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 31P DOSY experiment on VnmrJ 2.2

Initial setup

1. Turn temperature regulation off (**Start:Spin/Temp: Temp Off**) or set it just above room temperature: for example =24 (which is 25C)
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 90o pulse

Set up basic DOSY parameters

1. Use the Doneshot experiment set up previously
2. Adjust acquisition time and recycle delay
3. nt= any number (better - any multiple of 16)
4. Check signal:
   - uncheck box **Pulse Sequence : DOSY Options : DOSY on/off**
   - run to see spectrum
5. check box **Pulse Sequence : DOSY Options : DOSY on/off**

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 - allows for more diffusion but reduces signal intensity

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. Run experiment with [Acquire and Transform] button

Analysis

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

1. To see the progress click **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. create new workspace: **cexp(number)**
3. move parameters **mp(current number, new number)**
4. join and start acquisition