

Application Note AN-D-002

Anions and cations in beer

Streamlining beverage analysis with ion chromatography

Beverage analysis methods must be fast and robust to fulfill many quality control regulations. Optimal product quality is critical for consistent taste and other sensory characteristics in mass produced beverages. In the case of beer, the ionic composition heavily influences the taste. For example, potassium chloride salts lead to a bitter, astringent, and soapy taste while magnesium sulfates form more sweet-sour flavors. Therefore, analytical control of anions and cations in beer is essential to guarantee the quality and meet consumer needs.

Major anions in beer are precisely determined with ion chromatography (IC) and suppressed conductivity detection, while cations are quantified with non suppressed conductivity. With a two-channel system, cations and anions can be determined simultaneously for the same sample. To reduce manual preparation steps, beer samples are automatically filtered with Inline Ultrafiltration. Features like automatic calibration and logical sample dilution streamline such beverage analyses and ensure the fast analysis of samples in high-throughput laboratories.



EXPERIMENTAL

Beer samples from different providers (e.g., WarsteinerTM) were automatically diluted and filtered through a 0.22 μ m membrane in the Ultrafiltration cell (**Figure 1**). Analyte concentrations outside of the calibration range are diluted with an optimal dilution factor and analyzed again with logical dilution, a feature of the chromatographic software MagIC Net. Hence, the results always fit within the calibration range.

After Inline Sample Preparation is performed, the sample is injected into two analytical channels that simultaneously analyze cations and anions under isocratic elution conditions (Table 1). Conductivity is a

universal and sensitive detector to determine all relevant ions present in the beer sample. The MagIC Net software offers time-saving reliable calibration from a single standard solution by injecting increasing volumes on to the separation column (MiPT – Metrohm intelligent Partial Loop Injection Technique). This avoids pipetting errors during standard preparation. Furthermore, samples can be injected with the most suitable injection volume. Together with logical dilutions, sample concentrations in the range 1:10,000 can be analyzed reliably. High accuracy of results is achieved by an optimal fit for the calibration points (feature: high-low calibration).

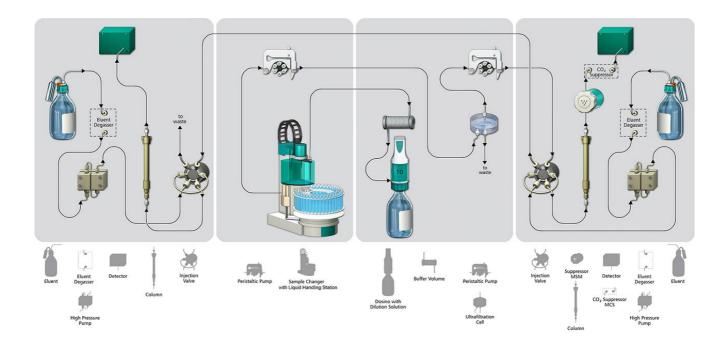


Figure 1. One autosampler (including filtration and dilution equipment) prepares the sample for two analysis channels such that anions and cations are determined in parallel from the same sample. The IC for cation analysis is displayed on the left side, and the IC used for anion analysis is shown on the right side.

Table 1. Measuring conditions for the determination of anions and cations in beer with ion chromatography.

| Method parameter | Anions | Cations |
|---------------------|---|--|
| Column | Metrosep A Supp 10 - 100/4.0 | Metrosep C 6 - 150/4.0 |
| Eluent | 4 mmol Na $_2$ CO $_3$ + 6.0 mmol/L NaHCO $_3$ + 5.0 μ mol/L NaClO $_4$ | 2.3 mmol HNO ₃ + 1.7 mmol/L dipicolinic acid |
| Flow rate | 0.7 mL/min | 0.9 mL/min |
| Temperature | 30 °C | 35 °C |
| Injection | 20 μL | 20 μL |
| Detection | Suppressed conductivity | Non-suppressed conductivity |

RESULTS

Potassium was identified as a major cation in all beer samples, while the concentration of other cations (e.g., Na⁺, Ca²⁺, and Mg²⁺) was lower than 100 mg/L (**Figure 2**). The results reveal the effect of K⁺ in beer, as it provides a bitter and astringent taste. Other ions such as ammonium (eluting between Na⁺ and K⁺) can

also be determined.

Chloride, phosphate, nitrate, and sulfate were the main anions detected in beer (Figure 2). Sulfite, a common preservative, can be determined next to other anions in the same run (retention time approximately 11 minutes).

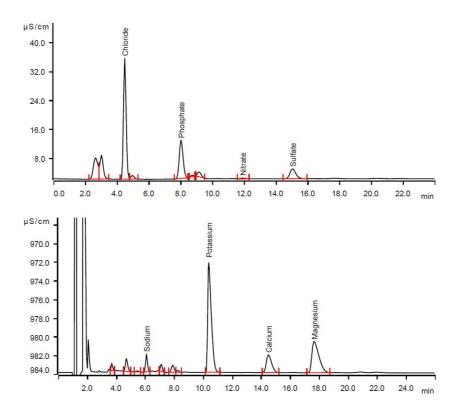


Figure 2. Analysis of a Warsteiner Pils sample (10-fold dilution) containing chloride (229 mg/L), phosphate (352 mg/L), nitrate (5 mg/L), and sulfate (60 mg/L) as major anions (top), and sodium (13 mg/L), potassium (365 mg/L), calcium (53 mg/L), and magnesium (56 mg/L) as major cations (bottom).

CONCLUSION

Ion chromatography is a robust and straightforward analytical technique to monitor beer production and to control its quality. Beverage samples are automatically diluted and filtered prior to analysis to

protect the analytical system. All essential anions and cations are simultaneously quantified in one analysis run. Features like logical dilution further save time and reduce manual work.

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CONFIGURATION







930 Compact IC Flex Oven/SeS/PP/Deg

The 930 Compact IC Flex Oven/SeS/PP/Deg is the intelligent Compact IC instrument with column oven, sequential suppression, peristaltic pump for suppressor regeneration and built-in degasser. The instrument can be used with any separation and detection methods.

Typical areas of application:

- Anion or cation determinations with sequential suppression and conductivity detection

IC Conductivity Detector

Compact and intelligent high performance conductivity detector for intelligent IC instruments. 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.

Metrosep A Supp 10 - 100/4.0

The Metrosep A Supp 10 - 100/4.0 separation column is based on a high-capacity polystyrene-divinylbenzene copolymer with a particle size of only 4.6 μ m. This column is characterized by high plate numbers and high selectivity. Sulfite and sulfate thus can be reliably separated in the eluent without the addition of organic modifiers. These characteristics are supplemented by great flexibility with respect to column temperature, flow and the composition of the eluent.

Its robust construction, excellent price-performance ratio and very good separating efficiencies, in conjunction with moderate chromatography times, make the Metrosep A Supp 10 - 100/4.0 a universally applicable anion separation column.





Metrosep C 6 - 150/4.0

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



858 Professional Sample Processor

The 858 Professional Sample Processor processes samples from $500 \mu L$ to 500 mL. The sample transfer takes place either by means of a peristaltic pump on the 850 Professional IC system or with an 800 Dosino.



941 Eluent Production Module

The 941 Eluent Production Module enables the automatic production of an eluent. It enables continuous working without manual intervention and guarantees stable retention times. It can be combined with all Metrohm IC instruments and is monitored by MagIC Net.



800 Dosino

Drive with write/read hardware for intelligent Dosing Units. With fixed cable (length 150 cm).





MSM Rotor A

Suppressor rotor for all IC instruments with MSM (Metrohm Suppressor Module)



IC equipment: Inline dilution

Accessory set for Inline Dilution. For use with the 858 Professional Sample Processor, 800 Dosino and 741 Magnetic Stirrer.





MagIC Net 4.0 Professional: 1 license

Professional PC program for controlling all intelligent Professional IC systems, Compact IC systems and their peripherals, all detectors and various Auto samplers, 800 Dosino, 771 Compact Interface, etc. The software permits checks, data acquisition, data evaluation and data monitoring as well as report generation of ion chromatographic analyses.

Graphical user interface for routine operations, extensive database programs, method development, configuration and manual system control; very flexible user management, powerful database operations, extensive data export functions, individually configurable report generator, control and monitoring of all system components and the chromatography results.

MagIC Net Professional complies fully with FDA Regulation 21 CFR Part 11 as well as GLP.

MagIC Net is available in 16 dialog languages: German, English, Chinese, Traditional Chinese, French, Italian, Spanish, Portuguese, Bulgarian, Czech, Hungarian, Japanese, Korean, Russian, Slovakian, Polish

1 license

The installation and documents are supplied on a USB flash drive.

