

Highly Sensitive Instrumental Analytical-Method for the Simultaneous Determination of Principal Components in Salts

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Summary

We investigated a capillary zone electrophoresis (CZE) method with a capacitively contactless conductivity detector (C⁴D) for the determination of major constituents, such as Na⁺, K⁺, Mg²⁺, Ca²⁺, Cl⁻, and SO₄²⁻, in salts. A polyimide-coated fused-silica capillary with 72 cm total length (50 cm effective length) and 75 μm id (375 μm od) was used. Applied voltage was set at 20 kV with the sample inlet side as the anode for cations and the cathode for anions. The background electrolyte (BGE) was a mixture of 25 mM 2-(*N*-morpholino)ethanesulfonic acid (MES), 25 mM L-histidine, 2 mM 18-crown-6, and 0.01% m/v hydroxypropylmethylcellulose (HPMC). A sample was vacuum-injected (50 kPa) into the CZE apparatus for 3 s (33 nL). Linear calibration graphs were obtained using peak area; correlation coefficient, $r = 0.9975$ – 1.000 . The values of the relative standard deviation (RSD, $n = 5$, intra-day) of migration time, peak area, and peak height were, respectively, 0.25–0.62%, 0.79–5.3%, and 0.31–4.3%. The limits of detection (LODs, $S/N = 3$) and the determination limits ($S/N = 10$) were 0.11 and 0.36 mg/L for Na⁺, 0.012 and 0.040 mg/L for K⁺, 0.028 and 0.093 mg/l for Mg²⁺, 0.014 and 0.047 mg/l for Ca²⁺, 0.11 and 0.36 mg/l for Cl⁻, and 0.015 and 0.050 mg/L for SO₄²⁻-S, respectively. The solutions of four kinds of salt (0.1 g/L) were analyzed using the proposed procedure. The LOQs for Na⁺ and Cl⁻ were sufficiently low, but the accuracy should be improved. The peaks for K⁺, Ca²⁺, and SO₄²⁻ were detected, but the LODs and LOQs were required to be improved. The Mg²⁺ peak was not separated from the Na⁺ peak. We intend to examine kinds of BGE and electrokinetic injection (EKI) for a sample solution to solve above problems and then to establish the sensitive CZE method for the simultaneous determination of the principal components in salts.