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Application Note 519

Parallel Sample Clean-Up and Analysis in Capillary LC

INTRODUCTION

Column switching techniques, i.e., using preconcentration columns, allow for automated sample clean-up and fast sample loading. However, additional loading or cleanup time is often required to desalt the samples or to load extra-large sample volumes. This can ultimately increase the total analysis time. In this application note we demonstrate a two fold increase of the sample throughput by performing sample clean-up and analysis simultaneously (parallel).

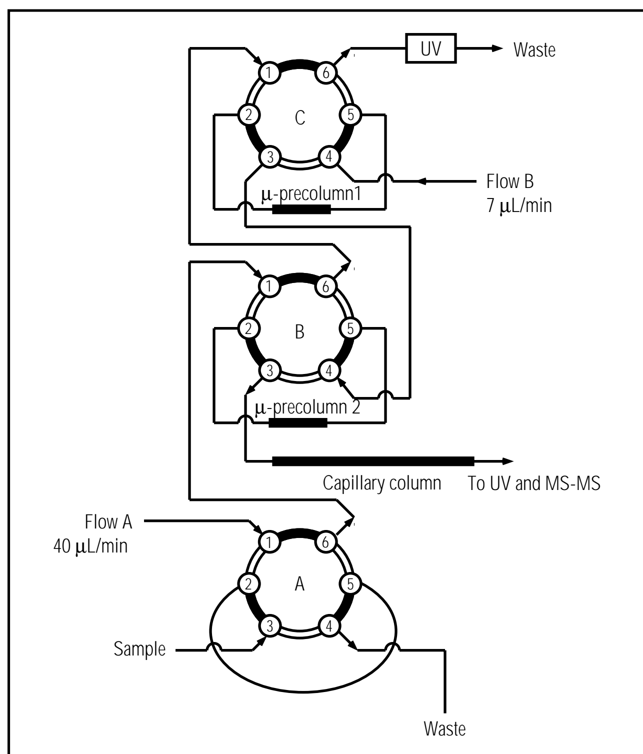

Figure 1. System set-up for high sample throughput Capillary LC/ ESI-MS-MS.

RESULTS AND DISCUSSION

The two additional column switching valves of the FAMOS™ Micro Autosampler can be used to improve sample throughput by a factor of two (provided that the clean-up time is equal or shorter than the analysis time). μ-Precolumns are connected to the column switching valves, as shown in the system setup. An example of a typical time event table is given in Table 1. In this case the chromatographic separation took 5 min and the total analysis time was equal to 7 min.

Table 1. Time Event Table

Time	Event	A	B	C
0'	Load μ-Precolumn 1; meanwhile desorption of μ-Precolumn 2 and analysis on capillary column	1-2	1-2	1-6
7'	Load μ-Precolumn 2; meanwhile desorption of μ-Precolumn 1 and analysis on capillary column	1-2	1-6	1-2
14'	Reinitialize	1-2	1-2	1-6

This kind of setup was demonstrated for the analysis of deuterated purine adducts of which the undeuterated counterparts are common plant hormones. The purine adducts were loaded with an aqueous solution onto a 300 μm I.D. x 5 mm μ-Precolumn at 40 μL/min. Desalting of the samples took 7 min after which the precolumn was switched in-line with the 300 μm I.D. x 15 cm reversed phase Capillary LC column. During sample analysis a second μ-Precolumn

was loaded with the next sample. The mass chromatograms show the separation of deuterated 9-glycoside isopentenyladenin (m/z 372), 9-ribose isopentenyladenine (m/z 342), isopentenyladenine (m/z 210), 9-glycoside zeatine (m/z 387), 9-riboside zeatine (m/z 357) and zeatine (m/z 225). The flow rate through the analytical column was maintained at 7 µL/min. Compound detection was conducted with UV absorption and MS-MS.

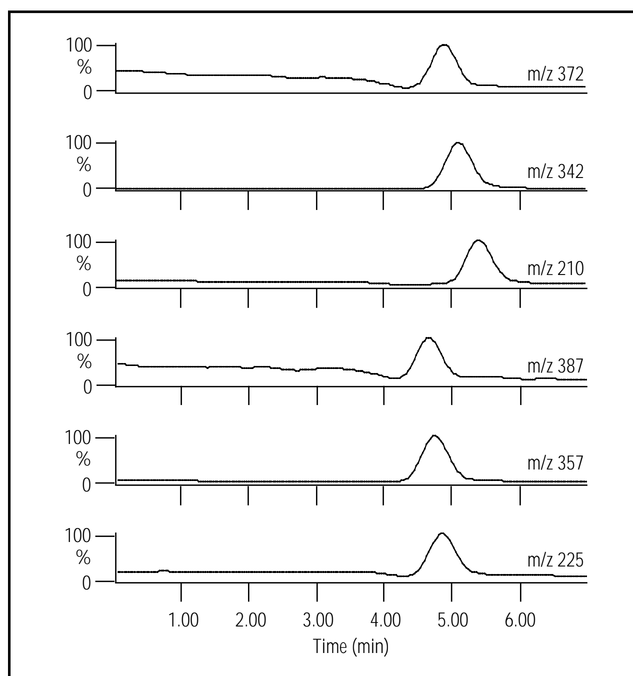

Figure 2. Chromatographic separation of purine adducts.[1]

REFERENCES

1. Courtesy E. Witters and H. Van Onckelen, Lab. Plantbiochemistry and Physiology, University of Antwerp and W. Van Dongen and E. Esmans, Nucleososide Research and Mass Spectrometry Unit, University of Antwerp.

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