



Dean L. Olson, Ph.D., Director
Applications, Installations, and Customer Support
Protasis/MRM Corporation
101 W. Tomaras Avenue, Savoy, IL 61874 USA
(217) 351-4359 (phone); (217) 352-6655 (fax)
E-mail: dolson@microNMR.com
Tech Support: www.microNMR.com

Technical Bulletin: MicroFlow NMR System Cleaning Procedure with Oxalic Acid 8 September 2009; DLO at Protasis/MRM

We recommend the following procedure to clean any MicroFlow NMR system, including the CapNMR probe, on a periodic basis. The frequency of cleaning should be determined by monitoring line shape, S/N, or lock level, whichever best suits your pattern of use.

This procedure can be programmed for One-Minute NMR users who employ D₂O in at least one of their pump solvents. Citation users can employ the method with D₂O as the selected solvent.

- 1) Make a 5% solution of oxalic acid in purified water (this is about 0.6 M)
 - Purified H₂O only, D₂O not necessary; 5 grams oxalic acid in 100 mL water is reasonable
 - Choose a high grade of oxalic acid purity such as Aldrich #241172
 - If you use the dihydrate, 0.6 M is about 7.5 grams in 100 mL of water
- 2) Switch the probe to water either manually or automatically
 - Before switching to water, be sure to rinse away any residual sample with its own solvent
 - Use acetone between immiscible solvents as needed
 - Keep in mind that oxalic acid is not soluble in DMSO and most organic solvents
- 3) Manually push in the 5% oxalic acid solution through the Valco filter and into the probe
 - Use a generous volume (~150 µL) to rinse the probe
 - Use the 250 µL syringe and the black syringe vise
- 4) Make sure the probe exit tube tip is immersed in clean water to keep the exit tip wet
- 5) Leave this mixture in the probe (no flow) at 45 degrees C for 4 hours
 - Push 50 µL of fresh cleaning solution through the probe at least twice during the heated time interval
- 6) Rinse well with water; keep in mind that oxalic acid is not soluble in DMSO and most organic solvents
- 7) Reconnect your solvent of interest, and rinse thoroughly

Oxalic acid exhibits three useful properties for cleaning:

- It's acidic
- It's reducing (reactive) to most contaminants
- It's a good chelation agent

Note

- If you shim using 5% chloroform in acetone-d₆, be certain to make a fresh sample, since an old one can get wet and not shim properly.
- Another sample option for shimming is 20-40 mM maleic acid in your solvent of interest.