

SIMULTANEOUS DETERMINATION OF INORGANIC ANIONS AND CATIONS IN WATERS WITH AN ION CHROMATOGRAPHY METHOD

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1. Introduction

Ion chromatography is a technique for separation and automatic measurement of ions in solution. Measurement consists of injecting a sample into an eluent that passes through an ion exchange column followed by a suppressor unit and a conductivity detector.

2. Objective

The purpose of the present study is the development, optimisation and validation of an ion chromatography analytical method for the simultaneous determination of 13 anions (F⁻, HCOO⁻, CH₃COO⁻, ClO₂⁻, Cl⁻, NO₂⁻, CO₃²⁻, SO₄²⁻, Br⁻, C₂O₄²⁻, NO₃⁻, ClO₃⁻ & PO₄³⁻) and 6 cations (Li⁺, Na⁺, NH₄⁺, K⁺, Ca²⁺ & Mg²⁺) - hereinafter Target Analytes (TAs). The method was applied to environmental samples from Greece. The determined values of the TAs were recorded for monitoring reasons as well as for the evaluation of their levels in accordance with applicable legislation and their potential danger (e.g., toxicity) from a Public Health and Environment protection point of view.

3. Methodology

The Thermo Scientific Dionex ICS-6000 HPIC system was used, allowing the separate analysis in two independent channels (e.g., anions and cations). In search of the best possible result and taking into consideration the simultaneous presence of all TAs, different instrumental analytical conditions were examined (temperature, flow rate and eluent), concluding to 30°C column temperature, 1mL/min flow & 28.50 to 40.00 mM KOH for anions and 0.750mL/min & 6.00 to 20.00 mM MSA for cations.

A great challenge was the peak resolution between TAs usually existing in low concentrations (e.g., NO₂⁻ & NH₄⁺) and others existing in relatively high (CO₃²⁻ - SO₄²⁻ & Na⁺ respectively). Finally, the validated method was applied in Proficiency Testing samples giving satisfactory results and in more than 530 water samples.

4. Results & Discussion

The analysis time was limited to only 17min, allowing a satisfying chromatographic separation for all 19 TAs and the quantitative determination of 13 of them, i.e., F⁻, Cl⁻, NO₂⁻, SO₄²⁻, Br⁻, NO₃⁻, PO₄³⁻, Li⁺, Na⁺, NH₄⁺, K⁺, Ca²⁺ and Mg²⁺. The method was successfully validated for environmental water matrices (drinking water, natural mineral water, ground water & surface water) and for water used for preparation of dialysis fluids, fulfilling legislative orders for parametric values and performance characteristics as well. Therefore, the method is considered fit for purpose.

Regarding the samples analysed for the TAs, most of the results complied with the current EU and national legislation and there are only a few occasions of exceedance of parametric values.

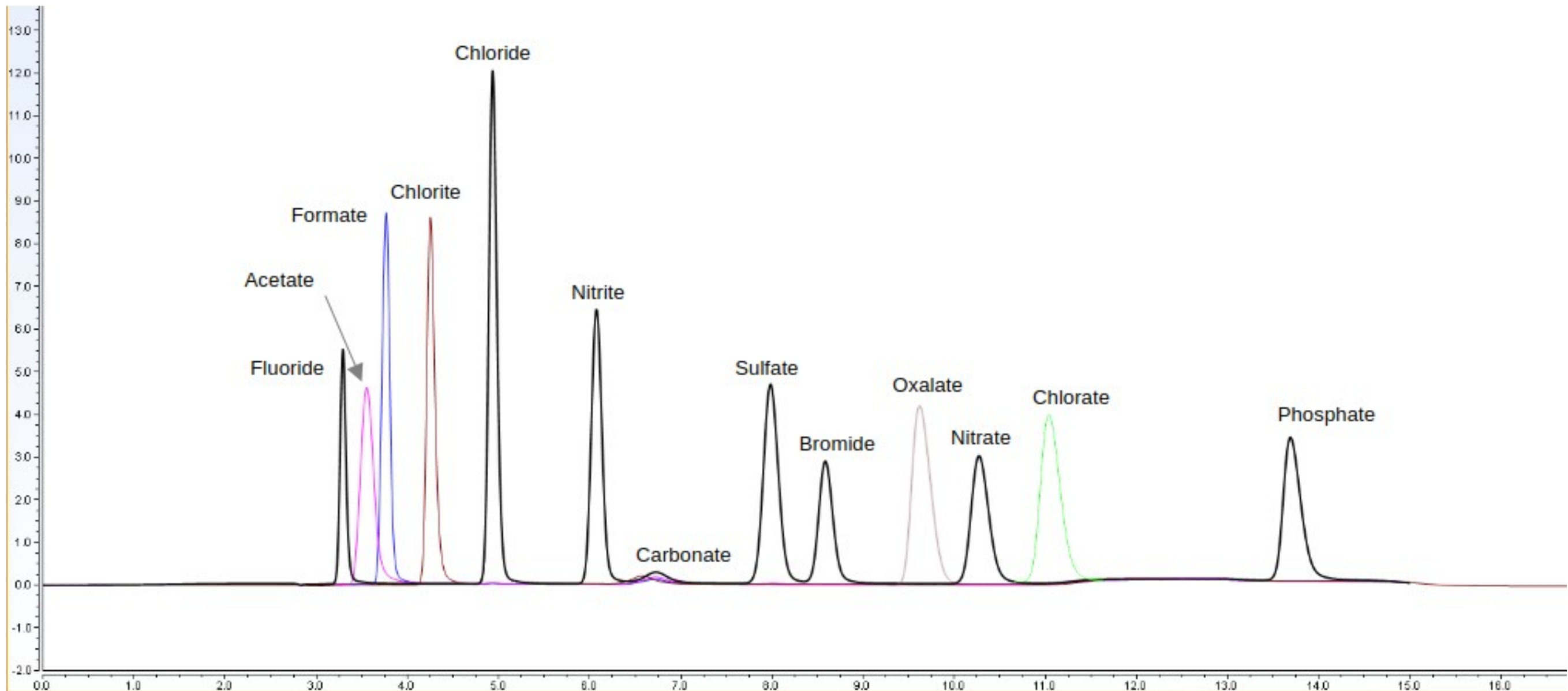


Figure 1: Chromatogram of the 13 Anions

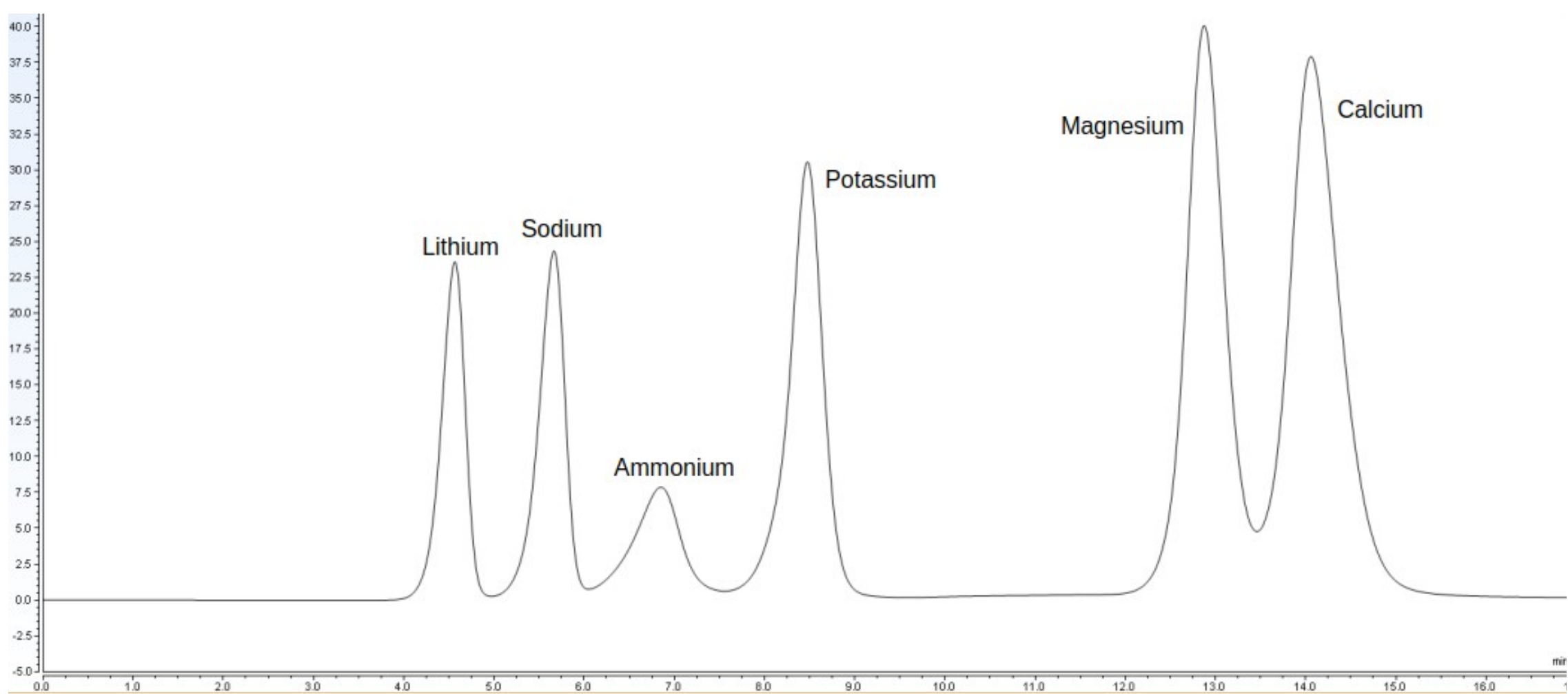


Figure 2: Chromatogram of the 6 Cations

Table 1: Range and Coefficient of determination

Target Analyte	Range (mg/L)	Coefficient of Determination
Fluoride (F ⁻)	0.2 - 10	0.99890
Chloride (Cl ⁻)	1 - 100	0.99993
Nitrite (NO ₃ ⁻)	0.03 – 1	0.99944
Sulfate (SO ₄ ²⁻)	5 - 100	0.99987
Bromide (Br ⁻)	1 - 100	0.99995
Nitrate (NO ₃ ⁻)	3 - 100	0.99990
Phosphate (PO ₄ ³⁻)	5 - 100	0.99949
Lithium (Li ⁺)	0.25 - 50	0.99996
Sodium (Na ⁺)	2 - 100	0.99997
Ammonium (NH ₄ ⁺)	0.05 - 2	0.99977
Potassium (K ⁺)	0.75 - 100	0.99996
Magnesium (Mg ²⁺)	0.5 - 100	0.99970
Calcium (Ca ²⁺)	1 - 100	0.99957

5. Conclusion

A quick, reliable and robust ion chromatography analytical method was validated, suitable for the simultaneous determination of 19 TAs in water matrices.

6. References

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