

**Title:** NMR Manual for 1D NOE NMR on Agilent Spectrometers

## Purpose

This manual will help NMR users understand the simplicity of proton-proton interaction thru space measurement by using 1D NOE method instead of 2D NOESY. Users can easily follow the procedures to conduct their experiments with little or even no NMR staff assistance.

## Introduction

NOESY experiment (Nuclear Overhauser Enhancement Spectroscopy) is based on dipole-dipole coupling (also called thru-space decoupling) and used for measuring proton-proton distance. The disadvantage of 2D NOESY is that the experiment takes a relatively long time. For a small molecule, 1D NOE is more efficient if there's only one or two dipolar correlations expected.

The idea is that we can use NOE pulse sequence to irradiate one proton peak interested or two to observe any other protons nearby (usually the distance  $< 5 \text{ \AA}$ ). For a small molecule (usually MW  $< 1000$ ), the irradiated peak and NOE peaks have the same phase; while for a large molecule (usually MW  $> 2000$ ), the irradiated peak and NOE peaks have the opposite phase.

## Preparation

**Prerequisite:** users have done the basic NMR training

**Spectrometer:** Any, but NMR-Hg400 and F500 recommended

**NMR Sample Prep:** Relatively higher concentration (20 mg or more in 1 mL deuterium solvent)

**Reading:**

- Agilent VnmrJ 3.2 Experiment Guide, page 220-224  
[http://129.105.219.103/nmr/vj\\_experiment\\_guide.pdf](http://129.105.219.103/nmr/vj_experiment_guide.pdf)
- Agilent example  
[https://www.chem.agilent.com/Library/applications/5990-8881en\\_lo.pdf](https://www.chem.agilent.com/Library/applications/5990-8881en_lo.pdf)

## Experiment Setup

- 1) Login on FOM system, then, login on spectrometer computer by netid
- 2) Insert NMR sample into magnet and do locking and shimming as usual
- 3) Tune H1
- 4) Run a regular 1D H1 (starting from exp1)
- 5) Setup 1d NOE based on regular 1d H1 in exp2, by commands **jexp2 mf(1,2) wft**
- 6) Load NOSY1D experiment into exp2 (Figure 1)
- 7) Select a proton peak to be irradiated (Figure 2 and 3)  
Note: delta value between two cursors is better in a range 0.02 – 0.2 ppm
- 8) May change Number of scans and Mixing Time (Figure 2), then click button **Go!**  
Note: mixing time is better in a range 100 – 800 ms (the smaller molecule, the larger mixing time)
- 9) Save all the data (exp1 and exp2) into a correct folder



Figure 1. Experiment Selector Panel

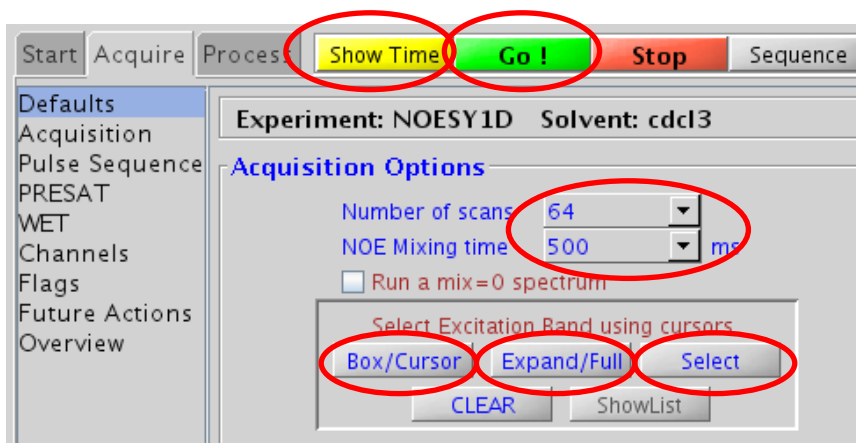


Figure 2. NOESY1D Setup Panel

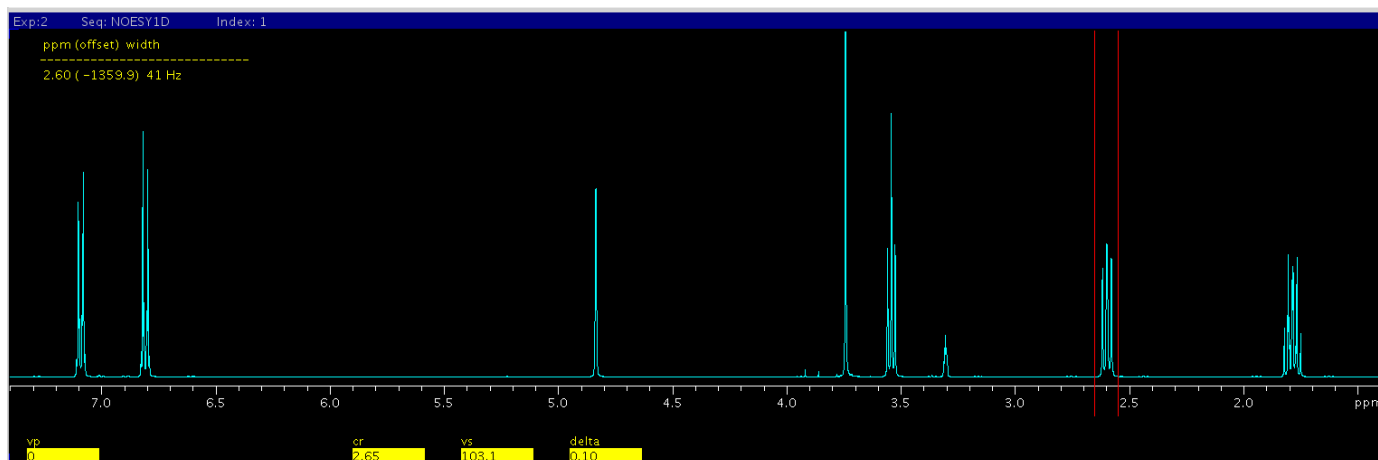


Figure 3. Select a peak to be irradiated

## Ending Work

- 1) Switch a dummy sample into the magnet
- 2) Lock on CDCl<sub>3</sub>
- 3) Write a note on Log Book
- 4) Keep desk top clean
- 5) Logout from spectrometer computer and FOM

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