PRELIMINARY STUDY ON AN ION CHROMATOGRAPHIC METHOD FOR ANION DETERMINATION

ÉTUDE PRÉLIMINAIRE SUR UNE MÉTHODE CHROMATOGRAPHIQUE DE DÉTERMINATION D'ANIONS

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Ion chromatography is a well-established method for determination of anions in water samples. However, to ensure precise and reliable results the method should be optimized and validated in each laboratory.

This report presents the results from a preliminary study of an ion-chromatographic method for determination of fluoride, bromate, chloride, nitrite, bromide, nitrate, phosphate and sulphate in natural water. The method characteristic working range, linearity, limit of detection and limit of quantification were studied according to the international regulations for method validation.

An ion chromatograph equipped with autosampler, the samples filtration system with a 0.2 µm membrane, injection valve, high-pressure pump, suppressor module and conductivity detector was used. The separation was performed on Metrosep A Supp 7-250 (Metrohm) anion separation column, with 3.6 mmol/l Na₂CO₃ as eluent in isocratic mode at flow rate of 0.7 ml/min, temperature 45 °C and injected volume of 20 µl.

The obtained experimental results showed that at the selected conditions the ion chromatographic method allowed determination of low concentration of targeted anions in waters. The limit of detection (LOD) were 0.7, 7, 10, 6, 2, 0.8 and 6 µg L⁻¹ for F⁻, BrO₃⁻, Cl⁻, NO₂⁻, Br⁻, NO₃⁻ and PO₄³⁻, respectively. LOD was calculated as: LOD = 3.3 × s₀', where s₀' is the standard deviation: s₀' = s₀/√n. s₀ is the estimated standard deviation of m single results at or near zero concentration.

The limit of quantification (LOQ) were found to be 0.002, 0.01, 0.02, 0.01, 0.007, 0.002 and 0.01 mg L⁻¹, respectively for the same anions. LOQ was calculated as: LOQ = kQ × s₀', where factor kQ value is 10 by IUPAC to RSD of 10 % and 5 or 6 to RSD value of 20% and 17% respectively.

Acknowledgments: This study has been financially supported by the Operational Programme "Science and education for smart growth" 2014-2020 of the European Union cofounded by the European Social Fund through the project BG05M2OP001-2.009-0015

Topic: Environmental; Poster presentation