

A Benchtop NMR Spectrometer as a Tool for Monitoring Mesoscale Continuous-Flow Organic Synthesis: Equipment Interface and Assessment in Four Organic Transformations

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Interfacing the flow unit with the NMR spectrometer

To interface the flow unit to the picoSpin NMR spectrometer, we took advantage of the fact that both the inlet and drain assemblies on the NMR instrument are compatible with the standard PEEK nuts used on our Vapourtec E-series flow unit and on other similar mesoscale flow apparatus that is commercially available. As shown on page 2, the exit of the back-pressure regulator is linked to the NMR spectrometer by means of a length of PFA tubing. We operate in a bypass mode whereby we briefly divert the stream exiting the flow unit through the spectrometer. We achieved this by using the “waste / collect” valve on the flow unit. When the valve was set to “collect” the exit stream passed in to a collection vessel. When the valve was set to “waste” the exit stream passed into the NMR spectrometer capillary. Once material was loaded in to the capillary, we turned the valve back to “collect”. In this way, we could then record the NMR spectrum of the sample while the material was static. When we were ready to take another sample, we turn the valve back to “waste” to push out the prior sample and refill the spectrometer with new analyte.

