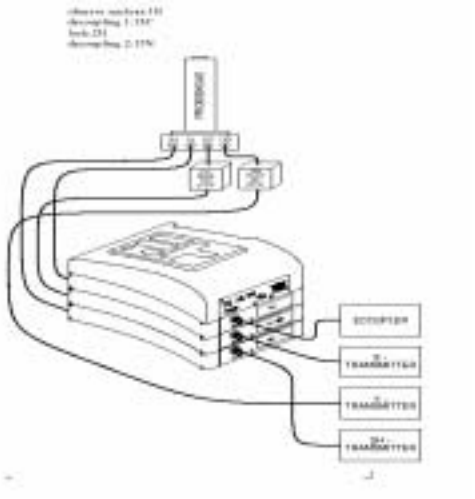
Basic Operation for NMR Systems in Core Facility

09/29 *Lab Session Work Sheet*

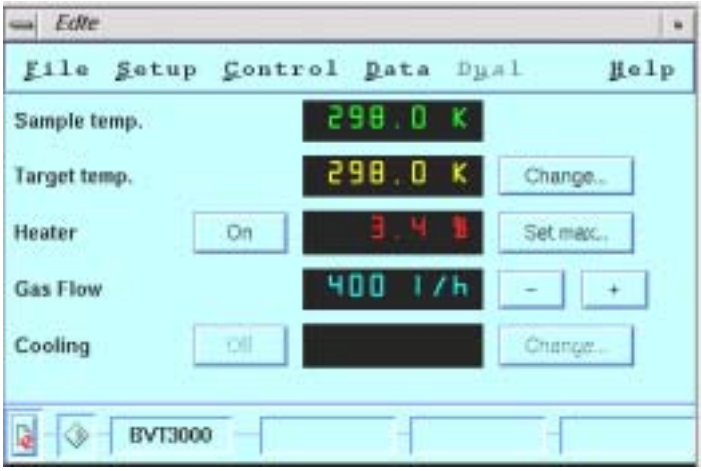
Simple Outline

- Hardware connection
  - (i) make sure there's no sample in the probe
  - (ii) check the connection of cables, heater, sensor, auto-detector……



- Loading Sample

(i) check temperature ("edte" {command line}: edit temperature)



(ii) check spinner (sample holder) position

(iii) lift gas (make sure there's lift gas) {click botton}

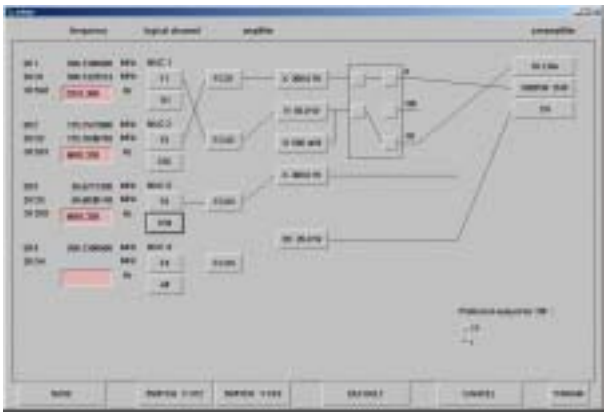
(iv) load sample

(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)

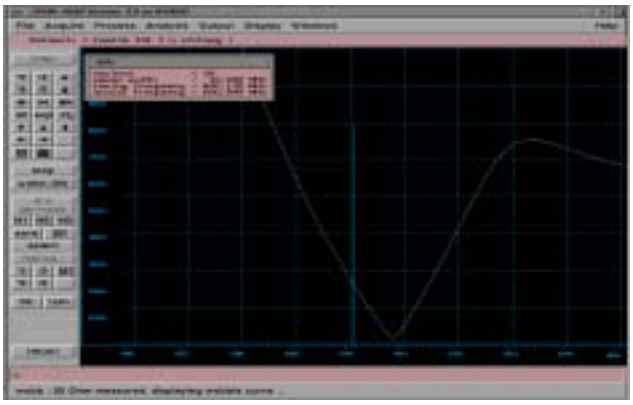
- Wobble (one or two channels)

(i) change connection if necessary (“edasp” {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 (do not over tune !!)

(v) type “stop” to stop wobble{command line}

- Shimming (gradient shim)

(i) read exist shim file , then shim manually

BBO: rsh std_BBO_* {command line}

TXI: rsh std_TXI_* {command line}

QXI: rsh std_QXI_* {command line}

(ii) Or, if available, run gradient shim (still need to adjust those axis without gradient)

- Data Collection Easy 1-2-3

1. start a new experiment : new or edc
2. read in parameters for standard experiment :rpar “std_*”
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”

Exercises

Experiment (1)

- Experiment Name: 1D ZG
- Experiment Type: one pulse for 1H
- Standard Parameter Set: std_1D_1H_ZG
- Pulse Program: zg
- AQ parameters to check

  1H pulses pl1(high power, ex: 0dB), p1(90° pulse at pl1, ex: 10usec)

  Others o1, sw, td, d1, ns, rg

Experiment (2)

- Experiment Name: 1D H2O suppression
- Experiment Type: H2O suppression using presaturation
- Standard Parameter Set: std_1D_1H_ZGPR
- Pulse Program: zgpr
- AQ parameters to check

1H pulses

pl1(high power, ex: 0dB), p1(90° pulse at pl1)

pl9 (low power for presat, 58-63db)

Others

o1(on H_2O), sw, td, d1,ns,rg

Experiment (3)

- Experiment Name: 2D 1H TOCSY MLEV17
- Experiment Type: H2O suppression using 3-9-19 & gradient
- Standard Parameter Set: std_2D_TOCSY-MLEV
- Pulse Program: mlevgpph19
- AQ parameters to check

1H pulses

pl1(high power, ex: 0dB), p1(90° pulse at pl1)

pl10 (power for p6, MLEV mixing), p6(90° mixing pulse, ex: 25u)

pl18 (power for 3-9-19, ex: 10 db), p0,p27(90° pulse at pl18)

Others

d9(mixing time, ex: 60-70ms), d19(=1/2d, d=distance of next null in Hz)

o1(on H_2O), 1 sw, 1 td (for F1 dimension), 2 sw, 2 td (for F2 dimension)

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

Experiment (4)

Experiment Name: 2D 15N-1H HSQC

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

Standard Parameter Set: std_2D_15N_HSQC_ET

Pulse Program: invietf3gpsi

AQ parameters to check

1H pulses

pl1(high power, ex: 0dB), 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

Experiment (5)

- Experiment Name: 3D HNCO
- Experiment Type: Using Echo/antiecho , f1: H, f2:C, f3:N, F1(CO), F2(N), F3(H)
- Standard Parameter Set: std_3D_HNCO
- Pulse Program: hncogp3d.2
- AQ parameters to check

1H pulses

pl1 (high power, ex: 0db), p1(90deg at pl1), p2(180deg at pl1)

pl19 (low power for dipsi2,pcpd1), p26(90deg at pl19), pcpd1(90deg ,ex: 40-50usec)

sp1 (shape pulse power for Sinc.1000) , p11(pulse length for sp1, ex: 2m)

Others

o1 (for 1H), o2 (for 13CO), o3 (for 15N)

1 sw, 1 td (for F1 dimension, ie: 13C), 2 sw, 2 td (for F2 dimension, ie:15N),

3 sw, 3 td (for F3 dimension, ie:1H)

d1, rg, ns(=8*n), ds (≧ 16)

Simple Operation Guide for BRUKER AV System

Preparation (Please make sure the probe in magnet is the one you need, and check all connections (cables, sensor, heater, gradient, gas line, detector)

Step 1: Starting xwinnmr

1. Login into PC
2. Double click on "xwin3.1" icon (The xwinnmr window will pop out)

Step 2: Temperature control

1. Type "edte" <enter>
2. The temperature control sub-window will pop out, set up your temperature
3. Type "te" <enter> to update your record

Step 3: Loading sample

1. Adjust sample position
2. Click "Lift" button on [BSMS] unit, you should hear the air flow
3. Load your sample
4. Click "Lift" button on [BSMS] again, the sample should reach the probe

Step 4: Wobble: Match and Tune

1. Start a new data set : (a) type "new" <enter> , or (b) type "edc" <enter>
2. Type "rpar std_*" to choose parameter set from standard experiments (see the " STANDARD EXPERIMENT LIST" for detail)
3. Type "edasp" <enter> to check the cable connection, then <EXIT>
4. Type "wobb" <enter>
5. Type "acqu" <enter> to observe wobble curve
6. Matching and Tuning on [HPPR] (do "X" channel first, then switch to "H")
7. Type "stop" <enter>

Step 5: Lock and Shimming

1. Type "lockdisp" <enter> , the lock sub-window will pop out
2. Type "lock" <enter> to choose the solvent
3. Type "rsh" <enter> to read an existing shim file, and shim Z1, Z2 manually
4. Type "gradshim" <enter>
5. Choose appropriate shim method (ex: 1D for 90% H2O, 1D2H for other 2H solvent)
6. Choose shim step file, then click on Start Gradient Shimming

Data collection (all data will store on d:/data/username/nmr)

- **Easy steps for beginner (1D):**

1. Type "rpar" <enter> to load an appropriate parameter set
2. Type "ns" <enter> to input number of scan
3. Type "rga" <enter> to find appropriate receiver gain
4. Type "zg" <enter> to collect spectrum
5. Type "ft" <enter> to do Fourier Transfer
6. Click on phase to phase spectrum
7. Click on return, then save to save the spectrum
8. Print out the spectrum
9. Save your data on floppy

- **Steps for optimize 90 deg pulse:**

<Look for H2O position, ie. O1 value>

1. "rpar " <std_1D_1H_ZG>
2. "zg" → "ft" → <phase>
3. Click on <utility> to define O1 position

<To find H 90 deg pulse> (for example:, pL1=0db, p1=? u, p1=35u. pL1=?db.....)

4. "rpar" <std_1D_1H_ZGPR> , and change O1 to the value from step 3 (if not in H2O, just use std_1D_1H_ZG to determine 90 deg pulse

(trick: you may use "gs" to optimize O1 value more correctly)

5. Set pL1= 0db (or 10db or ? db), p1=5u

(on the other hand, you may fix p1=40u*2, and look for pL1, ex: pL1=20db)

6. "rga" → "zg"→ "ft" →<phase> → <save>
7. Keep pL1, increase p1= expected 180 deg

(or, keep p1=180 deg pulse length, decrease pL1=expected power level for p1 value)

8. "zg"→ "fp" →check if the spectrum almost flat, if not, repeat step 8
9. Now, you should have a table with 1H pulse information of your sample
   For example:

XX protein	Power level (dB)	90 deg Power length (usec)
High power	0	?
Lower power	10	?
Decouple power	?	40
For Mixing	?	25
Others	?	?
Others	?	?

- **Experiment set up steps for advance users**

1. Type "rpar" <enter> to load an appropriate parameter set (refers to "Standard Experiment List")
2. Check "Experiment Guide" for parameter information (only need to change those parameters list on the guide, i.e. the parameter in red)

Experiment Name: 1D ZG

Experiment Type: one pulse for 1H

Standard Parameter Set: std_1D_1H_ZG

Pulse Program: zg

AQ parameters to check

1H pulses

pl1(high power, ex: 0dB), p1(90° pulse at pl1, ex: 10usec)

Others

o1

sw

td

d1

ns

rg

3. Input the correct parameters for your specific sample
4. Type "zg" <enter> to collect spectrum (or "multizg" <enter> if you have several experiment to run)
5. Transfer your data to floppy
   (To save the space on local disk for all users, please do not process your data on the local disk. Or, please remove your process data before logout.)

Still have problems? Please contact facility staffs for assistant.

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STANDARD EXPERIMENT LIST for BBO

by Chi-Fon Chang 2002/12/3

Experiment Type	Parameter Set	Pulse Program	Notes	Page
1D 1H				
one pulse 90deg	std_BBO_1D_1H_ZG	zg		1
one pulse 30deg	std_ BBO_1D_1H_ZG30	zg30		2
H2O suppression	std_ BBO_1D_1H_ZGPR	zgpr		3
homo-decoupling	std_ BBO_1D_1H_ZGHD	zghd		4
2D 1H				
COSY 45 deg	std_ BBO_2D_COSY-45	cosyqf45		5
COSY 90 deg	std_ BBO_2D_COSY-90	cosyqf90		6
TOCSY (mlev17)	std_ BBO_2D_TOCSY-MLEV	mlevph		7
NOESY	std_ BBO_2D_NOESY	noesyph		8
J-resolved	std_ BBO_2D_J-resolved	jresqf		9
1D 13C				
one pulse any deg	std_BBO_1D_13C_ZG0DC	zg0dc		10
one pulse 30deg	std_ BBO_1D_13C_ZGPG30	zgpg30		11
dept90	std_ BBO_1D_13C_DEPT90	dept90		12
dept135	std_ BBO_1D_13C_DEPT135	dept135		13
2D 1H-13C				
1H-13C HMBC	std_BBO_2D_13C_HMBC	inv4lplrndqf		14
1H-13C HMQC	std_BBO_2D_13C_HMQC	inv4ph		15
1D 11B				
one pulse	std_BBO_1D_11B_ZG	zg		16
with 1H decoupled	std_ BBO_1D_11B_ZGPG	zgpg		17
1D 15N				
one pulse	std_BBO_1D_15N_ZG	zg		18
with 1H decoupled	std_ BBO_1D_15N_ZGIG	zgig		19
1D 31P				
one pulse 30 deg	std_BBO_1D_31P_ZG30	zg30		20
with 1H decoupled	std_ BBO_1D_31P_ZGPG30	zgpg30		21

STANDARD EXPERIMENT LIST for Biomoleclues (I)

by Chi-Fon Chang 2002/12/3

Experiment Type	Parameter Set	Pulse Program	Notes	Page
1D 1H				
one pulse	std_1D_1H_ZG	zg		1
H2O suppression	std_1D_1H_ZGPR	zgpr		2
H2O suppression	std_1D_1H_ZGGPWG	zggpwg		3
jump return	std_1D_1H_P11	p11		4
p1331	std_1D_1H_P1331	p1331		5
p3919	std_1D_1H_P3919	p3919gp		6
2D 1H				
DQF-COSY	std_2D_COSY-DQF_3919	cosydfgpph19	*	7
DQF-COSY	std_2D_COSY-DQF_ET	cosydfetgp.2		8
Clean TOCSY	std_2D_TOCSY-CL	clmlevgpph19		9
TOCSY (mlev17)	std_2D_TOCSY-MLEV	mlevgpph19		10
TOCSY (dipsi2)	std_2D_TOCSY-DIPSI_ST	dipsi2gpph19		11
TOCSY (dipsi2)	std_2D_TOCSY-DIPSI_ET	dipsi2etgpsi19	*	12
NOESY	std_2D_NOESY_3919	noesygpph19		13
NOESY(flip back)	std_2D_NOESY-FP_3919	noesyfpgpph19		14
NOESY(flip back)	std_2D_NOESY-FP_WG	noesyfpgpphwg	*	15
2D 1H-X				
1H-15N HSQC	std_2D_15N_HSQC_ET	invietf3gpsi	*	16
1H-15N HSQC	std_2D_15N_HSQC_WG	invifpf3gpphwg		17
1H-15N HSQC	std_2D_15N_HSQC_FAST	fhsqcf3gpph		18
1H-15N T1	std_2D_15N_T1	invit1etf3gpsi	*	19
1H-15N T2	std_2D_15N_T2	invit2etf3gpsi	*	20
1H-15N NOE	std_2D_15N_NOE	invinoef3gpsi	*	21
1H-13C HSQC	std_2D_13C_HSQC_ET	invietgpsi	*	22

* If there are more than one version, * is somehow the more sensitive version.

STANDARD EXPERIMENT LIST Biomoleclues (II)

by Chi-Fon Chang 2002/12/3

Experiment	Parameter Set	Pulse Program	Notes	Page
3D Double Resonance				
3D 15N edit experiments				
TOCSY-HSQC (mlev)	std_3D_15N_TOCSYHSQC-MLEV	mleviietf3gp3d		23
TOCSY-HSQC (dipsi)	std_3D_15N_TOCSYHSQC-DIPSI	dipsiif3gpsi3d.2	*, 13C decp.	24
NOESY-HSQC	std_3D_15N_NOESYHSQC_ET	noesiietf3gp3d		25
NOESY-HSQC (sensitivity enhance)	std_3D_15N_NOESYHSQC_ETSI	noesiif3gpsi3d	*	26
NOESY-HSQC	std_3D_15N_NOESYHSQC_3919	noesiif3gp193d		27
HNHA (dihedral)	std_3D_15N_HNHA	hnhagp3d.2	*	28
HNHB (dihedral)	std_3D_15N_HNHB	hnhbgp3d		29
3D 13C edit experiments				
HCCH-COSY	std_3D_13C_HCCHCOSY	hcchcogp3d		30
HCCH-TOCSY	std_3D_13C_HCCHTOCSY	hcchdigp3d	*	31
NOESY-HSQC	std_3D_13C_NOESYHSQC_ET	noesiietgp3d.2	*	32
NOESY-HSQC (13C &15N together)	std_3D_13C_NOESYHSQC_SM	noesiigpsism3d		33
3D Triple Resonance				
Backbone Assignment				
HNCA	std_3D_HNCA	hncagp3d.2		34
HN(CO)CA	std_3D_HN(CO)CA	hncocagp3d.2		35
HNCO	std_3D_HNCO	hncogp3d.2	*	36
HN(CA)CO	std_3D_HN(CA)CO	hncacogp3d	*	37
HNCACB	std_3D_HNCACB	hncacbgp3d	*	38
HN(CO)CACB	std_3D_HN(CO)CACB	hncocacbgp3d	*	39
CBCANH	std_3D_CBCANH	cbcanhgp3d.2		40
CBCA(CO)NH	std_3D_CBCA(CO)NH	cbcaconhgp3d.2		41
Side Chain Assignment				
HBHANH	std_3D_HBHANH	hbhanhgs3d.2		42
HBHA(CO)NH	std_3D_HBHA(CO)NH	hbhaconhgs3d.2		43
H side chain	std_3D_HCCCONH_H	hccconhgs3d2		44
C side chain	std_3D_HCCCONH_C	hccconhgp3d3		45
C side chain	std_3D_CCCONH	cconhgs3d		46

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