

Advantages of multidimensional ion chromatography for trace analysis

R. Odermatt¹, T. Rönneval² and J. Kleimann¹

Summary

The analytical challenge treated in the present work consists in detecting trace concentrations (ppb) of bromide in the presence of a strong chloride matrix. This problem was overcome by separating the bromide ions from the main fraction of the early eluting chloride matrix (several g/L) by applying two sequential chromatographic separations on the same column. After the first separation, the main fraction of the interfering chloride matrix is flushed to waste, while the later eluting anions are diverted to an anion-retaining preconcentration column. After elution in counter flow, the bromide ions are efficiently separated from the marginal chloride residues. The four-point calibration curves for bromide and sulfate are linear in the range of 10...100 µg/L and 200...800 µg/L and yield correlation coefficients of 0.99988 and 0.99953, respectively.

For the method shown here, a second injection valve and a preconcentration column are the only additional devices needed to master this demanding separation problem.

Introduction

Multidimensional chromatography techniques are extremely powerful tools for solving demanding separation problems. Interest in these techniques is therefore increasing in all fields of chromatography.

Ionic standards have to comply with exacting purity requirements. For the preparation of such standards it is of paramount importance to quantify impurities in the feedstock used. However, the main anionic component of the solution (typical concentration 10 g/L) produces an extremely large peak with severe tailing. All ions with higher retention times are affected by the elution behavior of the main component. They are either hardly detectable under the peak tail of the main component or they elute as ill-defined humps on top of a drifting baseline. All this adversely affects the limit of detection and the precision of the determination.

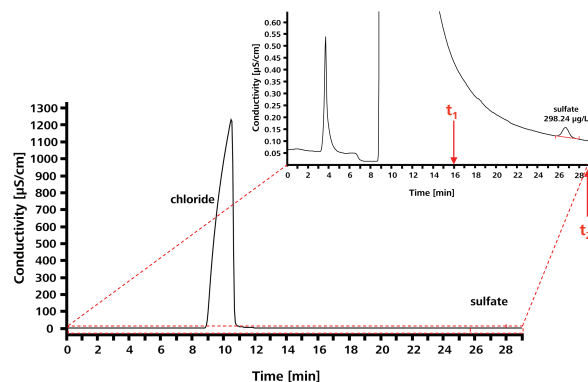
This presentation highlights the advantages of multidimensional ion chromatography (IC) techniques in typical trace analyses. While classical approaches in multidimensional separations involve the use of different columns and detectors, we will present a technique that can be performed with any ion chromatograph and needs hardly any additional equipment.

Instrumentation

- 850 Professional IC Anion – MCS – Prep 2
- 858 Professional Sample Processor – Pump
- Metrosep A Supp 16 – 250



Conventional IC – chromatogram

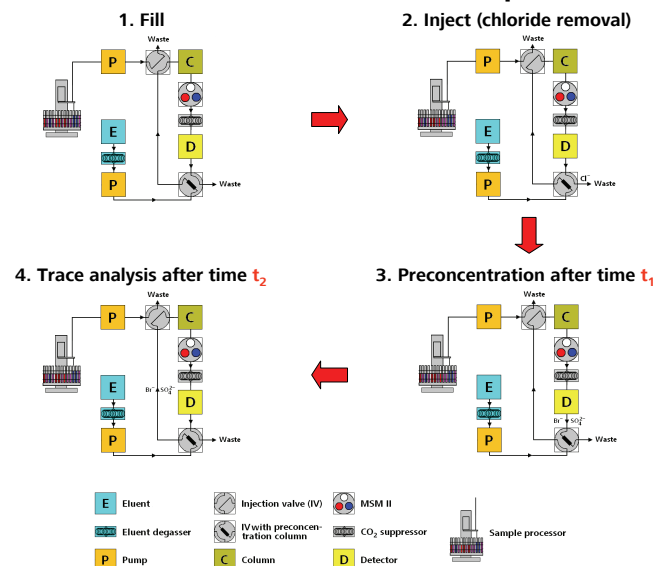


Sample: 10 g/L NaCl in ultrapure water
Column: Metrosep A Supp 16 – 250
Eluent: 7.5 mmol/L Na₂CO₃
0.75 mmol/L NaOH

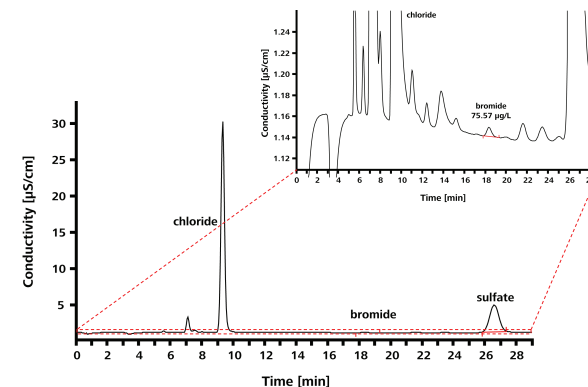
Flow: 0.8 mL/min
Loop: 20 µL

Bromide elutes with the chloride matrix and can thus not be accurately determined.

Multidimensional IC – setup



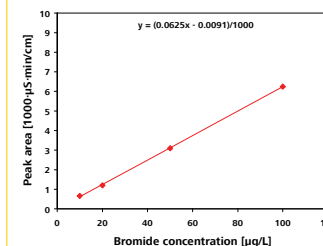
Multidimensional IC – chromatogram



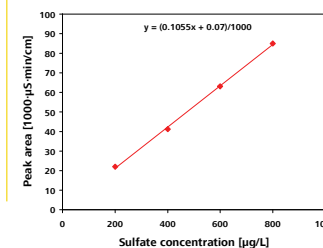
Chromatographic conditions correspond to those mentioned for conventional IC. By switching the injection valve at the defined time intervals t_1 and t_2 ,

- a large excess of the chloride matrix is efficiently removed allowing for a
- bromide and sulfate determination at ppb levels.

Calibration



Bromide	Concentration [µg/L]	Peak area [1000-µS-min/cm]
Standard 1	10	0.66
Standard 2	20	1.20
Standard 3	50	3.10
Standard 4	100	6.25
Correlation coefficient	0.99988	
RSD ¹	1.728%	



Sulfate	Concentration [µg/L]	Peak area [1000-µS-min/cm]
Standard 1	200	21.96
Standard 2	400	41.22
Standard 3	600	63.11
Standard 4	800	85.00
Correlation coefficient	0.99953	
RSD ¹	2.167%	