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 ($c(Li^+)$ = 25 µg/L, $c(Na^+, NH_4^+)$ = 125 µg/L, $c(K^+, Mg^{2+}, Ca^{2+})$ = 250 µg/L) was prepared from 1000 mg/L stock solutions (Standards for IC, *Trace*CERT®, 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.

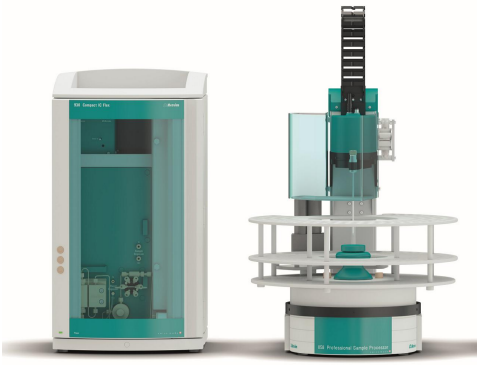

Figure 1. nstrumental setup including a miniaturized 930 Compact IC Flex Oven/Deg/MB and an 858 Professional Sample Processor.

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. 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)	$c(HNO_3)$ = 1.7 mmol/L c(DPA) = 1.7 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).

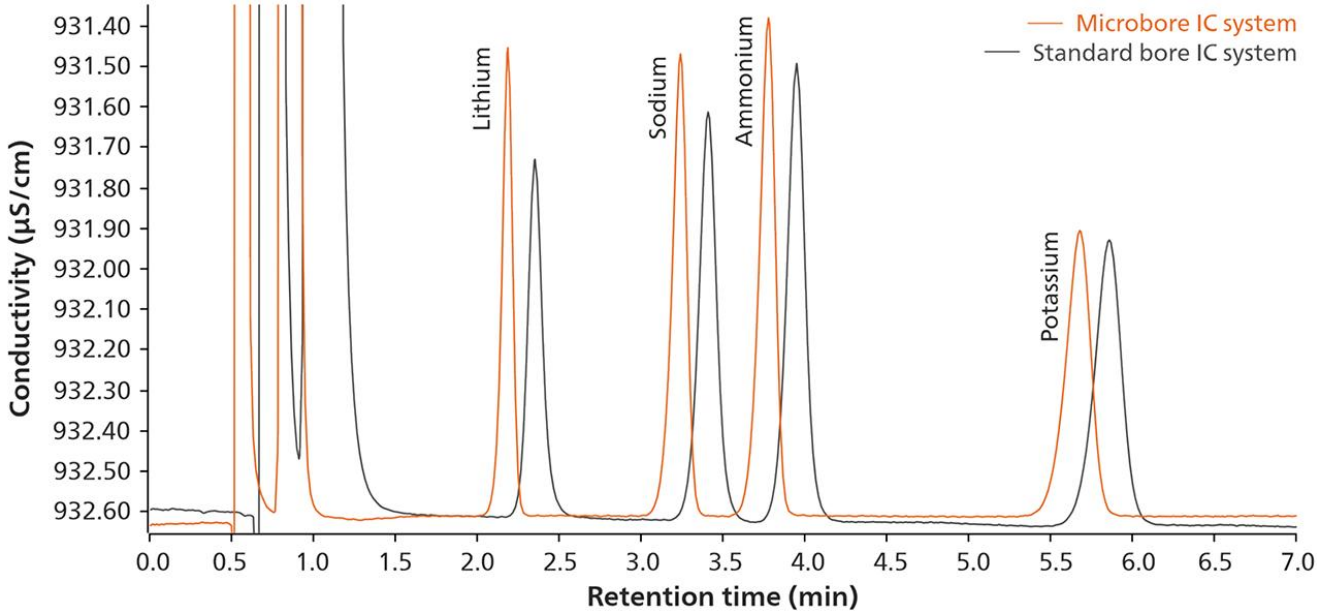

Figure 2. Comparison of the chromatograms for alkali metal ions (lithium, sodium, and potassium) and ammonium on a Metrosep C 6 microbore column with microbore IC (MB, orange chromatogram) and on a standard bore IC system (SB, grey chromatogram). The microbore IC system shows improved peak shapes, increased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

The 930 Compact IC Flex Oven/Deg/MB is the intelligent Compact IC instrument with **column oven, without suppression** and with built-in **degasser**. The instrument can be used with any separation and detection methods.

Typical areas of application:

- Anion and cation determinations without suppression with conductivity detection
- Simple applications with UV/VIS or amperometric detection
- Optimized for microbore (2 mm) applications, ideally suitable for coupling techniques (IC-MS or IC-ICP/MS)

Supported with MagIC Net 4.1 and higher

IC Conductivity Detector MB

Compact and intelligent high performance conductivity detector for intelligent IC instruments. Optimized for microbore columns. Outstanding temperature stability, the complete signal processing within the protected detector block and the latest generation of DSP – Digital Signal Processing – guarantee the highest precision of the measurement. No change of measuring ranges (not even automatic ones) is required, due to the dynamic working range.

Typical areas of application:

- Anion or cation determinations with chemical suppression, sequential suppression or without suppression and conductivity detection
- Optimized for microbore (2 mm) applications, ideally suitable for coupling techniques (IC-MS or IC-ICP/MS)

Specification at a glance:

- 0–15000 µS/cm without range switching
- Cell volume: 0.3 µL
- Ring-shaped electrodes made of stainless steel X2CrNiMo17-12-2 (316 L), compatible with MSA
- Maximum operating pressure: 10.0 MPa (100 bar)
- Cell temperature: 20–50 °C in increments of 5 °C
- Temperature stability: < 0.001 °C
- Baseline noise: < 0.2 nS/cm typical for sequential suppression
- Capillaries: ID 0.18 mm

Supported with MagIC Net 4.1 and higher

Metrosep C 6 - 150/2.0

The high-capacity C 6 material makes the microbore version of the Metrosep C 6 - 150/4.0 column the optimum solution for separating standard cations with high differences in concentration with reasonable retention times. Drinking water with low ammonium contents can be determined with this column.

The column is suitable for use in IC-MS coupling.

Metrosep C 6 Guard/2.0

The Metrosep C 6 Guard/2.0 contains the C 6 column material and is used to protect against particles and contamination. This considerably lengthens the service life of the analytical separation column. The Metrosep C 6 Guard/2.0 works according to the "On Column Guard System" and is screwed directly onto the respective separation column with virtually no dead volume.

IC equipment: MiPT

Accessory set for assembling a Dosino for Partial-Loop-Injection.