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.

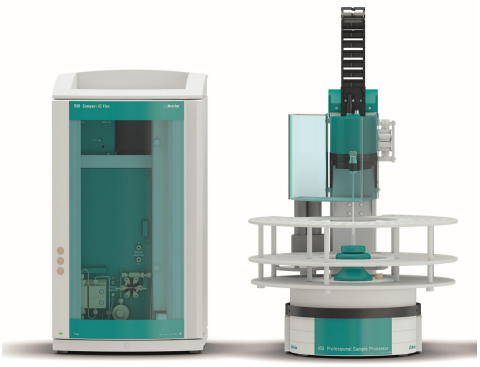

Figure 1. nstrumental setup including a miniaturized 930 Compact IC Flex Oven/Deg/MB and an 858 Professional Sample Processor.

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).

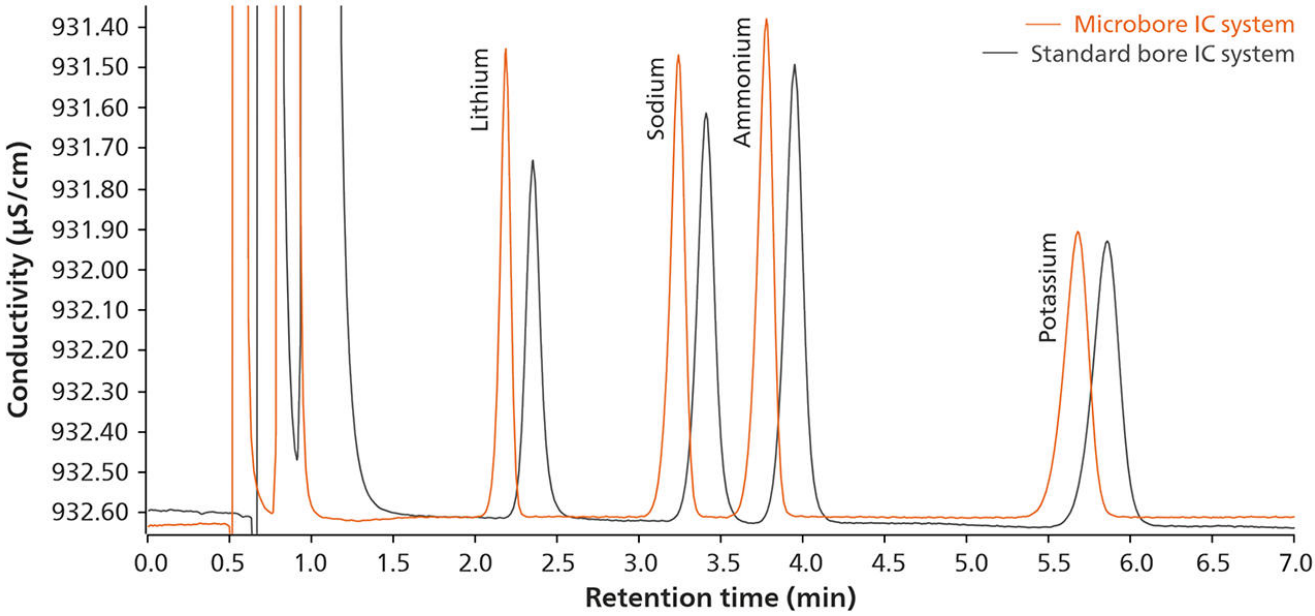

Figure 2. Comparison of the chromatograms for alkali metal ions (lithium, sodium, and potassium) and ammonium on a Metrosep C 6 microbore column with microbore IC (MB, orange chromatogram) and on a standard bore IC system (SB, grey chromatogram). The microbore IC system shows improved peak shapes, increased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

CONTACT

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CONFIGURATION

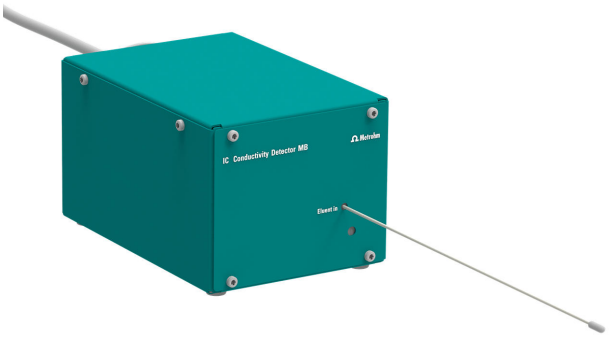
930 Compact IC Flex Oven/DEG/MB

930 Compact IC Flex Oven/DEG/MB は**カラムオーフン**と内蔵式**脱気装置を備えたサフレッションの無い**インテリシェントコンハクトIC 装置てす。この装置は任意の分離メソットおよひ検出メソットによって使用することかてきます。

典型的な使用領域:

- 電気伝導度検出器による、サフレッション無しの陰イオンおよひ陽イオンの測定
- UV/VIS 検出器またはアンヘロメトリック検出器によるシンフルな使用
- マイクロホア (2mm) アフリケーション向けに最適化、カッフリンク技術 (IC-MS または IC-ICP/MS)に最適

MagIC Net 4.1以上に対応



IC Conductivity Detector MB

インテリシェントIC装置のためのコンハクトかつインテリシェントな高出力電気伝導度検出器。マイクロホアカラム向けに最適化。優れた温度安定性、保護された検出器フロック内の総合的な信号処理、最新版の DSP (Digital Signal Processing) か高精度の測定を保証します。稼動範囲かタイナミックなのて測定範囲の変更は(自動のものも含めて)不必要てす。

典型的な使用領域:

- 電気伝導度検出器による、化学的サフレッションまたは連続的な化学的サフレッションのある、もしくはサフレッション無しの陰イオンおよび陽イオンの測定
- マイクロホア (2mm) アフリケーション向けに最適化、カッフリンク技術 (IC-MS または IC-ICP/MS)に最適

仕様概要:

- 0~15000 µS/cm、エリアの切り替えなし
- セル容量: 0.3 µL
- リンク状のステンレス製電極 X2CrNiMo17-12-2 (316 L)、MSA と互換
- 最大運転圧力: 10.0 MPa (100 ハー)
- セル温度: 20~50° C、5° C刻み
- 温度安定性: < 0.001° C
- ヘースラインノイス: < 0.2 nS/cm、連続サフレッションの平均値
- キャヒラリー: ID 0.18 mm

MagIC Net 4.1以上に対応

Metrosep C 6 - 150/2.0

容量の大きい C 6 の材料により、Metrosep C 6 - 150/4.0 カラムのマイクロホアハーションは、妥当な保持時間ての濃度差の大きい標準陽イオンの分離に最適なソリューションてす。アンモニウム含有量の少ない飲料水は、このカラムて測定することかてきます。

カラムは、IC-MS カッフリンクての使用に適しています。

Metrosep C 6 Guard/2.0

Metrosep C 6 Guard/2.0 には C 6 カラムの材料か含まれており、粒子や汚れから保護します。これにより、分析の分離カラムの耐用期間か格段に長くなります。Metrosep C 6 Guard/2.0 は «On Column Guard System» に則して機能し、それぞれの分離カラムにほほテットホリュームなして直接取り付けることかてきます。

IC: MiPT

ハーシャルルーフインシェクションのトシーノ設置のための付属品セット。