Fully automated sample preparation for liquid chromatographic content determinations

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Ion Chromatography

Summary

Inline coupling of the 815 Robotic Soliprep with an ion chromatograph (IC) allows the straightforward determination of anions and cations in tablets. After automatic solvent addition and subsequent comminution, the homogenized tablet samples (Singulair and Bezafibrat) are filtered and subsequently transferred to the injector. The completely automated sample preparation saves both time and money, guarantees traceability of each sample preparation step and yields correct and precise results. In the range of 0.2…50 mg/L, six-point calibration curves for anions and cations yield correlation coefficients better than 0.99990 and 0.99991, respectively. While relative standard deviations (RSDs) for sub-ppm levels of nitrate, sulfate, calcium and magnesium in Singulair and Bezafibrat are smaller than 3.64%, RSD of ppm levels of chloride is better than 0.83%.

The application of further inline sample preparation steps such as pulverizing, extracting, filtering or diluting facilitates numerous custom-tailored setups for ion determinations in exacting matrices such as animal feed, sediments or food.

Introduction

Only liquid samples can be fed directly into a liquid chromatograph (LC). However, solid samples such as foodstuffs and tablets can be made injectable by homogenization, filtration and dilution with the 815 Robotic Soliprep.

Full automation saves time and eliminates error-prone manual sample preparation steps. Automation not only improves reproducibility and accuracy, but also increases throughput as well as lab safety.

This poster gives a description of the instrumental setup and demonstrates the usefulness of automated sample preparation by means of determinations of ionic ingredients in the anti-allergy drug Singulair and in the hypolipidemic Bezafibrat.

Sample preparation

In order to perform these sample preparation steps fully automatically, the sample processor is equipped with the Polytron 1300 D® dispersing device and a Luer tip that is connected to an 800 Dosino. First, the solvent is added to the sample via the tip. After thorough comminution, the homogenized sample is filtered through a disposable filter. The filtrate can either be directly injected into the LC instrument or transferred to an empty vial for subsequent dilution. Based on tablet weight and added solvent volume, MagIC Net™ automatically calculates the ion contents. Since comminution and any dilution steps are performed during the recording of the previous sample's chromatogram, these sample preparation steps do not prolong the analysis.

Instrumentation

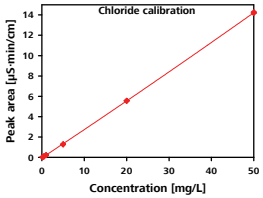
- 850 Professional IC AnCat – MCS
- 815 Robotic Soliprep for Liquid Chromatography
- Polytron PT 1300 D

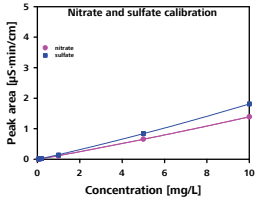
Anions

Column:	Metrosep A Supp 5 – 100	**Column temp.:**	30 °C
Eluent:	3.2 mmol/L sodium carbonate 1.0 mmol/L sodium hydrogen carbonate	**Liquid medium:**	Ultrapure water
		Loop:	20 µL
Flow:	0.7 mL/min		

a) Calibration

	Chloride	Nitrate	Sulfate
Regression	quadratic	quadratic	quadratic
Correlation coefficient	0.99999	0.99990	0.99999
RSD [%]	1.059	2.889	2.021

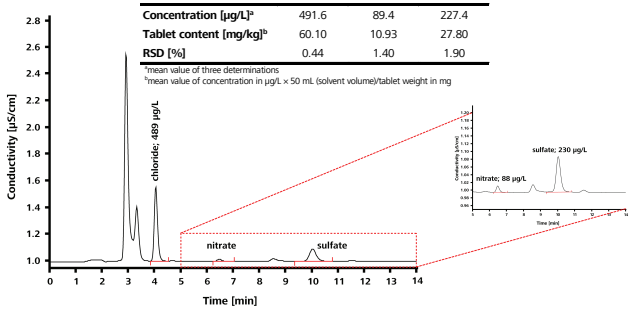




b) Singulair

	Chloride	Nitrate	Sulfate
Concentration [µg/L][a]	491.6	89.4	227.4
Tablet content [mg/kg][b]	60.10	10.93	27.80
RSD [%]	0.44	1.40	1.90

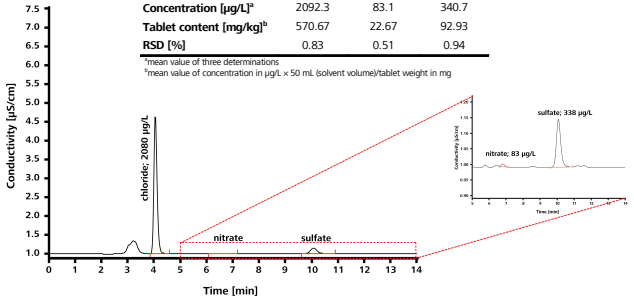
[a]mean value of three determinations
[b]mean value of concentration in µg/L × 50 mL (solvent volume)/tablet weight in mg



c) Bezafibrat

	Chloride	Nitrate	Sulfate
Concentration [µg/L][a]	2092.3	83.1	340.7
Tablet content [mg/kg][b]	570.67	22.67	92.93
RSD [%]	0.83	0.51	0.94

[a]mean value of three determinations
[b]mean value of concentration in µg/L × 50 mL (solvent volume)/tablet weight in mg

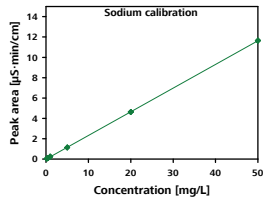


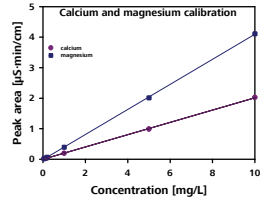
Cations

Column:	Metrosep C 4 – 100	**Column temp.:**	30 °C
Eluent:	1.7 mmol/L nitric acid 0.7 mmol/L dipicolinic acid	**Liquid medium:**	Ultrapure water
		Loop:	20 µL
Flow:	1.0 mL/min		

a) Calibration

	Sodium	Calcium	Magnesium
Regression	linear	linear	linear
Correlation coefficient	0.99999	0.99991	0.99995
RSD [%]	0.529	2.228	1.639

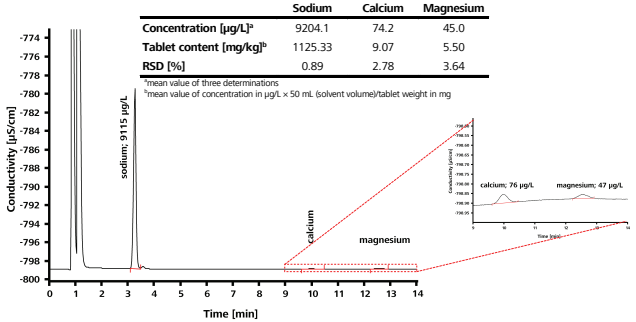




b) Singulair

	Sodium	Calcium	Magnesium
Concentration [µg/L][a]	9204.1	74.2	45.0
Tablet content [mg/kg][b]	1125.33	9.07	5.50
RSD [%]	0.89	2.78	3.64

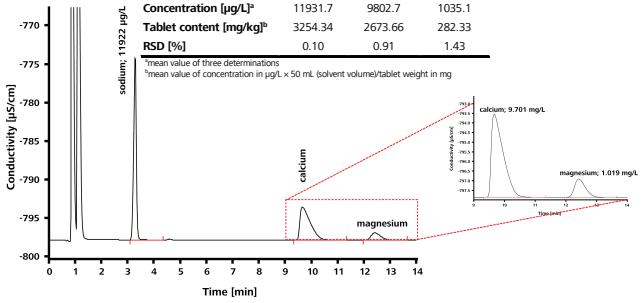
[a]mean value of three determinations
[b]mean value of concentration in µg/L × 50 mL (solvent volume)/tablet weight in mg



c) Bezafibrat

	Sodium	Calcium	Magnesium
Concentration [µg/L][a]	11931.7	9802.7	1035.1
Tablet content [mg/kg][b]	3254.34	2673.66	282.33
RSD [%]	0.10	0.91	1.43

[a]mean value of three determinations
[b]mean value of concentration in µg/L × 50 mL (solvent volume)/tablet weight in mg



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