

## 1-D nOe Difference by noemult

IU NMR Facility – August 6, 2004

### *Summary:*

The  $^1\text{H}$  to  $^1\text{H}$  nOe effect is through-space (as opposed to through-bond) and can be detected for protons up to about 5 Angstroms apart, providing valuable positional information that can otherwise only be obtained with X-ray crystallography.

In the classic nOe difference experiment a peak is irradiated with low decoupler power for several seconds, then the 1-D NMR spectrum is recorded. Then another 1-D NMR spectrum is recorded with off resonance irradiation. The off resonance reference spectrum is subtracted from the original one to reveal nOe enhanced resonances.

In this simple and effective version the subtraction is done through phase cycling so only one spectrum is recorded – the actual nOe difference spectrum. In addition, with noemult you can irradiate up to 6 individual transitions of a multiplet (i.e. each of the 4 peaks of a quartet) with the lowest possible power for maximum selectivity.

You should consider running the 1-D selective NOESY or 2D NOESY experiments as alternatives!

### *Procedure:*

- 1) Turn spinning off. Acquire a  $^1\text{H}$  spectrum, phase and reference it. Type **gain='y'**.
- 2) Move the data to another experiment if you want to preserve the original, use **mf(1,2)** for example.
- 3) Set the decoupler frequencies for irradiation. Up to 6 can be specified, plus a last one that will always be the off resonance reference. Place the cursor on the first peak to irradiate, use **nl** to help find the top of the peak. Then use **sd ds** to create a new frequency list and to set the first irradiation frequency. Use **sda ds** to add the rest. Your last frequency will be the off resonance reference so needs to be somewhere with no nearby peaks – at one of the ends for example. If you have more than 6 peaks in a multiplet try irradiating between peaks.
- 4) Type **da** to make sure you have no more than 7 total frequencies and that the last one is off resonance. If you have say 3 total frequencies the first 2 should be on resonance and the third should be the off resonance reference.
- 5) Type **noemult** to convert the current experiment and you're ready to **go**.

### *Notes:*

- 1) You can also run the traditional nOe difference with one irradiation using noemult. Set up on and off resonance frequencies and increase **dpwr** to about 10-14. Place the on resonance frequency in the middle of the desired multiplet.
- 2) Pure, clean, not-too-concentrated samples will give the best results. In addition it is usually worth degassing your sample by at least bubbling  $\text{N}_2$  through it.