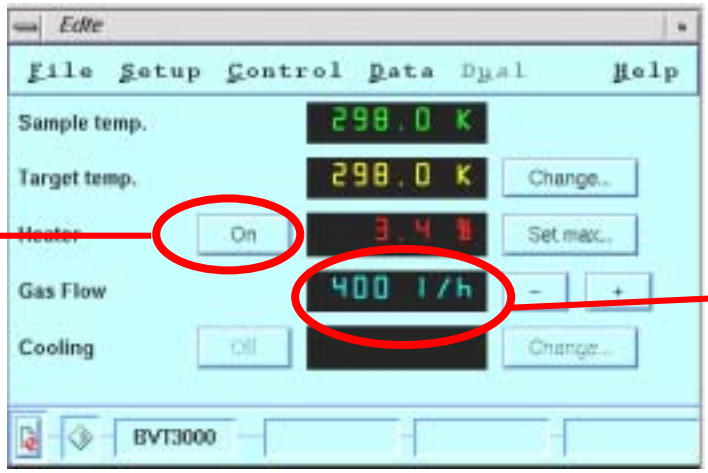
2004 NMR Users Training Course

02/27 *Lab Session Work Sheet*

Simple Outline

- Hardware (CRYOPROBE)
  Please make sure everything is normal for the Cryoprobe system !!

- Loading Sample
  (i) check temperature (“edte” {command line}: edit temperature)



Always ON !!!!

Keep the default setting value. Never Change !!!!

(Temp limitation for CryoProbe is 283K-333K, check Temp Cal. Table for setting desired temp)

(ii) check spinner (sample holder with yellow line) position
(iii) lift gas (make sure there’s lift gas) {click botton}
(iv) load sample
(v) lift off (now the sample should go down slowly) {click botton}
(vi) lock the solvent (“lock” {command line}, and click on your D-solvent)

- Wobble (Don’t need to change cable anymore!!!)

(i) check “edasp” setting {command line}
(ii) type “wobb” {command line} (this might take 20-30 sec, please be patient)
(iii) type “acqu” {command line} to observe wobble curve
(iv) adjust “Tune” and “Match” of probe **(Carefully!! Never over tune !!)**
(v) click on “ wobb-sw” to switch to another nuclei if necessary (F3→F2→F1)

- Shimming (gradient shim)

Exercises

- Data Collection Easy 1-2-3
  1. start a new experiment : new or edc
  2. read in parameters for standard experiment :rpar "CRP_*"
  3. Adjust parameters for your own sample

  (p.s.) Users just need to optimize experiments by adjusting parameters that list under "AQ parameter to check"

- Pulse & Parameter Calibration 1-2-3
  1. optimize O1 , especially for H2O suppression purpose
  2. optimize useful 1H pulse length
     (Please read the Pulse and Power Recommendation from Bruker)
  3. d1+aq has to be longer than 1 sec

Experiment (1)

Experiment Name: 2D 15N-1H HSQC

Experiment Type: Using echo-antiecho, f1: H, f3:N

Standard Parameter Set: CRP_2D_15N_HSQC_ETSI

Pulse Program: hsqcetf3gpsi2

AQ parameters to check

1H pulses

pl1(high power, ex: -5dB), p1(90^o pulse at pl1), p28 (trim pulse, ex:1m)

Others

cnst4 (J_{H-N} , ex: 90Hz), d24 ($1/4J_{H-N}$), o1 (for 1H), o3 (for 15N),

1 sw, 1td (for F1 dimension, N), , 2 sw, 2 td (for F2 dimension, H)

d1, ns(=1*n), ds(>=16), rg

**** Let's see what happen without 15N decouple??**

Experiment Name: 2D 15N-1H HSQC

Experiment Type: Using echo-antiecho, f1: H, f3:N

Standard Parameter Set: CRP_2D_15N_HSQC_ETSI

Pulse Program: hsqcetf3gpsi2

AQ parameters to check

Keep everything the same as above, but pL16=120dB !!!!!!

Experiment (2)

Experiment Name: 2D 15N-1H TROSY

Experiment Type: Using echo-antiecho, f1: H, f3:N

Standard Parameter Set: CRP_2D_15N_TROSY_ETSI

Pulse Program: trosyetf3gpsi

AQ parameters to check

1H pulses

pl1(high power, ex: -5dB), p1(90^{o} pulse at pl1)

Others

cnst4 (J_{H-N} , ex: 90Hz), o1 (for 1H), o3 (for 15N),

1 sw, 1td (for F1 dimension, N), , 2 sw, 2 td (for F2 dimension, H)

d1, ns(=4*n), ds(>=16), rg

Experiment (3)

Experiment Name: 2D 15N-1H CRINEPT

Experiment Type: Using echo-antiecho, f1: H, f3:N

Standard Parameter Set: CRP_2D_15N_CRINEPT

Pulse Program: crineptgpph

AQ parameters to check

1H pulses

pl1(high power, ex: -5dB), p1(90^{o} pulse at pl1)

p11 (shape pulse , ex: 1000us), sp1 (shape pulse level for Sinc1.1000)

Others

cnst4 (J_{H-N} , ex: 90Hz), o1 (for 1H), o3 (for 15N),

1 sw, 1td (for F1 dimension, N), , 2 sw, 2 td (for F2 dimension, H)

d1, ns(=2*n), ds(>=16), rg

(p.s.) Using AU-program “ splitcrinept” to process data
