

# Automated On-Line Salt Removal in Micro HPLC ESI-MS

## INTRODUCTION

Compound mixtures isolated from biological matrices often contain considerable amounts of non-volatile buffer salts. The presence of such buffers may interfere with the operation of electrospray ion sources by clogging the skimmer and obscuring or suppressing ionization.

## RESULTS AND DISCUSSION

Figure 1 gives the valve set-up. The second low-dispersion 6-port valve of a FAMOS micro autosampler was used to direct the flow postcolumn to the source of the mass spectrometer or to waste. The valve switching time was determined by measuring the current in the ESI source. Figure 2A shows the injection of a sample

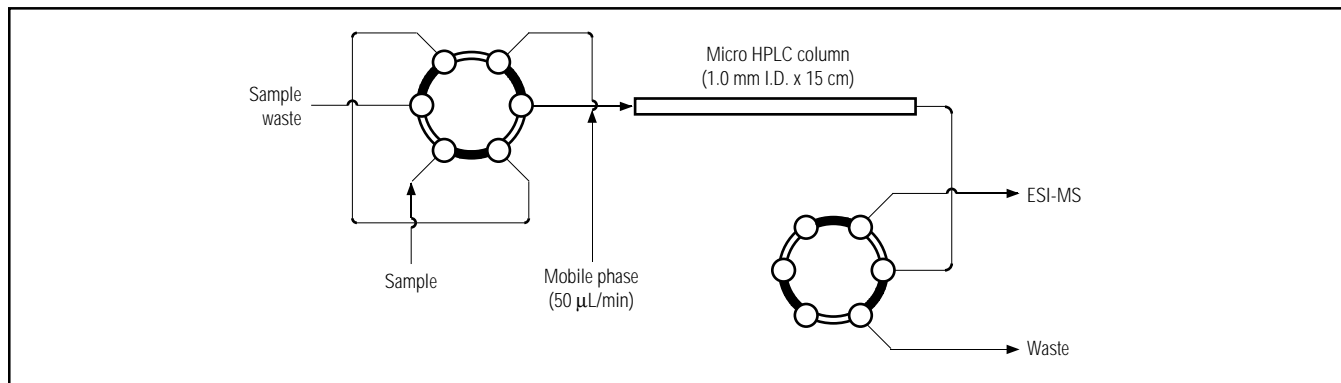


Figure 1. Instrumental set-up for on-line buffer removal in Micro HPLC prior to ESI-MS.

containing non-volatile buffer salts—after separation on a 1.0 mm I.D. Micro HPLC column—that was allowed to enter the ionization source. The current plot in Figure 2B corresponds to the same sample injection, with the exception that the column effluent was guided to waste for the first two minutes. Clearly, no buffer salts were introduced into the mass spectrometer with post-column flow switching.

The developed valve switching method was employed to study the presence of drugs in samples obtained from dialysis studies in artificial CSF containing 0.9% NaCl. Injections volumes were 5–10  $\mu\text{L}$ . This was achieved with a microliter pick-up injection routine (for details see Application Note 510). The results are given in the mass chromatogram (Figure 2C). With this valve switching configuration, the FAMOS™ micro autosampler allows buffer-salt removal prior to ESI-MS analysis. This facility of FAMOS is particularly useful in the analysis of  $\mu$ -dialysates and minute volumes of protein and peptide solutions.

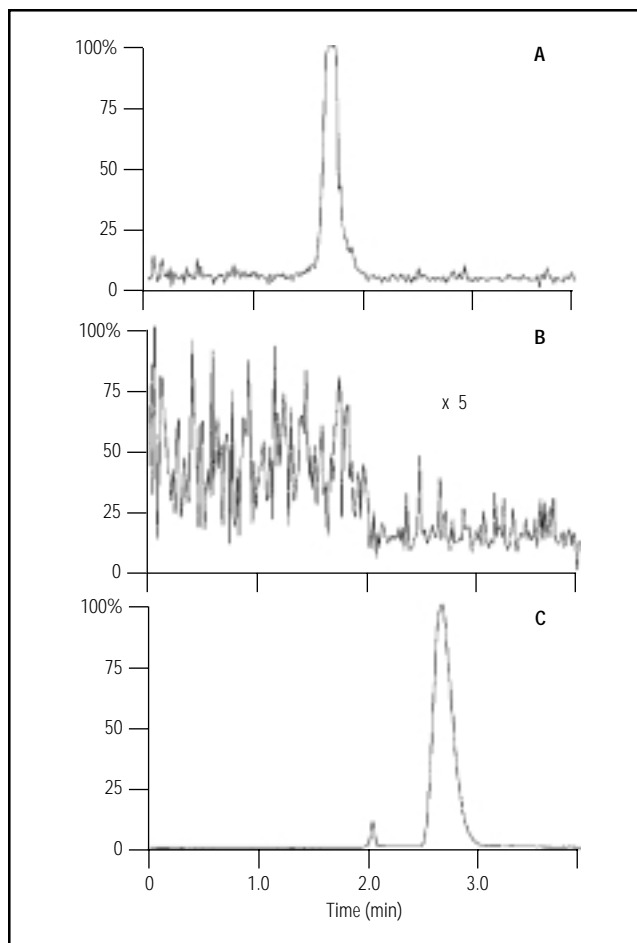


Figure 2. Current plots: A) micro LC column effluent introduced into the ESI-MS source containing salt and B) column effluent guided to waste for the first two minutes to remove salt. Plot C shows a reconstructed mass chromatogram after on-line salt removal, 5  $\mu\text{L}$  injection out of 6  $\mu\text{L}$  sample (rat dialysate).



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**Dionex Corporation**  
1228 Titan Way  
P.O. Box 3603  
Sunnyvale, CA  
94088-3603  
(408) 737-0700

**Dionex Corporation**  
Salt Lake City Technical Center  
1515 West 2200 South, Suite A  
Salt Lake City, UT  
84119-1484  
(801) 972-9292

**Dionex U.S. Regional Offices**  
Sunnyvale, CA (408) 737-8522  
Westmont, IL (630) 789-3660  
Houston, TX (281) 847-5652  
Atlanta, GA (770) 432-8100  
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**LC Packings**  
USA (415) 552-1855 The Netherlands 31 20 683 9768

**Dionex International Subsidiaries**

Austria (01) 616 51 25 Belgium (32) 3-353 42 94 Canada (905) 844-9650 China (852) 2428 3282 Denmark (45) 36 36 90 90  
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Switzerland (062) 205 99 66 United Kingdom (01276) 691722

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