

Title: NMR Manual for Hetero-nuclear Overhauser Enhancement Spectroscopy (HOESY) Experiment

Purpose

This HOESY manual will help NMR users to understand the F-H 2D correlation NMR experiment and follow the procedures to conduct their experiments with little or even no NMR staff assistance

Introduction

Proton (H1) and fluorine (F19) are coupled to each other in the same molecule through two mechanisms: 1) scalar-coupling-based or called through-bond interaction, and 2) dipolar-coupling-based or called go-thru-space interaction.

HOESY (also called F-HOESY) is a dipolar coupling based experiment. If there are a proton and a fluorine (simplest case) close to each other (usually the distance $< 5 \text{ \AA}$), there will be a cross peak on the 2D HOESY map. The peak intensity indicates the distance between two atoms, which helps chemist to determine the molecular structure.

Formerly, this type of experiment was very difficult because both nuclei are in the same channel (high-band channel). Thanks to the funding from International Institute of Nanotechnology, IMSERC purchased a brand new NMR-F500 in 2013 which is equipped with a three-channel console (high-band/broad-band/high-band) and a special HFX probe. With this new machine, we are able to use a special pulse sequence to radiate both nuclei in the different channels and to run HOESY experiments easily.

Preparation

Spectrometer: NMR-F500 only

Probe: HFX probe only (also called idauto probe)

Prerequisite: users have done the basic NMR training

Reading: <http://www.chem.agilent.com/Library/applications/5990-9229EN.pdf>

Experiment Setup

- 1) Login on FOM system, then, login on F500 computer by netid
- 2) Insert NMR sample into magnet and lock and shim as usual
- 3) Tune H1 and F19
 - i. H1 is in channel 1 and F19 is in channel 3
 - ii. Open a tune panel by click **Start > Standard > Tune (Figure 1)**
 - iii. Tune both H1 and F19 at same time by click H1/F19 (**Figure 2**)
 - iv. Wait for a minute or two, until it's finished automatically (status window showing **Idle**)
- 4) Run a regular 1D H1 and 1D F19 NMR and save data to a correct folder
- 5) 2D HOESY set up is based on 1D F19 experiment
- 6) Optional: you may use commands **mf(#1,#2) jexp#2** to copy 1D F19 to a new experiment
- 7) From StudyQ, select **Protocol > Liquids > HF_Expts>FH_HOESY (Figure 3)**

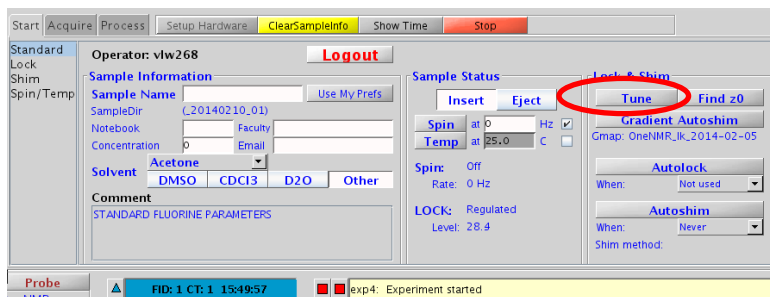


Figure 1. VnmrJ Start/Std Interface showing Tune Button.

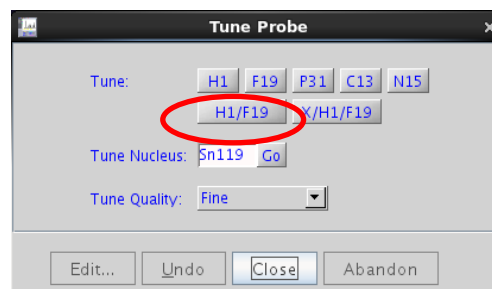


Figure 2. Tune interface



Figure 3. Expt Selector.

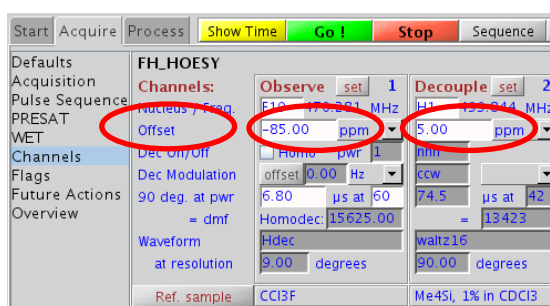


Figure 4. Parameters in Channels panel.

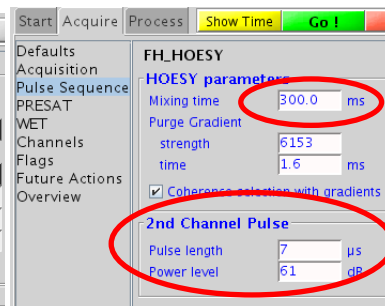


Figure 5. Pulse Sequence panel.

- 8) Check all parameters, such as **sw**, **sw1**, **tof**, **dof**, **mix**, **2nd Channel pulse**, etc.
 - i. **sw** is the spectral width from 1D F19 (Hz or ppm)
 - ii. **sw1** is the spectral width from 1D H1 (Hz or ppm)
 - iii. **tof** is the transmitter offset from 1D F19 (Hz or ppm) (Figure 4)
 - iv. **dof** is the transmitter offset from 1D H1 (Hz or ppm) (Figure 4)
 - v. **mix** is mixing time in milliseconds (use default or 300) (Figure 5)
 - vi. **2nd channel pulse** is manually copied from 1D H1 spectrum (Figure 5)

9) Acquire data

10) Save data in a correct folder, i.e. /walkon/PI_name/user_netid/

Ending Work

- 1) Switch a dummy sample into the magnet
- 2) Lock on CDCl₃
- 3) Write a note in Log Book
- 4) Keep desk top clean
- 5) Logout from F500 computer and FOM