Rapid-Injection NMR

(1) Manufacturing the rapid-injection tubing

Material: PEEK tubing (0.02’’ID x 1/32’’ OD with OD/ID tolerances of +/-0.0005’’, orange, 50ft, order number 1569L from IDEX Health&Science, formerly Upchurch Scientific). PEEK shows high chemical durability, high precision for small diameter tube grafting, stiffness, low moisture adhesion and a low temperature expansion coefficient. Cut the tube to ~45 inches (aim slightly higher) and adjust the length to an exact void volume of 230 µl (this length is adequate for a 500 MHz spectrometer). This is achieved by pushing THF through the PEEK tubing with a gas-tight syringe. The needle is attached to the tubing by gluing on a 20 GA PTFE tubing spout with epoxy glue applied to the end of the PEEK tubing. This PTFE spout needs to be around 3 inches long (half of the length glued to the tubing and half serving for the needle to be inserted into).

(2) Setting up the reaction

Under vacuum flame-dry an NMR tube (22cm length, uncut, Norrell). The NMR tube is closed with an inner septum that is cut off at the top and an outer septum that wraps around. While under Argon, insert the PEEK tubing half-height into the NMR tube by feeding it through an 18 GA 2’’ Teflon hub needle. Then retract the needle to the other end of the PEEK tubing. Let the assembly purge for at least 5 minutes. Attach the injection syringe loaded with 330 µl of the solution to be injected in the NMR probe (230 µl void volume + 100 µl sample) at the PTFE spout. Inject 500 µl of reactant directly into NMR tube with tubing remaining at half-height. Cool the assembly down to -78 °C in a dry-ice bath with the Argon line still in place. Carefully feed the tubing to 2 mm from the bottom of the NMR tube (this matches the point in the NMR tube where the curvature starts). Injection at this point facilitates most efficient mixing when injecting. Remove the Argon line, grease the septum to close the punctured seal and transport the assembly to the NMR machine.

Remove the NMR tube from the cold bath, insert it into the spinner and mount it on the NMR machine. Be careful not to tug on the PEEK tubing to avoid altering the 2 mm spacing. Insert the sample as the tubing follows along with it. Be careful that the tubing is not tangled on top and that the syringe is safely placed on top of the spectrometer. Attempt to proceed swiftly as not to warm the NMR sample too much. Let the sample cool for about one to two minutes. Vigorously inject the total volume of 330 µl (100 µl actual). The NMR acquisition should be started prior to the injection.