Optimized Sample Delivery and Direct Carbon Detection for Two Microcoil Flow Probe Designs

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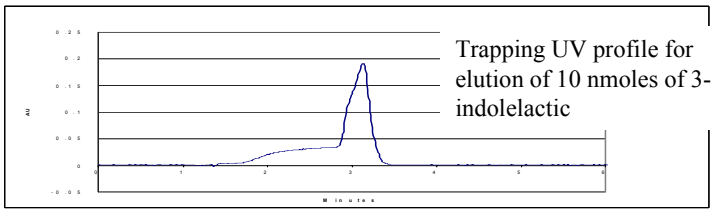
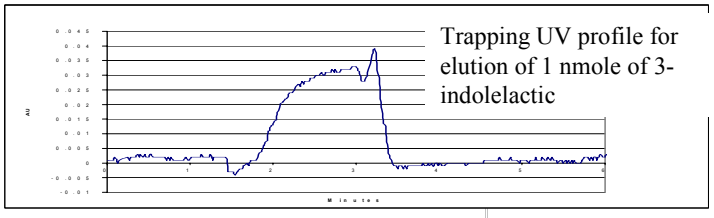
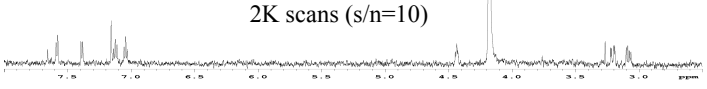
Abstract: In the previous ENC, we showed a methodology (column trapping), which allowed efficient transfer of samples into the 5 μL flow cell of a MRM capillary probe[1]. This methodology utilizes micro to capillary size HPLC columns as effective SPE (solid phase extraction) columns, which are then eluted in very volumes (peak width < 5 μL) into the NMR flow cell. In this session we build upon our work with an evaluation of the efficacy using a variety columns/packings in regards to % of analytes trapped, peak shape, and the volume of the analyte peaks. In addition, we will describe a valving and syringe configuration to maximize direct injection sample delivery. We will evaluate these transfer methods using a commercially available flow probe and a custom designed probe with an even smaller flow cell volume (3 μL).

These microcoil flow probes use a single coil triply tuned to deuterium, proton, and carbon. Unlike standard ID probes, this does not result in a filling factor loss when directly detecting carbon. Since this design achieves a high ^{13}C sensitivity, we will evaluate direct observe ^{13}C performance for the two probes.

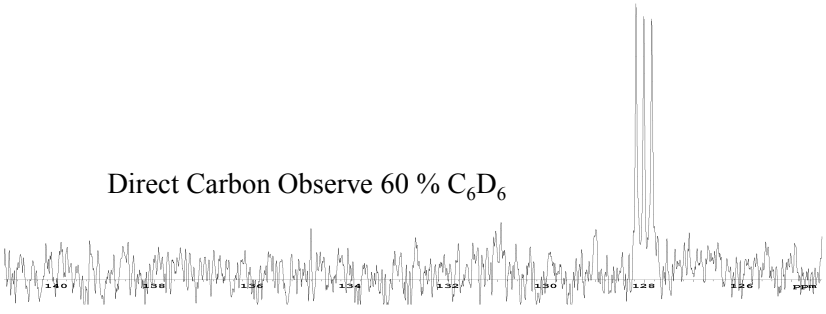




600 MHz ^{1}H-NMR spectrum of 1 nmole 3-indolelactic acid
2K scans (s/n=10)



Direct Carbon Observe 60 % C_6D_6



[1]**Column Trapping on an MRM CapNMR(TM) Probe,** Andreas Kaerner & Tim A. Smitka, Poster Presentation, ENC 2004