Application of electrodeposition to elemental separation in atomic spectrometry

Jan Knápek

Two systems for preconcentration of cadmium were studied, one with electrochemical microcell and graphite rod electrodes, the second with large surface area electrode from reticulated vitreous carbon (RVC), an open-pore foam material of honeycomb structure. Cadmium electrolytic deposited on the carbon electrode was dissolved in 100 μ l of 0.1 mol l⁻¹ HNO₃ and transported by nebuliser capillary to air – acetylene flame. Although deposition of cadmium in first microcell was increased by coating graphite rod electrode with bismuth film, thus higher sensitivity of determination was achieved by application of RVC electrode. The optimal conditions of electrolysis, such as pH of solution, deposition current and flow-rate of sample solution, were evaluated by using method of artificial neural network. The arrangement with RVC electrode was employed for determination of cadmium in waters with high content of salts. Using this arrangement detection limit of 10 μ g l⁻¹ Cd was attained.

A novel method for determination of heavy metals in high saline matrix is presented. The method is based on the electrodeposition of metals and subsequent laser ablation coupled to inductively coupled plasma optical emission spectrometry (ICP-OES). Three arrangements for electrodeposition, two with stationary working electrode with placement of the electrodes in the cell face-to-face or with position of working electrode at the bottom of electrochemical cell and one with rotating working electrode; the use of different working electrode materials and suitable conditions for electrodeposition of Cd, Cr and Cu (pH, deposition current, time of electrolysis) from high salt matrices were studied. Nickel was chosen as the optimal electrode material. The current 10 mA, pH = 4.8, the speed of stirring 300 rpm for stationary or 196 rpm for rotating electrode arrangement and 10 min deposition time were selected as optimum. After electrodeposition the metal accumulated on the surface of electrode was evaporated/ablated by Nd:YAG laser into ICP-OES spectrometer. The detection limits were 0.13 mg L⁻¹Cd, 0.15 mg L⁻¹Cu and 1.9 mg L⁻¹ Cr for arrangement with stationary bottom working electrode and 0.25 mg L⁻¹ Cd, 0.05 mg L⁻¹ Cu, 0.8 mg L⁻¹ Cr for arrangement with rotating electrode. The precision expressed as RSD was in range 3.8-10.3%. Analysis of waste water was performed by standard addition method.