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Burkhard Horstkotte, Kateřina Fikarová, David J. Cocovi-Solberg, Hana Sklenářová, Petr Solich, Manuel Miró

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Burkhard Horstkotte[1], Kateřina Fikarová[1], David J. Cocovi-Solberg[2], Hana Sklenářová[1], Petr Solich[1], Manuel Miró[1,2]

[1]Charles University, Faculty of Pharmacy, Department of Analytical Chemistry, Heyrovského 1203, 500 05 Hradec Králové, Czech Republic

[2]FI-TRACE group, University of the Balearic Islands, Department of Chemistry, Carreterra de Valldemossa km 7.5, 07122 Palma de Mallorca, Spain

Abstract

A proof of concept of a novel automatic sample cleanup approach for metal assays in troublesome matrixes as a front-end sample pre-treatment to inductively coupled plasma optical emission spectroscopy - ICP-OES - is herein presented. Target metals, namely, copper, lead, and cadmium were complexed in-system quantitatively using ammonium pyrrolidine dithiocarbamate (APDC) and transferred into a minute volume of toluene as extractant employing lab-in-syringe magnetic stirring-assisted dispersive liquid-liquid microextraction (LIS-MSA-DLLME). After discharge of the sample, the analytes were back-extracted into nitric acid and injected on-line into ICP-OES. To promote and expedite this process in-syringe, advantage was taken from oxidative decomposition of the chelate by potassium iodate, reported in this article for the first time.

Experimental conditions for LIS-MSA-DLLME were optimized by Box-Benkhen multivariate analysis using the geometric mean of analyte recoveries as the desirability function. Times of extraction and back-extraction of 300 s and 100 s, respectively, pH 5.5 at 30 mmol/L acetate, 300 µL of extraction solvent, and 600 µmol/L of APDC were finally applied. Online interfacing to ICP-OES for back-extract analysis yielded average repeatabilities for Cd, Cu, and Pb of 2.9 %, 3.5 %, and 3.5 % with limits of detections (3s) of 1.9, 1.4, and 5.6 ng/mL, respectively. Oxidative back-extraction was proven reliable for the determination of metal species in coastal seawater, surrogate digestive fluids and soil leachates with recovery values for Cd, Cu, and Pb ranging from 90 % to 118 %, 68 % to 104 %, and 86 % to 112 %, respectively.

Graphical Abstract