). This indicates that the weight/weight lead concentration in the egg tissue was less than a minimum detectable concentration of 27 ppm.

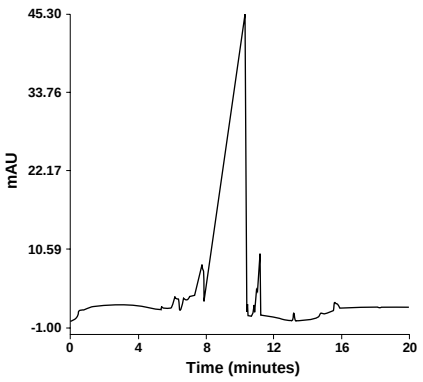

Fig. 5. Analysis of acid extract of preserved egg for lead content.

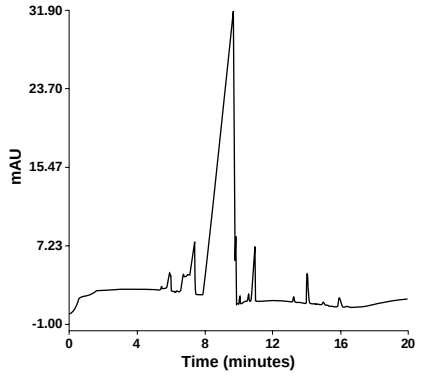

Fig. 6. Analysis of acid extract of preserved egg spiked with 25 ppm of lead.

Reference

Shi, Y., and Fritz, J.S., "New electrolyte system for the determination of metal cations by capillary zone electrophoresis," *J. Chromatogr. A*, **671** 429-435 (1994).

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