



Application Note AN-C-195

Cation quantification with increased performance using microbore IC

Benefits of microbore ion chromatography for cation analysis

Analytical performance with ion chromatography (IC) is typically determined by the signal-to-noise (S/N) ratio that the analytical equipment can reach. The S/N ratio strongly depends on chromatographic peak shapes. Peak shapes improve in miniaturized IC systems with less dead volume [1].

Microbore IC combines 2 mm separation columns, microbore capillaries, and a conductivity detector with reduced cell volume to create a miniaturized IC system with optimal sensitivity [2]. Such systems provide shorter retention times and consume less

eluent, increasing sample throughput and reducing the costs of daily routine analytics.

In this Application Note, a microbore IC system (MB) was compared to a standard bore IC system (SB). The microbore IC system showed improved resolution and better peak heights (a factor of ~30% more for lithium ions). Microbore IC uses less solvents and can result in cost reductions of up to 75% compared to using standard bore ion chromatography systems. Using MB systems has the potential to improve the performance of many typical IC applications.

SAMPLE AND SAMPLE PREPARATION

This study was conducted with alkali metal ions, alkaline earth metal ions, and ammonium. A mixed standard solution ($c(\text{Li}^+) = 25 \mu\text{g/L}$, $c(\text{Na}^+, \text{NH}_4^+) = 125 \mu\text{g/L}$, $c(\text{K}^+, \text{Mg}^{2+}, \text{Ca}^{2+}) = 250 \mu\text{g/L}$) was prepared

from 1000 mg/L stock solutions (Standards for IC, TraceCERT®, Sigma-Aldrich, Merck) by dilution in ultrapure water.

EXPERIMENTAL

A microbore IC system comprised of a 930 Compact IC Flex Oven/DEG/MB together with an IC conductivity detector MB (Figure 1) was compared to its respective standard bore IC system configuration (930 Compact IC Flex Oven/DEG).

The MB setup from Metrohm has a reduced dead volume with shorter capillaries and smaller capillary inner diameters (0.18 mm) wherever possible.

The microbore conductivity detector has a small inner cell volume (0.3 μL) and a low noise level (<0.1 nS). Furthermore, it even tolerates challenging eluents such as methanesulfonic acid (MSA). Microbore columns, which have a 2 mm inner diameter and associated reduced eluent flow rates, lead to better S/N. This increases sensitivity even further and lowers limits of detection.

The mixed cation standard solution was injected using a 5 μL loop and then separated on a 2 mm version of the Metrosep C 6 column on both tested IC systems.

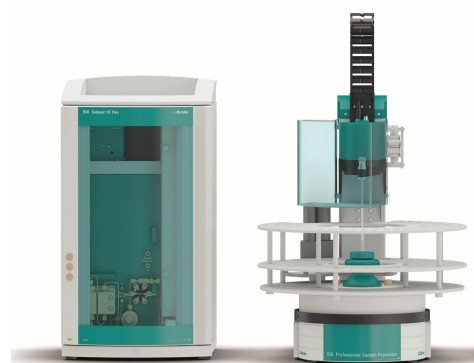


Figure 1. Instrumental setup including a miniaturized 930 Compact IC Flex Oven/Deg/MB and an 858 Professional Sample Processor.

The conductivity was directly recorded (non-suppressed cation analysis, Table 1).

Table 1. IC method parameters for both standard bore and microbore IC systems.

Column	Metrosep C 6 - 150/2.0
Eluent (from Merck concentrate Sigma-Aldrich, Merck 19399)	$c(\text{HNO}_3) = 1.7 \text{ mmol/L}$ $c(\text{DPA}) = 1.7 \text{ mmol/L}$
Flow rate	0.25 mL/min
Temperature	30 °C
Injection volume	5 μL
Detection	Direct conductivity

For performance comparison reasons, the retention times, resolution, peak heights, and repeatability were

evaluated with MagIC Net software (version 4.1).

RESULTS

Overall performance was improved when using the MB system for analysis. Retention times were shorter with the MB system (approximately 0.2 minutes in this case) than with the SB system (Figure 2).

Resolution with the MB system was ~115% better than with the SB system (Table 2). Peak heights were higher, with most improvement shown for the early-

eluting peaks (lithium, sodium, ammonium) on the MB system (Table 3). The noise was comparable for both tested IC setups.

Minimal improvement effects were observed for later eluting peaks (e.g., potassium, magnesium, and calcium). For all other relevant parameters, MB and SB showed similar results (e.g., repeatability).

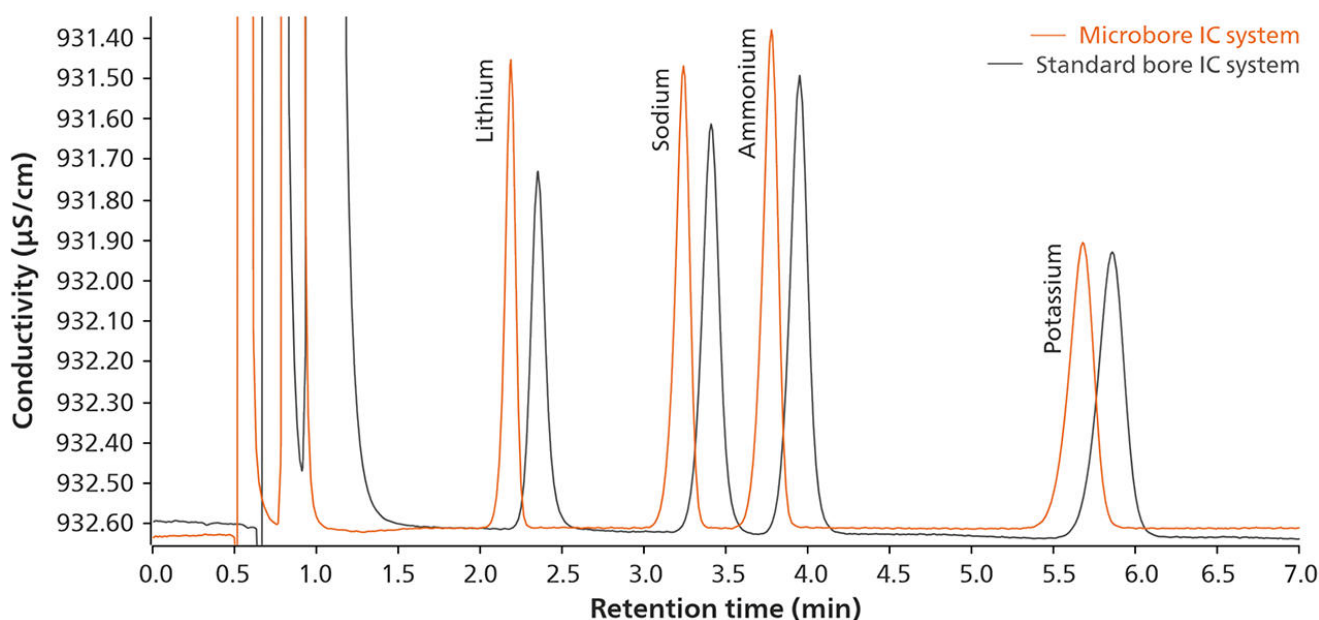


Figure 2. Comparison of the chromatograms for alkali metal ions (lithium, sodium, and potassium) and ammonium on a Metrosep C 6 microbore column with microbore IC (MB, orange chromatogram) and on a standard bore IC system (SB, grey chromatogram). The microbore IC system shows improved peak shapes, increased peak heights, and shorter retention times.

Table 2. Comparison of peak resolution for alkali metal ions and ammonium as measured by MB and SB systems.

Resolution	MB	SB
Lithium	5.6	5.6
Sodium	3.0	2.6
Ammonium	7.9	7.3
Potassium	6.0	5.8

Table 3. Comparison of peak heights and associated improvement factors for MB vs. SB systems.

Peak height [$\mu\text{S}/\text{cm}$]	MB	SB	Improvement factor
Lithium	1.16	0.88	131%
Sodium	1.14	1.01	113%
Ammonium	1.23	1.13	108%
Potassium	0.71	0.70	100%

CONCLUSION

The MB system combines microbore capillaries, a conductivity detector with reduced cell volume, and a 2 mm separation column—all of which lead to improved peak shapes and shorter retention times. This enables increased sensitivity and lower limits of detection. Lower flow rates reduce eluent consumption and overall running costs.

Non-suppressed MB systems in combination with 2 mm columns deliver significant improvements with respect to resolution and sensitivity. For sequentially suppressed IC systems (SES) including a microbore

CO_2 suppressor (MCS) with reduced dead volume, the main improvement is shorter retention times. This is helpful with low flow rates, and especially in combination with gradient applications as changes in the eluent composition will quickly impact the analysis and the effect will not be delayed by unnecessary dead volume.

MB systems can be used with 2 mm as well as 4 mm separation columns. These systems are suitable for all IC applications.

REFERENCES

1. Diederich, V.; Riess, A. K. Best Practice for Separation Columns in Ion Chromatography (IC) – Part 2. *Analyze This – The Metrohm Blog*, 2021.
2. Metrohm AG. Metrohm Microbore Ion Chromatography – Maximize the Efficiency of Your Ion Chromatography!, 2023.

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CONFIGURATION



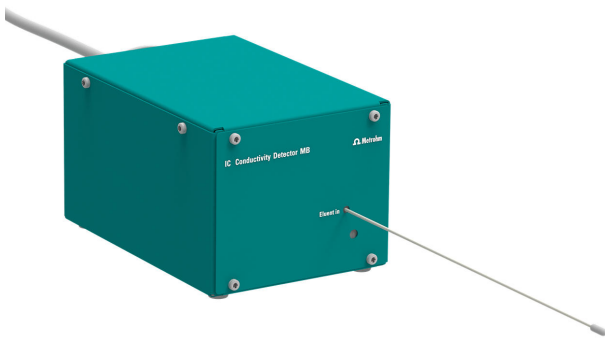
930 Compact IC Flex Oven/DEG/MB

Il 930 Compact IC Flex Oven/DEG/MB è lo strumento IC Compact intelligente con **forno per colonna, senza soppressione** e con **Degasser** integrato. Lo strumento può essere impiegato con qualsiasi metodo di separazione e di rilevazione.

Campi d'impiego tipici:

- Determinazione di anioni e cationi senza soppressione con rilevazione in conducibilità
- Applicazioni semplici con rilevazione UV/VIS o amperometrica
- Ottimizzato per le applicazioni Microbore (2 mm), ideale per le tecniche di accoppiamento (IC-MS o IC-ICP/MS)

Supportato da MagIC Net 4.1 e versioni successive



IC Conductivity Detector MB

Rilevatore della conducibilità ad alte prestazioni compatto e intelligente per gli strumenti IC intelligenti. Ottimizzato per le colonne Microbore. Eccellente stabilità di temperatura, la completa elaborazione del segnale all'interno del blocco del rivelatore protetto e DSP – Digital Signal Processing (elaborazione del segnale digitale) – di ultima generazione garantiscono la massima precisione della misura. Grazie al range dinamico di lavoro non sono necessari cambiamenti (anche non automatici) del range di misura.

Campi d'impiego tipici:

- Determinazione di anioni o cationi con soppressione chimica, soppressione sequenziale o senza soppressione e rilevazione in conducibilità
- Ottimizzato per le applicazioni Microbore (2 mm), ideale per le tecniche di accoppiamento (IC-MS o IC-ICP/MS)

Panoramica delle specifiche:

- Da 0 a 15.000 $\mu\text{S}/\text{cm}$ senza commutazione dell'intervallo
- Volume della cella: 0,3 μL
- Elettrodi ad anello in acciaio legato X2CrNiMo17-12-2 (316 L), compatibili con MSA
- Pressione d'esercizio massima: 10,0 MPa (100 bar)
- Temperatura della cella: da 20 a 50 °C in incrementi di 5 °C
- Stabilità della temperatura: < 0,001 °C
- Rumore di fondo: < 0,2 nS/cm tipico per la soppressione sequenziale
- Capillari: DI 0,18 mm

Supportato da MagIC Net 4.1 e versioni successive



Metrosep C 6 - 150/2,0

L'alta capacità del materiale C 6 rende la versione Microbore della colonna di separazione Metrosep C 6 - 150/4,0 la soluzione ottimale per la separazione di cationi standard con elevate differenze di concentrazione in tempi di reazione ragionevoli. Con questa colonna è possibile determinare le acque potabili a basso contenuto di ammonio.

La colonna è adatta all'impiego nell'accoppiamento IC-MS.



Metrosep C 6 Guard/2,0

La Metrosep C 6 Guard/2,0 contiene il materiale della colonna C 6 e serve come protezione da particelle e impurità. In tal modo viene prolungata notevolmente la durata della colonna di separazione analitica. La Metrosep C 6 Guard/2,0 funziona secondo il «On Column Guard System» e viene montata sulla rispettiva colonna di separazione pressoché senza volume morto.



Equipaggiamento IC: MiPT

Kit di accessori per il montaggio di un Dosino per l'iniezione a loop parziale.