AVIII-400 Solution

Diffusing coefficient measurement

Acquisition of diffusion data

- **Remove** magnet cap
- **Make** sure **LOCK** is off, **SPIN** is off
- **Eject** standard sample
- **Insert** new sample
- **Drag and drop** the 1H parameter file
- **Create** a new dataset
- **Check** the **temperature** with the edte command
  Since diffusion coefficient depends on temperature, controlling the temperature is particularly important; set it to 298K (25C) and wait for 15 minutes before making any measurement.
- Type **rsh std.shim** (Read standard shim file)
- **Lock** field selecting the correct solvent
- Type **runh** (collect a quick 1H spectrum)
- Type **wrpa 2**
- Type **re 2**
- **Spectral width optimization** can be done at this point, if necessary (How is spectral width optimization done?)
- **Calibrate 90o pulse**, and note down the 90o pulse width (How is 90o pulse calibration done?)
- Type **iexpno**
- Type **rpar** and select "Diffusion"
- Set **P1** to the value you determined in the 90o pulse calibration step
- Set **RG** to the value you determined after running "runh" macro
- Set **NS** to a value appropriate for your sample; set **DS** to 4
- Verify the following parameters are set correctly
  D1=5
  D16=0.0001
  D20=0.025
  GPNAM6=sin.100
  GPNAM7=sin.100
  GPZ7=-17.13
  P19=600
  P30=2000
- Type **gpz6 2**
- Type **rga**

- Type **zg;ef;apk;abs**
- Type **iexpno**
- Type **gpz6 95**
- Type **zg;ef;apk;abs**
- Compare the spectra of EXPNO 3 and EXPNO 4
- The intensity difference of the two spectra should be a factor of ~50, if the difference is <50, change P30 and D20 in both data sets and rerun the spectra; repeat until the intensity difference is ~50.
- Type **iexpno**
- Select the "AcqPars" tab by clicking on it
- Change the **PULPROG** to stebbpgp1s
- Change the dimension from 1D to **2D**
- Set **TDF1** to **16**
- Set **FnMode** to **QF**
- Type **dosy**
- Enter **2** for first gradient amplitude
- Enter **95** for the final gradient amplitude
- Enter **16** for the number of points
- Enter **l** for the ramp type
- Click **ok** to acquire

Processing of diffusion data

- Select the "**ProcPars**" tab
- Set the following parameters
  SI(F1) = 16
  ph_mod(F1) = no
- Type **setdiffparm**
- Type **xf2**
- Type **abs2**

Determine diffusion coefficient

- Click on **Analysis** and select **T1/T2** relaxation
- Click on Extract Slice
- Click on Spectrum
- Select Slice number 1
- Click on ok
- Phase-correct the spectrum manually
- Click Define Ranges
- Manually integrate the regions
- Click on "disk" icon to "Export Regions to Relaxation Module"

- Click “Relaxation Window”
- Enable Intensity (or Area depending upon the quality and the nature of the spectrum)
- Click on “Fitting Function”
- Click on Close
- Verify the following parameters
- Click on ok
- Click on “Start Calculation”
- Click Close
- You should be able to read diffusion coefficients from the display
- Click on “Display Report” to display intermediate and final information on diffusion coefficient measureemnt

Finish up

Make sure **SPIN** is off
Make sure **LOCK** is off
Eject sample
Insert standard sample
Lock field selecting D2O
Place cap on the magnet bore
Make logbook entry