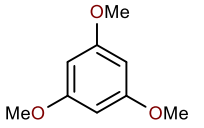
technique of the week

NMR Yields

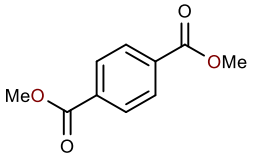
The SARLAH Group

Common NMR Internal Standards

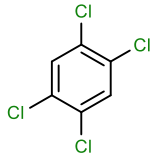
- Internal standard necessary to obtain NMR yields
- Signals of standard should not overlap with signals of product
- Some common NMR internal standards are shown to the right
- Check out the paper below for the chemical shifts and relaxation times of common NMR internal standards in different solvents



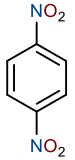
1,3,5-trimethoxybenzene



dimethyl terephthalate



1,2,4,5-tetrachlorobenzene



1,4-dinitrobenzene

Rundlöf, T. *J. Pharm. Biomed. Anal.* **2010**, *52*(5), 645–651. https://doi.org/10.1016/j.jpba.2010.02.007.

Sample Preparation

Step 1

Accurately weigh out your internal standard

Step 2

Make a solution of your internal standard

Step 3

Add an equimolar amount of your internal standard solution to your reaction(s)

Step 4

Work up your reactions as necessary (mini aqueous workup, syringe filter, etc.) and then concentrate and dry to avoid swamping your spectrum with solvent.

Any loss that occurs during the workup process should be approximately the same for your sample and internal standard, so adding the standard before workup gives the most accurate results

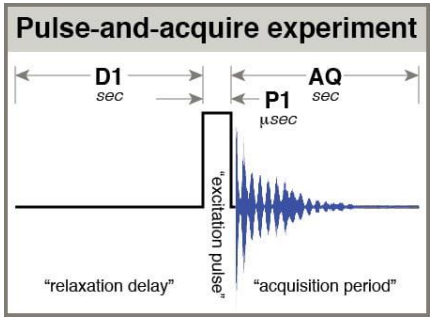
Step 5

Prepare your NMR sample normally

Step 6

Take your NMR spectrum. CB500 works best, but NMR yields can be taken on all the instruments.

Note on delay: it is recommended that the relaxation delay is 5x the T_1 delay of the proton you are measuring. This can be adjusted on the instrument software using d1 = [time (s)].



NMR Yields

Worked Example

Screening reaction conditions for a coupling. Here is the workflow I followed for my NMR yields:

- Expecting 0.026 mmol of product
- Prepared a 0.0354 M solution of dimethyl terephthalate
- Added 0.185 mL of internal standard solution to each reaction (0.026 mmol/0.0354 mmol/mL = 0.735 mL/4 ~= 0.185 mL)
- Mini aqueous workup, syringe filter the organic layer, and concentrate to dryness. Redissolved in $CDCl_3$
- Below is one of the NMR spectra I obtained (74% NMR yield for this reaction)