Abstract

This master thesis deals with the development of electrophoretic methods for determination of tantalum in recycled coolants used in the production of tantalum capacitors, as well as the separation of tantalum from chemically similar niobium. Developed methods are based on the formation of negatively charged complexes of tantalum and niobium with a number of ligands added to the separation electrolyte, especially tartaric and citric acid and 4-(2-pyridylazo)resorcinol. The separations were performed in a fused silica capillary of the inner diameter of 50 μm and the effective length of 36.5 cm, a negative voltage of –25 kV was applied to the capillary. Using a separation electrolyte containing 0.1 $\mathrm{mol \cdot l^{-1}}$ of tartaric and citric acid at pH = 2, separation of niobium and tantalum was achieved in less than 3 minutes; the absorption spectrometric detection was carried out in the ultraviolet region at a wavelength of 200 nm, the detection limit was 2.8 ppm of tantalum, and the quantification limit was 9.2 ppm of tantalum. Addition of 4-(2-pyridylazo)resorcinol reduced the detection limit to 0.8 ppm of tantalum and the quantification limit to 2.6 ppm of tantalum; the electrolyte contained 0.1 $\mathrm{mmol \cdot l^{-1}}$ of 4-(2-pyridylazo)resorcinol, 10 $\mathrm{mmol \cdot l^{-1}}$ of tartaric acid, 10 $\mathrm{mmol \cdot l^{-1}}$ of citric acid, and 25 $\mathrm{mmol \cdot l^{-1}}$ of ammonia to adjust the pH value between 6 and 7. Under these conditions, the separation of niobium and tantalum took 4 minutes, and the absorption spectrophotometric detection was performed in the visible region at a wavelength of 540 nm. The addition of niobium and tantalum standards has enabled the both metals detection in matrices of the real samples of recycled coolants by both developed methods, making these fast and simple electrophoretic methods convenient for application in real coolant production and recycling operations.

Key words

Tantalum, niobium, coolants, capillary electrophoresis, complexes