ABSTRACT

Part 1

Separation and determination of Zr(IV) and Hf(IV) as ternary complexes by high performance liquid chromatography

A method for the simultaneous separation and determination of Zr(IV) and Hf(IV) as ternary complexes formed with 2-(5-bromo-2-pyridylazo)-5-[N-propyl-N-(3-sulfopropyl) amino] phenol (PAPS) and fluoride was developed using ion interaction reversed-phase high-performance liquid chromatography (HPLC) on a C_{18} bonded phase column. Mobile phase compositions and pre-column complex formation conditions were optimised. Under the optimum conditions, the Zr(IV) and Hf(IV) complexes were eluted within 8 min with a mobile phase of methanol-water (65% v/v) containing 0.05 mM fluoride, 5 mM tetrabutylammoniumbromide (TBABr) and 10 mM acetate buffer at pH 5, with detection at 585 nm. The percentage relative standard deviation of retention times, peak area and peak height for Zr(IV) and Hf(IV) complexes were 0.60, 0.75, 1.37, 0.08, 1.01 and 1.70, respectively. The detection limits (signal to noise ratio = 3) for Zr(IV) and Hf(IV) were 0.38 µg L^{-1} and 1.53 µg L^{-1}, respectively. The method was applied to the analysis of Zr(IV) and Hf(IV) in rock sample.

ABSTRACT

Part 2

Determination of cations in fertilizers by capillary electrophoresis

Capillary electrophoresis was developed on fused silica column for the separation of NH_4^+, K^+, Ca^{2+}, Na^+, Mg^{2+}, Mn^{2+}, Co^{2+} and Cd^{2+}. Indirect UV method was used at 206 nm as the detection wavelength. To obtain the highest sensitivity without any peak interference, the applied voltage and composition of background electrolyte using amino acid buffer were investigated. The separation of eight cations was successful in 1.6 min. The relative standard deviation of NH_4^+, K^+, Ca^{2+}, Na^+, Mg^{2+}, Mn^{2+}, Co^{2+} and Cd^{2+} were in the range of 0.10-0.25% for migration time, 0.15-0.35% for peak area and 0.30-0.94% for peak height. Detection limits (determined by a signal to noise ratio of 3) of these ions were in the range 0.02-0.20 mg L^{-1}. The developed method was applied to determine the cationic nutrients in solid and fluid fertilizer samples. The results obtained from the capillary electrophoresis method agreed well with the flame atomic absorption spectrometry method.