Liquid handling applied to automated sample preparation in liquid chromatography

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Ion Chromatography

Summary

In routine chemical analysis, the predominant challenge involves a higher sample throughput, improved reproducibility, liquid handling flexibility and reduced personnel costs. In response to these requirements, the 872 Extension Module Liquid Handling in combination with the MagIC Net™ software and the well-proven Dosino technology expands the possibilities of inline sample preparation and opens up new fields of application. The module comprises an additional dual channel peristaltic pump for conveying any type of liquid in the low-pressure range, a six-port injection valve that can be used both in the high-pressure and in the low-pressure range as well as a ten-port selector valve. Among others, the module can be used, together with an optional mixing vessel, for pH adjustments, pre-column derivatizations, or the mixing of solutions.

As a representative of an inline sample preparation technique, this poster describes the performance of precise dilutions. By using only one single stable standard solution, multi-point calibration curves can be automatically recorded by diluting a concentrated standard in an external vessel. Carryover between two subsequent injections demonstrated by means of a 10 mg/L sample and blank injection was not observed. In the range of 0.2…10 mg/L, six-point calibration curves yield correlation coefficients (R) of 1 while relative standard deviations were smaller than 0.15%.

Introduction

Sample preparation plays an important role in all fields of liquid chromatography. While in the past a lot of tedious manual steps were required, nowadays automated sample preparation is gaining importance.

State-of-the-art equipment for automated sample preparation usually allows filtration, solid-phase extraction and dilution. More recently, ultrafiltration, intelligent dilution, preconcentration, combustion and dialysis have been made available as fully automated techniques.

In this presentation, we focus on liquid handling operations used for efficient automated sample preparation.

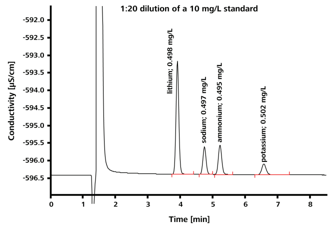
We discuss a so-called liquid handling module, that is a setup combining a selection manifold with dosing devices, reagent and mixing vessels, peristaltic pump and injection valve. In combination with an ion chromatograph, this system offers almost unlimited flexibility for all kinds of automated liquid handling operations.

Selected key applications involving, for example, dilution and/or calibration demonstrate the scope of liquid handling as applied to automated sample preparation.

Instrumentation

- 850 Professional IC AnCat
- 858 Professional Sample Processor
- 872 Extension Module Liquid Handling

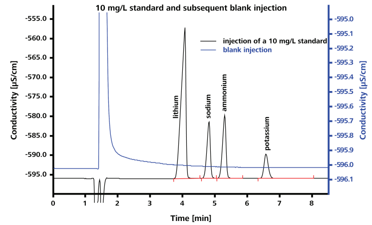
Separation conditions and carryover



After a 1:20 dilution of the 10 mg/L calibration standard, the sample was automatically injected.

All calibration standards for the required calibration range are carried out fully automatically by diluting the concentrated multi-ion standard in an external mixing vessel.

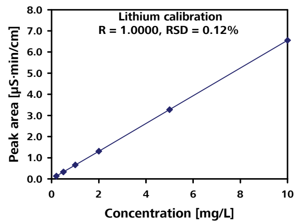
Column:	Metrosep C 4 - 150/4.0
Column temp.:	30 °C
Eluent:	2.5 mmol/L oxalic acid in ultrapure water
Dilution solution:	Ultrapure water
Flow:	0.9 mL/min
Loop:	20 µL



Carryover in the mixing vessel was evaluated by injection of a blank (ultrapure water) immediately after injection of a 10 mg/L standard.

Calibration and recovery

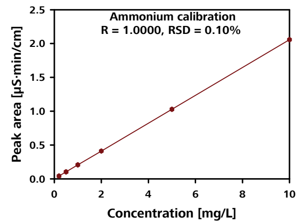
Lithium



		Dilution factor					
	Direct injection	1	2	5	10	20	50
Peak area[a] [$\mu S \cdot min \cdot cm^{-1}$]	6.5627	6.5508	3.2757	1.3084	0.6598	0.3308	0.1340
Recovery[b] [%]		99.82	99.83	99.69	100.54	100.81	102.09
RSD[c] [%]	0.02	0.02	0.23	0.82	0.85	0.73	0.80

[a]mean value of six determinations, [b]recovery = peak area (dilution)/peak area (direct injection)·dilution factor·100, [c]n = 6

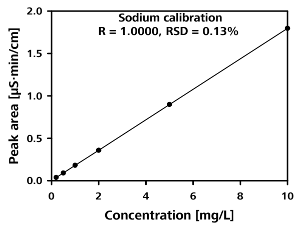
Ammonium



		Dilution factor					
	Direct injection	1	2	5	10	20	50
Peak area[a] [$\mu S \cdot min \cdot cm^{-1}$]	2.0620	2.058	1.0291	0.4117	0.2078	0.1041	0.0424
Recovery[b] [%]		99.81	99.82	99.83	100.78	100.97	102.81
RSD[c] [%]	0.001	0.02	0.22	0.67	0.79	0.79	1.28

[a]mean value of six determinations, [b]recovery = peak area (dilution)/peak area (direct injection)·dilution factor·100, [c]n = 6

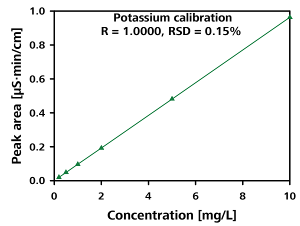
Sodium



		Dilution factor					
	Direct injection	1	2	5	10	20	50
Peak area[a] [$\mu S \cdot min \cdot cm^{-1}$]	1.7985	1.7950	0.8974	0.3585	0.1808	0.0908	0.0369
Recovery[b] [%]		99.81	99.79	99.67	100.53	100.97	102.59
RSD[c] [%]	0.001	0.02	0.23	0.77	0.86	0.65	0.81

[a]mean value of six determinations, [b]recovery = peak area (dilution)/peak area (direct injection)·dilution factor·100, [c]n = 6

Potassium



		Dilution factor					
	Direct injection	1	2	5	10	20	50
Peak area[a] [$\mu S \cdot min \cdot cm^{-1}$]	0.9625	0.9624	0.4810	0.1922	0.0969	0.0485	0.0198
Recovery[b] [%]		99.99	99.95	99.84	100.68	100.78	102.86
RSD[c] [%]	0.002	0.10	0.35	0.72	0.83	0.61	0.54

[a]mean value of six determinations, [b]recovery = peak area (dilution)/peak area (direct injection)·dilution factor·100, [c]n = 6

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