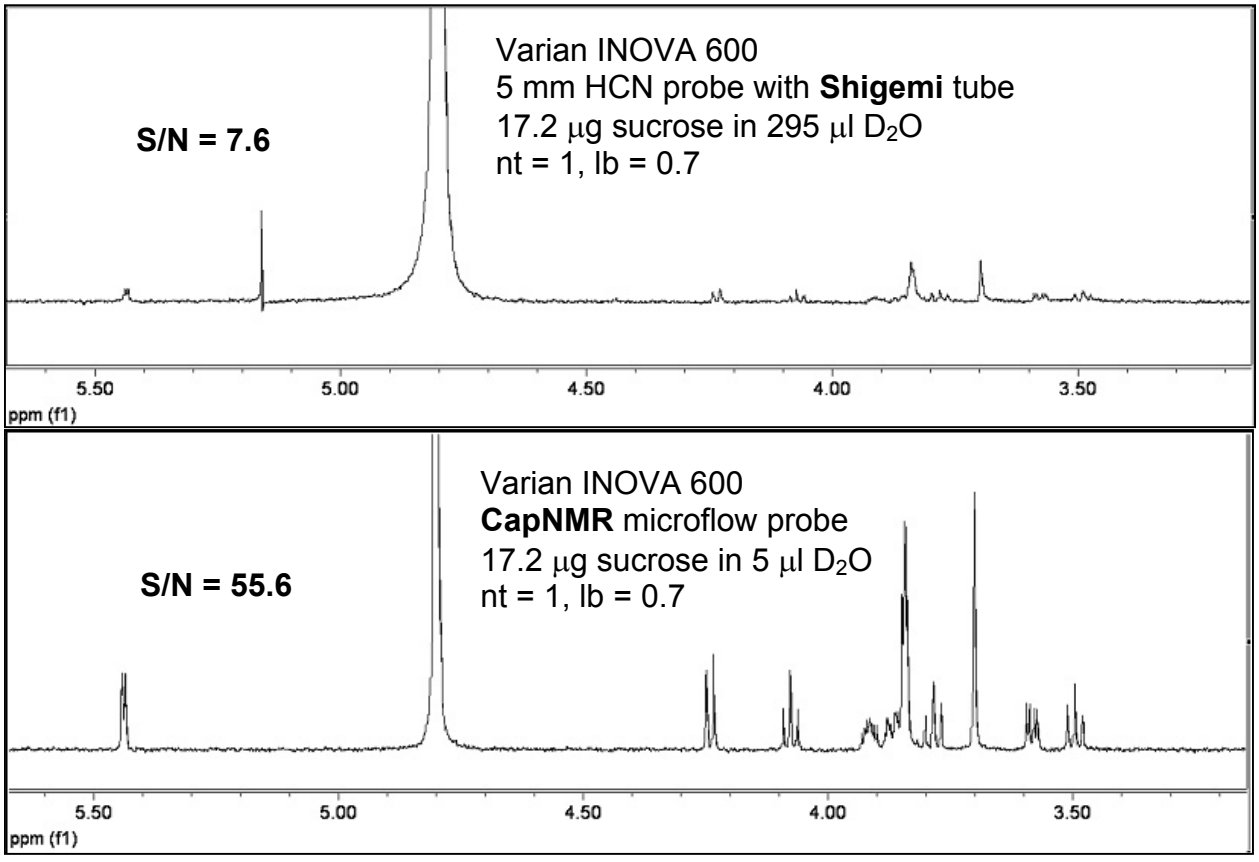
Protasis
Magnetic Resonance Microsensors

Mass-limited Sample Analysis using the CapNMR Probe:

Comparison of Capillary MicroFlow to Conventional 5 mm Probe with Shigemi Tube

Limitations in NMR sensitivity demand efficient utilization of sample mass for maximal signal-to-noise ratio. This need is most pronounced in the analysis of mass-limited quantities. The need to position the majority of the limited sample mass in the active (observe) volume of the radiofrequency coil has lead to development of susceptibility-matched tubes for this purpose. This application note demonstrates both the utility and the advantages of capillary microflow for mass-limited sample analysis.

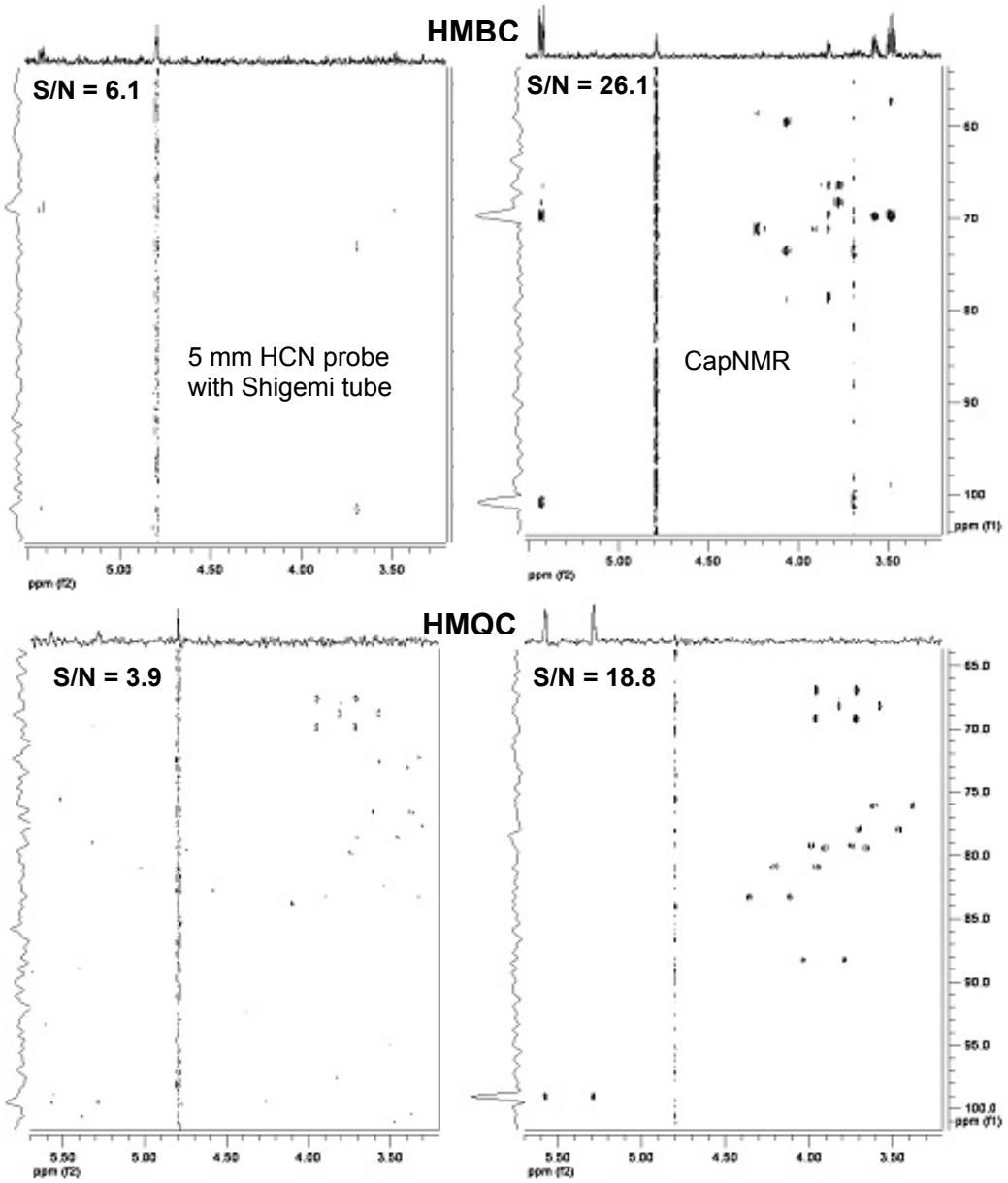
A comparison study between a Protasis/MRM HCN-gradient CapNMR probe and a conventional 5mm HCN-gradient probe with a Shigemi tube was conducted in the research laboratory of ***Dr. Frank Schroeder, Cornell University***. The spectra demonstrate significant advantage achieved from concentrating a limited mass of sample into a small volume and subsequently analyzing using a probe designed for enhanced mass sensitivity. Maximizing the ratio of the intensity of the sample signals to the intensity of the solvent signal provides emphasis of the signal features of interest while de-emphasizing the background and impurity peaks commonly associated with NMR solvents. The result is high spectral clarity and high signal-to-noise. Superior fluidic performance at the capillary scale combines with these enhanced sensitivity and signal quality advantages to provide a practical, cost-efficient, and reliable platform for mass-limited NMR analysis.



Single scan, 1H-NMR using 17.2 µg of sucrose in D2O. Experiments were conducted at 600 MHz using a Varian INOVA spectrometer. A line broadening of 0.7 Hz was employed.

top) results using a 5 mm HCN-gradient probe and Shigemi BMS sample tube with 295 µL of D2O solvent

bottom) results using a Protasis/MRM HCN-gradient CapNMR probe with 5 µL of D2O solvent. Data courtesy of Dr. Frank Schroeder, Cornell University, Ithaca, NY.



Heteronuclear multiple band coherence (top) and heteronuclear multiple quantum coherence (bottom) spectra from 17.2 µg of sucrose in D_2O. Experiments were conducted at 600 MHz using a Varian INOVA spectrometer. Acquisition times were 3.5 hrs and 14 hrs, respectively. Results demonstrate the S/N difference when comparing a conventional 5 mm HCN-gradient probe and Shigemi BMS sample tube with 295 µL of D_2O solvent vs. a Protasis/MRM HCN-gradient CapNMR probe with 5 µL of D_2O solvent. Data courtesy of Dr. Frank Schroeder, Cornell University, Ithaca, NY.

Experimental Conditions. For this study, the following experiments were performed: 1) 1D proton (single scan, previous page), 2) 2D non-gradient HMQC (3.5 hr, below), and 3) non-gradient HMBC (14 hr, below). In each case, 17.2 µg of sucrose as 5 µl of a 10 mM solution was used. For Shigemi tube analysis, the sucrose solution was diluted into 295 µL of D_2O in the tube. For microflow analysis, the 5 µL solution was directly injected into the CapNMR probe using a Protasis/MRM Manual Injection Module sample management platform (see Protasis/MRM Technical Bulletin G0020).

"With this most recent series of data demonstrations, we have now solidified on the Protasis/MRM CapNMR probe as our tool of choice for NMR analysis of precious samples of small molecules being studied in our research group. The probe is easy to use and provides superior data quality."

Dr. Frank Schroeder, Cornell University

The Protasis/MRM CapNMR Microflow probe provides an effective new approach to mass-limited analysis and is shown to provide performance levels above those achieved with NMR tubes. For a more detailed description of microflow techniques, see Protasis/MRM Technical Bulletin G0020 at www.microNMR.com.

For ordering information, contact your sales representative at (508)481-4163.