Application Note AN-C-195

使用微孔离子色谱法进行阳离子定量分析,性能提升

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.

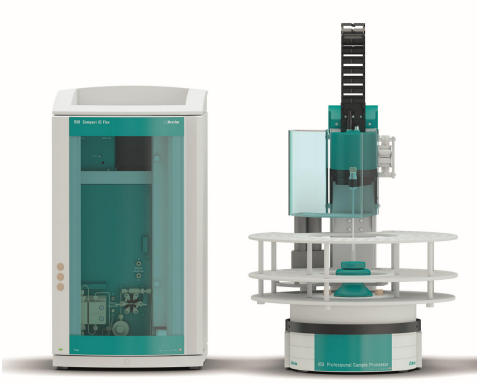

Figure 1. nstrumental setup including a miniaturized 930 Compact IC Flex Oven/Deg/MB and an 858 Professional Sample Processor.

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).

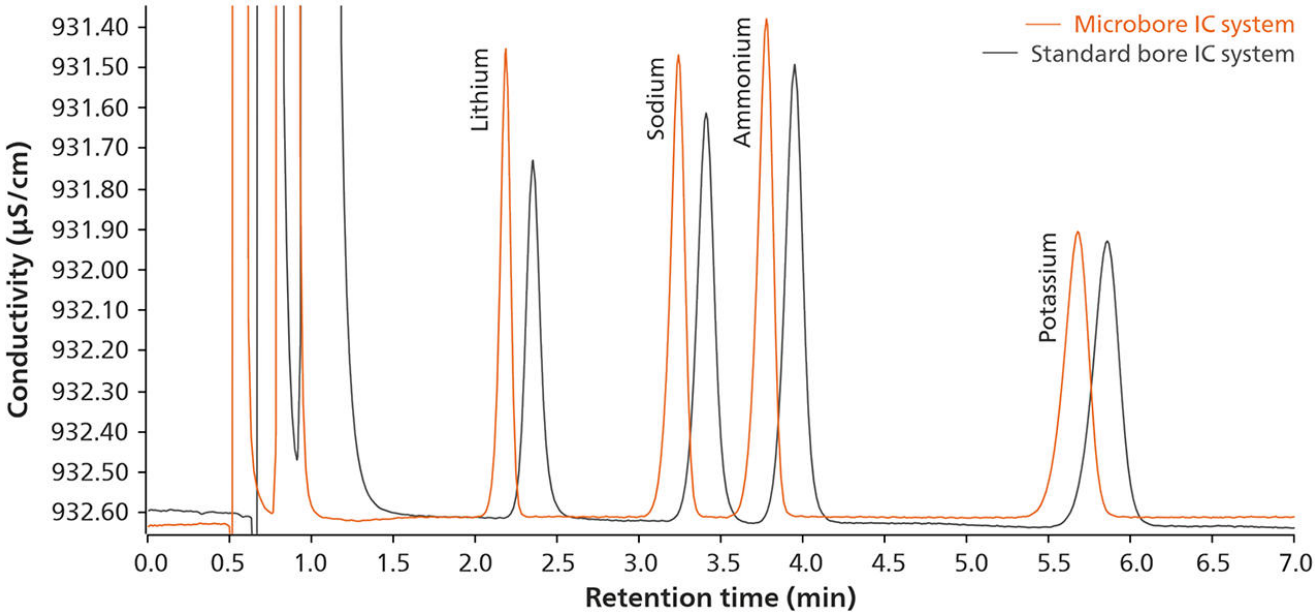

Figure 2. Comparison of the chromatograms for alkali metal ions (lithium, sodium, and potassium) and ammonium on a Metrosep C 6 microbore column with microbore IC (MB, orange chromatogram) and on a standard bore IC system (SB, grey chromatogram). The microbore IC system shows improved peak shapes, increased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

930 Compact IC Flex Oven/DEG/MB 是智能型带**柱加热炉、无抑制**的 Compact 离子色谱仪,并且内置**脱气装置**。该仪器可使用任意分离和检测方法。

典型的应用范围:

- 阴离子和阳离子测定,无抑制的电导检测
- 使用 UV-VIS 或安培检测的简单应用
- 针对微孔 (2mm) 应用经过优化,尤其适合耦合技术(IC-MS 或 IC-ICP/MS)

支持 MagIC Net 4.1 和以上版本

IC Conductivity Detector MB

用于智能型离子色谱仪的紧凑智能型高性能电导检测器。针对微孔柱经过优化。卓越的温度稳定性,在受保护的检测器端子板内完成整个信号处理过程以及最新一代的 DSP(数字式信号处理)均能确保测量的最高精确性。归功于动态工作范围,无需进行(也包括非自动)范围变更。

典型的应用范围:

- 阴离子或阳离子测定,化学抑制、序列抑制法或无抑制及电导检测
- 针对微孔 (2mm) 应用经过优化,尤其适合耦合技术(IC-MS 或 IC-ICP/MS)

规格说明书概览:

- 0 ... 15000 µS/cm 无区段切换
- 测量池容量:0.3 µL
- 由不锈钢 X2CrNiMo17-12-2 (316 L) 制成的环形电极,与 MSA 兼容
- 最大工作压力:10.0 MPa (100 bar)
- 池温:20 ... 50 ° C,步幅为 5 ° C
- 温度稳定性: 0.001 ° C
- 基线噪音:° 0.2° nS/cm,是使用序列抑制法的典型值
- 毛细管:ID 0.18 mm

支持 MagIC Net 4.1 和以上版本

Metrosep C 6 - 150/2.0

高容量的 C-6 材料使微孔径版本的 Metrosep C 6 - 150/4.0 柱成为在浓度差异很高时的正常保留时间内分离标准阳离子的解决方法。可用此柱测定铵含量很高的饮用水。

该柱适合用在 IC-MS 联用中。

Metrosep C 6 Guard/2.0

Metrosep C 6 Guard/2.0 包含 C-6 柱材料,用于防微粒和防污染。用它可显著延长分析用分离柱的使用寿命。Metrosep C 6 Guard/2.0 按照 «On Column Guard System» 来运行,可几乎无死点容积地直接安装到相应分离柱上。

MiPT

用于安装 Dosino 以进行局部循环进样的附件组。