

Varian 600: 1H DOSY experiment

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Instrument: Varian 600 MHz

Probe: AutoX_DB_PFG_8964

Goal: This document describes the basic setup of the 1H DOSY experiment on VnmrJ 2.2

Initial setup

1. Turn temperature regulation off (**Start:Spin/Temp: Temp Off**)
2. Prevent experiments from starting temperature regulation: issue **vttype=0**
3. Load good starting shims: rts ek_current
4. lock/tune/grad shim
5. Calibrate 1H 90o pulse

Set up basic DOSY parameters

1. Set up pulse sequence Dbppste_cc (menu **Experiments : DOSY : 2D Dosy with Convection Compensation : Bipolar Pulse Pair Stimulated Echo**)
2. Enter maximum gradient strength **gzlvl_max=26640** (I determined this from specs of the probe for 10G/cm and extrapolated to 60 G/cm)
3. **gzlvl1=22017** (this is to allow unbalanced bipolar gradients)
4. Enter proton parameters: **pw=.... tpwr=.... tof=-280** (for water, -3100 for 0ppm)
5. Adjust acquisition time and recycle delay
6. nt=16 (or any multiple of 16)
7. Check signal:
 - uncheck box **Pulse Sequence : DOSY Options : DOSY on/off**
 - run to see spectrum

Optimize DOSY-specific parameters

Find optimal gradient range

1. Set **Pulse Sequence : DOSY Setup : Number of increments** to 2
2. Click **Setup DOSY using conditions above**
3. Run experiment
4. **wft, dssh**
5. Adjust to obtain approx. 90% decay of signal intensity (less is OK too)
 - a. **Pulse Sequence : DOSY Parameters: Diffusion gradient length** to ≤ 3 ms
This is gradient pulse - strongly attenuates amount of signal

b. **Pulse Sequence : DOSY Parameters: Diffusion delay** to 25 - 100 ms

This is waiting time - less strong

Set up a gradient range

1. Set **Pulse Sequence : DOSY Setup : Number of increments** to 5 or 10
2. Click **Setup DOSY using conditions above**
3. issue **DAC_to_G=0.0017520**
(this value should be replaced when better calibration of the gradient strength is made)

Analysis

Important: **Do not** click **Process : DOSY Process : Calculate Full DOSY** until the data collection is complete!

1. To see the progress issue **wft dssh** or **Process : Default : Transform all 1D's and Display : Horizontal**
2. To process click **Process All Spectra** then check **Use peak heights**. Set threshold for peak detection and click **Calculate Full DOSY**
3. copy-paste the calculation results into a separate text document
4. save screenshots of the 1D and 2D spectra
5. Repeat p.2 if you need new region

Currently, system does not plot but you can print using File : Print Screen : Graphics Area

Running new experiment

1. issue **ni=0**
2. After changing the gradient array: issue **DAC_to_G=0.0017520**

6.0 to 7.2 cm²/sec