Metrohm

Automated ion chromatographic determinations over six orders of magnitude

N.H. Subramanian[1], T. Hartmann[2], and A. Wille[2]

Ion Chromatography

Summary

Metrohm`s intelligent Preconcentration Technique with Matrix Elimination (MiPCT-ME) excels in its capacity to perform automatic ion chromatographic determinations over 6 orders of magnitude. Crucial requirements for this are the system`s intelligence and the exact measurement of the sample volume. While the intelligence allows to compare results and take decisions, the dosing device takes over the high-precision liquid handling of even single-digit microliter volumes to the preconcentration column. By using only one analytical setup and without additional rinsing, samples containing both ultratraces and high concentrations can be analyzed.

As the other Metrohm Inline Techniques, the MiPCT-ME technique presented reduces the workload, ensures complete traceability, is free of carryover effects and significantly improves accuracy and reproducibility of the results.

Introduction

Trace analysis methods require contamination-free sample handling and accurate calibration graphs. In particular, the method calibration in the low ppb and ppt range is critical, as standard solutions in this range can hardly be handled without introducing errors. Moreover, if the sample concentration is outside the calibration range, laborious and error-prone dilution or preconcentration is required.

These drawbacks are overcome by inline coupling of the patented 800 Dosino and Metrohm`s intelligent ion chromatographs (ICs). The Dosino enables high-precision dosing of variable solution volumes into the sample loop or preconcentration column of the IC. Without compromising the accuracy of the results, a whole range of possibilities opens up for determinations over wide concentration ranges.

Furthermore, MiPCT-ME enables the user to work with low sample volumes and to remove interfering sample matrices. Combined with automated multi-point calibration, these techniques cover a measuring range of six orders of magnitude.

Combining intelligent software and hardware allows the system to compare results, take logical decisions, and carry them out: if, for example, the concentration of the analyte in the sample exceeds that of the calibration range, the system, after the first injection, compares peak areas, calculates the appropriate injection volume, and automatically re-injects the sample.

## Liquid Handling in the time of the Dosino

The Metrohm 800 Dosino is a precise and extremely flexible dosing device. In combination with the intelligent MagIC Net™ software it allows the IC system to compare results, take decisions, and carry them out.

➢ maximum flexibility: 4 freely definable dosing ports cope with complex setups

➢ fully controlled by MagIC Net™ software

➢ variable aspiration and dosing from 4...4000 µL

➢ 10000 volume increments

➢ virtually no carryover

Metrohm`s intelligent Preconcentration Technique with Matrix Elimination

Manual preparation of calibration standards generally starts from a multi-ion standard by appropriate manual dilution. This procedure is time-consuming and error-prone. Metrohm Inline Calibration allows to calibrate any standard concentration in the ng/L range by using one single standard solution at the µg/L level. A multipoint calibration emerges by varying the preconcentration volumes of the standard solution using the extremely precise dosing capabilities of the Dosino.

Detector

(2)

Dosinos

(1)

(4)

(3)

to waste
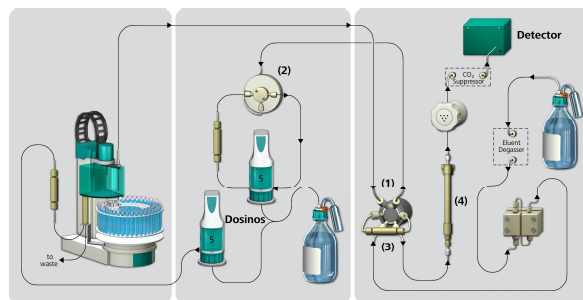
The sample is drawn through the injection valve (1) into the buffer volume (2). After switching the valve to the fill position, the exact volume is fed onto the preconcentration column (3). From there, the ions are eluted to the separation column (4).

Setup

Determinations over six orders of magnitude can be realized with any intelligent Metrohm IC coupled to the 800 Dosino and the 858 Professional Sample Processor; all components are controlled by MagIC Net™ software.


Application areas

While the analytes of interest are retained on the preconcentration column, interfering matrices are eliminated. The method is virtually free of carryover effects and thus ideally suited for trace and ultra-trace analysis in challenging samples from

➢ **power plants** (one IC copes with the analysis of all cooling circuit waters)

➢ **the semiconductor industry** (etching, extracting and rinsing solutions)

➢ **sewage treatment plants** (waters of high organic load)

➢ **food and beverage, pharmaceutical and petrochemical industry**

### Sample concentration is within calibration range (0.1…10 µg/L)

0.5 µg/L anion standard

Sample prep.:	MiPCT-ME
Volume_precon. :	4000 µL
Column:	Metrosep A Supp 5 - 150/4.0
Column temp.:	35 °C
Eluent:	3.2 mmol/L sodium carbonate
	1.0 mmol/L sodium hydrogen carbonate
Flow:	0.7 mL/min
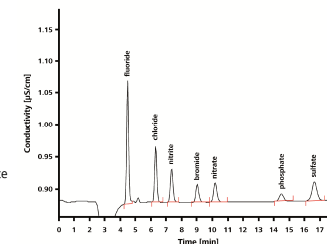
Sample concentration is outside calibration range (0.1…10 µg/L)

If, after the first injection, the concentration of the analyte to be determined exceeds that of the calibration range, only a fractional volume, previously calculated by MagiC Net™, is accurately transferred by the Dosino to the preconcentration column: 20 µL of a 1000 µg/L sample correspond to 20 ng.

Sample prep.:	MiPCT-ME
Volume_precon. :	**20 µL of a 1000 µg/L sample (= 20 ng)**
Column:	Metrosep A Supp 5 - 150/4.0
Column temp.:	35 °C
Eluent:	3.2 mmol/L sodium carbonate
	1.0 mmol/L sodium hydrogen carbonate
Flow:	0.7 mL/min
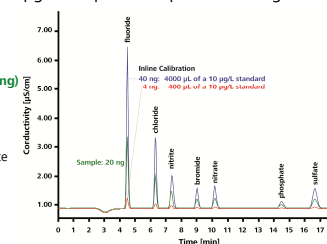
The different preconcentration volumes (4000…4 µL) cover three orders of magnitude (40…0.04 ng). When combined with multi-point calibration, which also copes with three orders of magnitude, the entire measuring range extends to six orders of magnitude.

	Preconcentration volume [µL]	Amount on column [ng]
10 µg/L standard	4000	40.00
	400	4.00
	40	0.40
	4	0.04
1000 µg/L sample	20	20.00

sample

Calibration range (three orders of magnitude)

Automatic calculation of preconcentration volume (three orders of magnitude)

[1]Metrohm USA, Riverview, FL 33578/USA, hari@metrohmusa.com, phone +1 813 316 4700
[2]Metrohm International Headquarters, Herisau/Switzerland, ht@metrohm.com