Automated dialysis as a sample preparation tool in ion chromatography

S. Rick and A. Wille

Ion Chromatography

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.

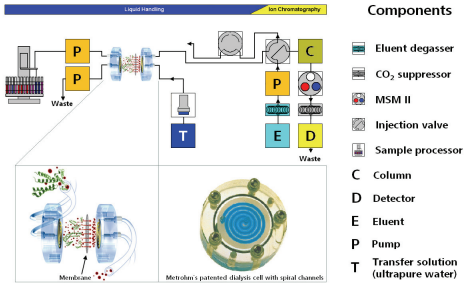
Using as examples an ultra-high temperature (UHT) processed milk and a baby milk powder sample, this poster presents a fully automated sample preparation setup coupled to the new ion chromatograph 881 Compact IC pro.

System setup

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

Compact stopped-flow dialysis

Dialysis is based on the selective diffusion of molecules or ions from one liquid (donor or sample solution) to another (acceptor solution) via a membrane. The driving force for the transfer is the concentration gradient across the membrane. Contrary to dynamic dialysis, where two solutions continuously pass through the dialysis module, at least one solution is temporarily stopped until the concentration in the acceptor solution is the same as that in the donor solution. This patented stopped-flow procedure takes between 10 and 14 minutes and can be directly coupled to an IC setup. As the dialysis is performed during the recording of the previous 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

[a]mean of four determinations

Calibration

	Anion[a]						
	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

[a]each calibration curve is based on five different standard concentrations

Recovery rates

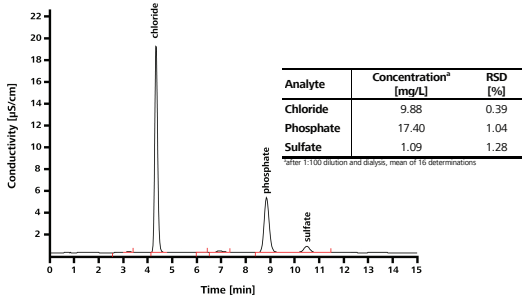
In order to determine recovery rates, results obtained by direct injection were compared to those obtained by injection of the dialyzate.

	Low standard concentration					High standard concentration				
	Direct injection		With dialysis		Recovery[c]	Direct injection		With dialysis		Recovery[c]
	Mean[a] [mg/L]	RSD [%]	Mean[b] [mg/L]	RSD [%]	[%]	Mean[a] [mg/L]	RSD [%]	Mean[b] [mg/L]	RSD [%]	[%]
Fluoride	1.06	0.12	1.03	0.24	**97.2**	10.81	0.09	10.57	0.06	**97.8**
Chloride	3.01	0.04	2.97	0.03	**98.7**	31.58	0.03	31.22	0.04	**98.9**
Nitrite	2.94	0.32	2.91	0.15	**99.0**	30.01	0.30	29.81	0.17	**99.3**
Bromide	1.02	0.08	1.01	0	**99.0**	10.50	0.04	10.38	0.03	**98.9**
Nitrate	3.02	0.07	2.97	0	**98.3**	30.80	0.03	30.40	0.03	**98.7**
Phosphate	3.81	0.17	3.47	0.10	**91.1**	33.74	0.02	31.83	0.07	**94.3**
Sulfate	3.52	0.09	3.35	0.07	**95.2**	35.57	0.04	34.17	0.07	**96.1**

[a]mean of three determinations
[b]mean of nine determinations
[c]concentration dialyzate/concentration direct injection ×100

Examples

UHT milk sample

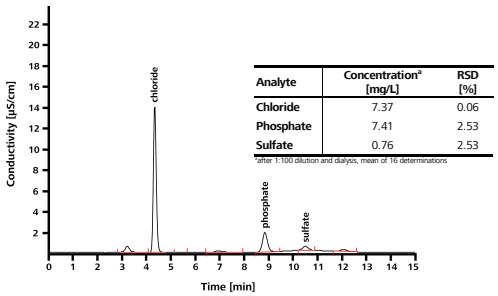


Analyte	Concentration[a] [mg/L]	RSD [%]
Chloride	9.88	0.39
Phosphate	17.40	1.04
Sulfate	1.09	1.28

[a]after 1:100 dilution and dialysis, mean of 16 determinations

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



Analyte	Concentration[a] [mg/L]	RSD [%]
Chloride	7.37	0.06
Phosphate	7.41	2.53
Sulfate	0.76	2.53

[a]after 1:100 dilution and dialysis, mean of 16 determinations

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

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Metrohm AG, CH-9101 Herisau/Switzerland, ric@metrohm.com, phone +41 71 353 87 68