

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 contactless conductivity detector (CCD) for the determination of major constituents, such as Na^+ , K^+ , Mg^{2+} , Ca^{2+} , Cl^- , and SO_4^{2-} , in salts. The total capillary length was 80 cm and its inner diameter was 50 μm . The position of the CCD was freely changeable. Applied voltage was set at 20 kV with the sample inlet side as the anode for cations and the cathode for anions. The following background electrolyte (BGE) was used: (A) a mixture of 9 mM L-histidine, 15 mM malic acid, and 2 mM 18-crown-6 and (B) a mixture of 25 mM 2-(*N*-morpholino)ethanesulfonic acid (MES), 25 mM L-histidine, and 2 mM 18-crown-6. The difference between the migration time for Ca^{2+} and that for Na^+ using the BGE (B) was larger than that using the BGE (A). The BGE (B) is suitable for salts analysis. Linear calibration graphs for peak area were obtained when the standard solutions of cations and anions were separately analyzed using the BGE (B) and vacuum injection method (sample injection period 3 s (33 nL)); correlation coefficient, $r=0.9146-0.9984$ for cations and $r=0.9970-0.9996$ for anions. The limits of detection (LODs) were 0.012–0.023 mg/l for cations and 0.009–0.045 mg/l for anions at a signal-to-noise ratio of three. The values of the relative standard deviation (RSD, $n=3$, intra-day) of migration time, peak area, and peak height were, respectively, 0.0–0.31%, 0.20–5.8%, and 0.10–3.0% for cations (0–1.0 mg/L) and 0.10–0.48%, 1.2–4.5%, and 1.5–2.5% for anions (0–4.5 mg/L Cl^- , 0–0.56 mg/l SO_4^{2-} –S). The solution of commercially available salt (0.1 g/L) was vacuum injected into the apparatus for 1 s and 3 s. When the injection period was 1 s, the high Na^+ peak and low K^+ and Ca^{2+} peaks were observed with base line separation but Mg^{2+} was not detected. When the injection period was 3 s, the higher Na^+ and K^+ peaks were observed but Ca^{2+} peak became broad. Mg^{2+} was not detected. When the standard solutions were analyzed using electrokinetic injection (EKI), the RSDs were inferior to those for vacuum injection. In addition, linear calibration graphs could not be obtained both for cations and anions using EKI. We intend to examine sample injection procedure, CCD position, BGE component, salt concentration to establish the sensitive CZE for the simultaneous determination of the principal components in salts.