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Note

Determination of nitrate, chloride and sulphate in drinking water by capillary free-zone electrophoresis*

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Zone electrophoresis in stabilizing media has been a frequently used analytical technique from its inception¹ to the present time². Zone electrophoresis in free solutions still lacks wide practical utilization, despite its high separation potential shown with the use of both rotating tubes³ and capillary columns with model mixtures^{4,5}.

The aim of this paper is to demonstrate the practical application of free zone electrophoresis in an isotachophoretic device using a capillary with on-line potential gradient detection for the rapid and sensitive determination of nitrate, chloride and sulphate in drinking water, which is an analytical problem of permanent interest⁶.

EXPERIMENTAL

The experiments were carried out in an isotachophoretic column⁷ consisting of a block of Perspex with electrode chambers, injection port, control valves and sensing electrodes of the potential gradient detector. The separations were performed in a capillary of rectangular cross-section (1.0 × 0.2 mm), 200 mm long, created by a groove in the organic glass block and covered with PTFE foil pressed on the block with a thermostated metal plate. A high-voltage constant-current supply controllable up to 400 μ A at a maximum of 16 kV was used.

Potential gradients were detected by a device⁸ consisting of a voltmeter with a high input resistance. The baseline signal corresponding to the background electrolyte (approximately 3 V) was electrically compensated on the output side of the voltmeter. The changes in the output signal (*ca.* 100 mV) were recorded by means of a Servogor RE 571 line recorder (Goertz, Vienna, Austria). In order to decrease the detection limit of the anions being analysed, the noise of the signal was suppressed by a low-pass filter (band width d.c. to 0.1 Hz) on the input of the voltmeter. In this way, the useful signal-to-noise ratio was increased approximately three-fold.

For selectively influencing the effective mobilities of the separated anions, complex formation with Cd^{2+} as the counter ion⁹ was employed. A solution of cadmium acetate served as the background electrolyte; it was generated in the separation

* Dedicated to Clark Hamilton, the founder of the Scientific Exchange Agreement, supporting research in chromatography and related methods in Europe.

capillary by an anionic isotachophoretic run with 0.004 *M* cadmium nitrate as the leading electrolyte and 0.01 *M* acetic acid as the terminator. After the isotachophoretic boundary had passed the detector, zone electrophoresis was performed in the adjusted terminator. The driving current was 120 μ A in all experiments.

The chemicals used were of analytical-reagent grade (Lachema, Brno, Czechoslovakia). Solutions of nitrate, chloride, sulphate and nitrite (10^{-3} *M*) were sampled by means of a 1- μ l microsyringe (Hamilton, Whittier, CA, U.S.A.).

RESULTS AND DISCUSSION

Fig. 1a shows the record of the analysis of a model mixture containing $5 \cdot 10^{-10}$ mol of nitrate, chloride, sulphate and nitrite. It can be seen that complete resolution and detection were achieved within 5 min. Fig. 1b shows the record of the analysis of a real sample of drinking water, containing 22.9, 30.4 and 62.2 mg/l of nitrate, chloride and sulphate, respectively.

For quantitation, peak areas of the potential-gradient detection signal were used. The calibration graph for sulphate, nitrate and chloride in Fig. 2 shows a linear dependence of the peak area on the amount sampled in the range 0.1–0.7 nmol. The relative standard deviation was 2% for nitrate at the 0.5 nmol level (five analyses).

In conclusion, it can be said that currently used equipment for analytical capillary isotachopheresis can be used successfully for free zone electrophoresis. It allows the simple and rapid determination of common anions in various aqueous

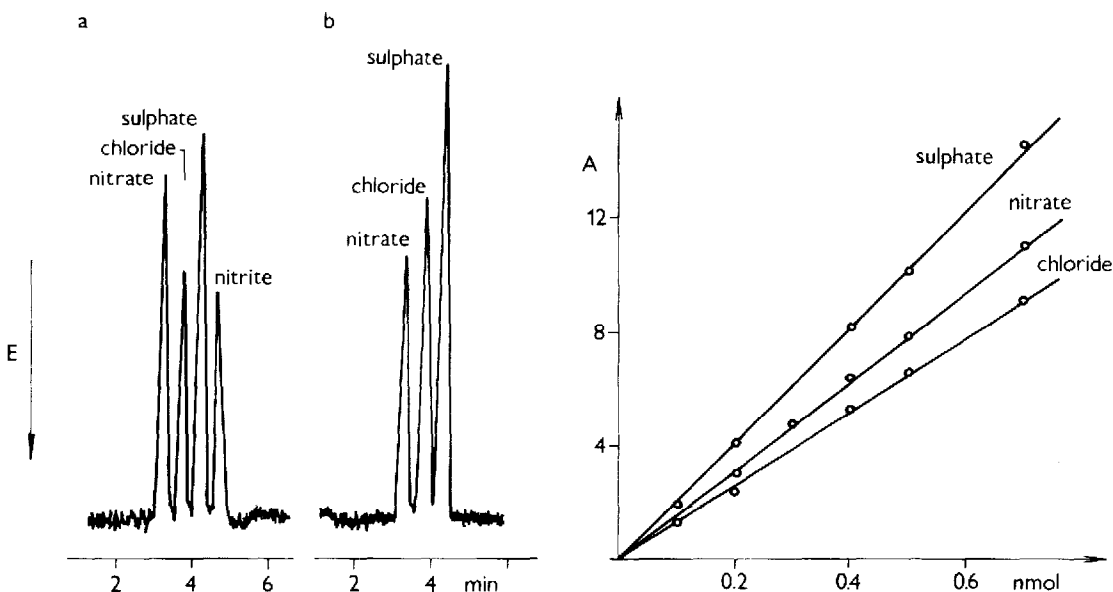


Fig. 1. Potential gradient records of zone-electrophoretic analyses of anions. Sample: (a) 0.50 μ l of 10^{-3} *M* nitrate, chloride, sulphate and nitrite; (b) 1.00 μ l of drinking water.

Fig. 2. Calibration graphs [peak area in arbitrary units (*A*) versus amount sampled] for sulphate, nitrate and chloride.

samples, the detection limits being at the 10 pmol level for each of the anions. A recent paper⁶ on non-suppressed ion chromatography shows lower detection limits, but the zone electrophoretic analysis described here is much faster, with analysis times of less than 5 min.

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