

Summary

The analytical challenge treated in the present work consists in the determination of chloride, phosphate and sulfate in the presence of difficult sample matrices that interact with the stationary column phase or even render it unusable. Metrohm's patented stopped-flow dialysis coupled to the new 881 Compact IC pro ion chromatograph overcomes these drawbacks.

Two standard solutions covering the concentration ranges 1.0...3.6 mg/L and 10...36 mg/L as well as two samples, an ultra-high temperature (UHT) processed milk and a baby milk powder, were characterized in terms of analyte concentration, relative standard deviation, calibration quality, carryover and recovery rates. While the five-point calibration curves yielded correlation coefficients (R) better than 0.9999, carryover (between two subsequent injections of a concentrated sample and a blank) was less than 0.49%. Recoveries for the low (10...36 mg/L) and high standard concentrations (1.0...3.6 mg/L) were within 91...99% and 94...100%, respectively.

Automated compact stopped-flow dialysis is a leading-edge sample preparation technique that ensures optimum separation performance by effectively protecting the column from detrimental matrix compounds.

Introduction

Ion chromatography (IC) as an analytical technique has experienced an enormous surge in popularity partly due to the simplicity and robustness of the method, the improved reliability and the great choice of columns, detectors and applications. For a sample in a homogeneous ionic form, very little sample preparation is required and results can be obtained within minutes. In complex matrices carrying high organic loads such as waste water, soil eluates or dairy products, a more extensive sample preparation is mandatory to prevent destruction of the column.

Although numerous sample preparation techniques have been developed, such as the Carrez precipitation for protein-containing samples, most of them are tedious and error-prone. To overcome these shortcomings, Metrohm launched the first coupling of IC with dialysis in 1997. Since then the procedure has been further improved and allows for an efficient inline elimination of undesired matrix components in a variety of demanding sample types.

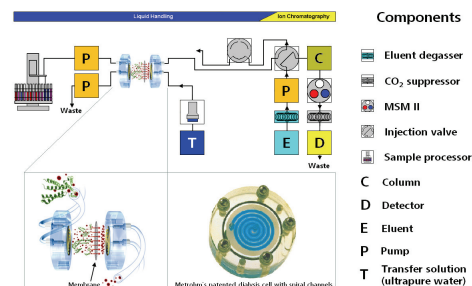
Using as examples an ultra-high temperature (UHT) processed milk and a baby milk powder sample, this poster presents a fully automated sample preparation setup coupled to the new ion chromatograph 881 Compact IC pro.

System setup

- 881 Compact IC pro
- 858 Professional IC Sample Processor
- 800 Dosino
- Dialysis equipment



Compact stopped-flow dialysis



sample's chromatogram, the overall analysis time is not prolonged.

Whereas in the conventional setup 2 two-channel peristaltic pumps transport the sample and the acceptor solution to and from the dialysis cell, in compact dialysis a Dosino doses ultrapure water through the acceptor compartment of the cell. The stopped-flow status is achieved by stopping the Dosino and blocking the outlet capillary of the cell by feeding it through the valve of the 858 Professional IC Sample Processor. The latter, depending on its valve position, allows or blocks the acceptor solution flow.

System characteristics

Carryover

Carryover was evaluated by injection of a blank (ultrapure water) immediately after injection of a standard.

	Fluoride	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate
Low standard concentration ^a	0.24	0.15	0.17	0.20	0.18	0.11	0.28
High standard concentration ^a	0.49	0.12	0.13	0.22	0.11	0.00	0.38

^amean of four determinations

Calibration

	Fluoride	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate
Correlation coefficient ^a	0.99995	0.99996	0.99999	0.99996	0.99994	0.99990	0.99997
RSD [%]	1.516	1.242	0.834	1.169	1.479	2.491	1.176

^aeach calibration curve is based on five different standard concentrations

Recovery rates

In order to determine recovery rates, results obtained by direct injection were compared to those obtained by injection of the dialyzate.

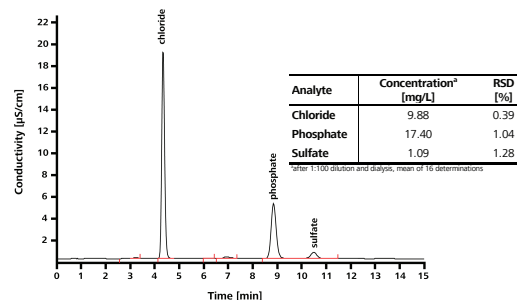
	Low standard concentration			High standard concentration		
	Direct injection	With dialysis	Recovery ^c	Direct injection	With dialysis	Recovery ^c
	Mean ^a	RSD		Mean ^a	RSD	
	[mg/L]	[%]		[mg/L]	[%]	
Fluoride	1.06	0.12	0.24	97.2	10.81	0.09
Chloride	3.01	0.04	2.97	0.03	31.58	0.03
Nitrite	2.94	0.32	2.91	0.15	30.01	0.30
Bromide	1.02	0.08	1.01	0	10.50	0.04
Nitrate	3.02	0.07	2.97	0	30.80	0.03
Phosphate	3.81	0.17	3.47	0.10	33.74	0.02
Sulfate	3.52	0.09	3.35	0.07	35.57	0.04

^amean of three determinations
^bmean of nine determinations

^cconcentration dialyzate/concentration direct injection × 100

Examples

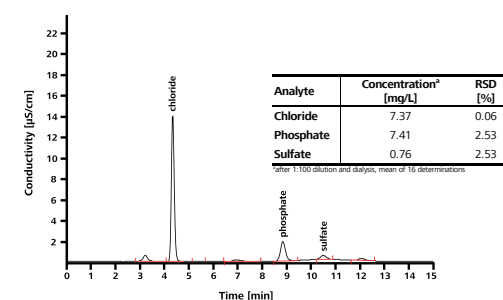
UHT milk sample



Sample prep.: 1:100 dilution, dialysis
Column: Metrosep A Supp 5 – 100
Column temp.: 30 °C
Eluent: 3.2 mmol/L sodium carbonate
1.0 mmol/L sodium hydrogen carbonate

Acceptor sol.: Ultrapure water
Dialysis time: 14 min
Flow: 0.7 mL/min
Loop: 20 µL

Baby food milk powder



Sample prep.: 1:100 dilution of the prepared milk, dialysis
Column: Metrosep A Supp 5 – 100
Column temp.: 30 °C
Eluent: 3.2 mmol/L sodium carbonate
1.0 mmol/L sodium hydrogen carbonate

Acceptor sol.: Ultrapure water
Dialysis time: 14 min
Flow: 0.7 mL/min
Loop: 20 µL