NMR titration procedure

Four identical NMR tubes containing:

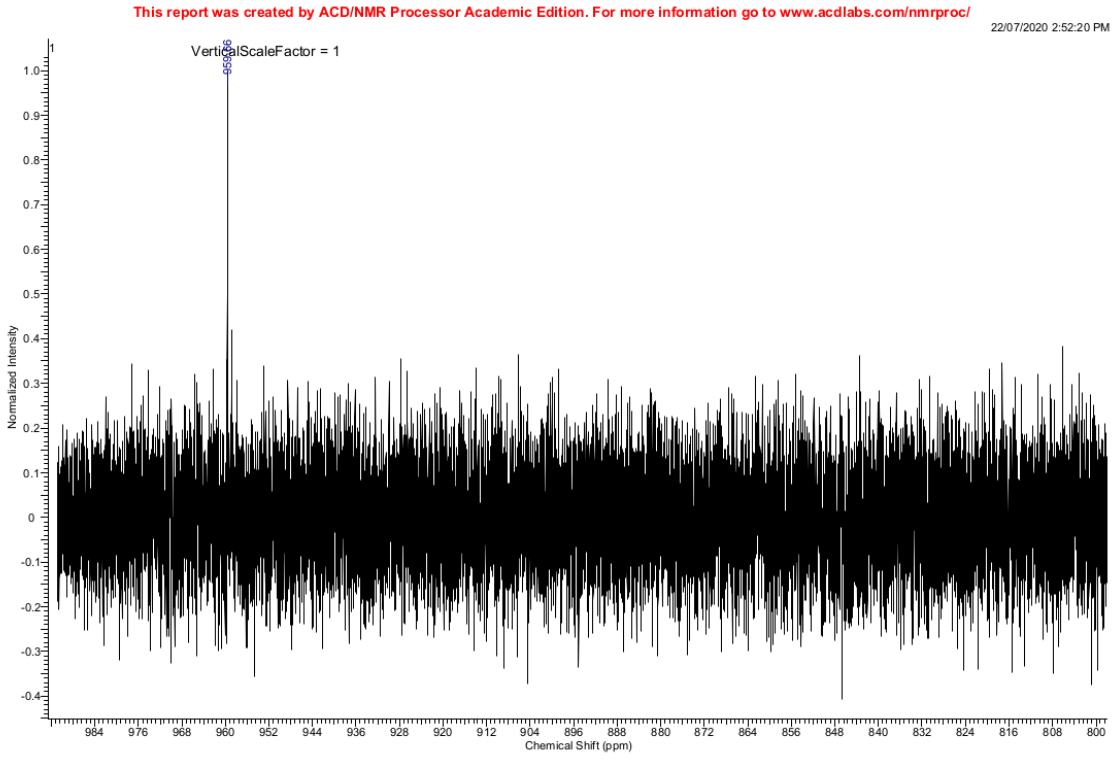
- 0.1M ebselen in CHCl3 (500uL)
- 200uL CDCl3 for lock

5M solutions of the following Lewis bases in CDCl3:

- Diethylsulfide (DES)
- Triethylamine (TEA)
- Dimethylacetamide (DMAc)
- Pyridine (py)

Note the ebselen doesn't fully dissolve initially. This doesn't seem to be a problem. It should dissolve once a few equivalents of base have been added.

Spectra should be acquired using a 60 degree pulse with 1s relaxation delay. 5 minute per spectrum is usually enough to give an unambiguous signal (appears as a singlet at 959.66ppm for pure ebselen, lower in the presence of base). Below is an example of an adequate spectrum, acquired over 112 scans



Add a 20uL aliquot of the base, mix, and acquire another spectrum. The signal should move upfield somewhat. If there's no significant change add more base (maybe up to 100uL at a time?). Rinse and repeat! Stop when the NMR tube is full 😊

Aim for 15-20 data points, recording the chemical shift and the amount of base added.

Below is a representative example with ebselen and DMAP (a very good base). The 77Se chemical shift moved upfield by 60ppm. This probably won't happen with any of these bases, but we'll see.