

Overview of CryoProbe Operation in Core Facility

Chi-Fon Chang

02/27/2004

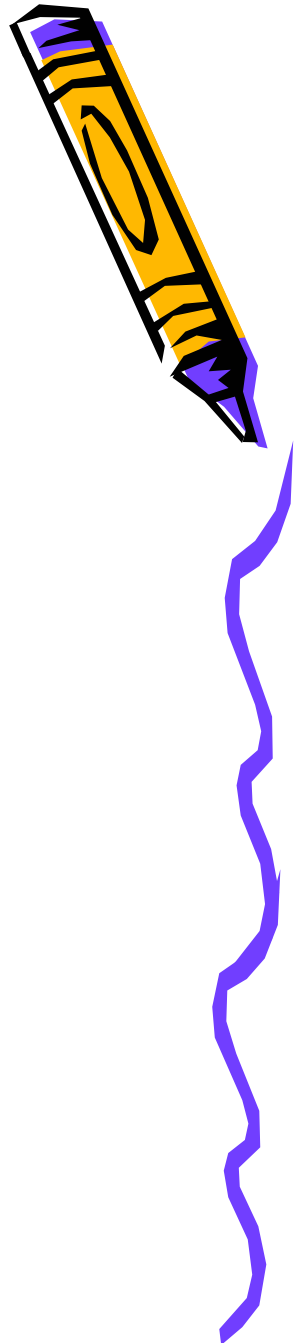


Using CryoProbe

The most important thing is

Be Careful....

請專心!!
請小心!!



Current Status of NMR LAB

Overview of NMR Facility

AV500 in IBMS : 5mm TXI-Z ($^1\text{H}/^{15}\text{N}/^{13}\text{C}$, with Z gradient → stand by)

5mm TXI-Z CryoProbe

AV600 in IBMS: 5mm TCI-Z CryoProbe

AV600 in CHEM: 5mm BBO & TXI-Z ($^1\text{H}/^{15}\text{N}/^{13}\text{C}$, with Z gradient)

DRX600 in IBMS: 5mm QXI-Z ($^1\text{H}/^{15}\text{N}/^{13}\text{C}/^{31}\text{P}$ with Z gradient) ,

5mm TXI-XYZ ($^1\text{H}/^{15}\text{N}/^{13}\text{C}$, with XYZ gradient) and others

5mm : ^1H , $^1\text{H}/^{19}\text{F}$, BBO, TXI($^1\text{H}/^{15}\text{N}/^{13}\text{C}$) , TXI-Z ($^1\text{H}/^{13}\text{C}/^{31}\text{P}$)

8mm : TXI ($^1\text{H}/^{13}\text{C}/^{15}\text{N}$) 8mm with Z gradient

10mm: ^1H , $^1\text{H}/^{19}\text{F}$, BBO

AV800 in IBMS (not available yet) : 5mm TXI-Z & CryoProbe



CryoProbe Installation



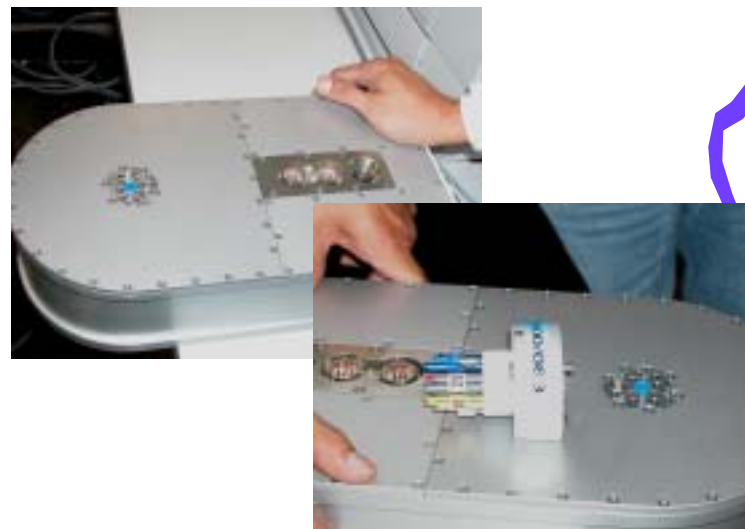
B2 NMR Room



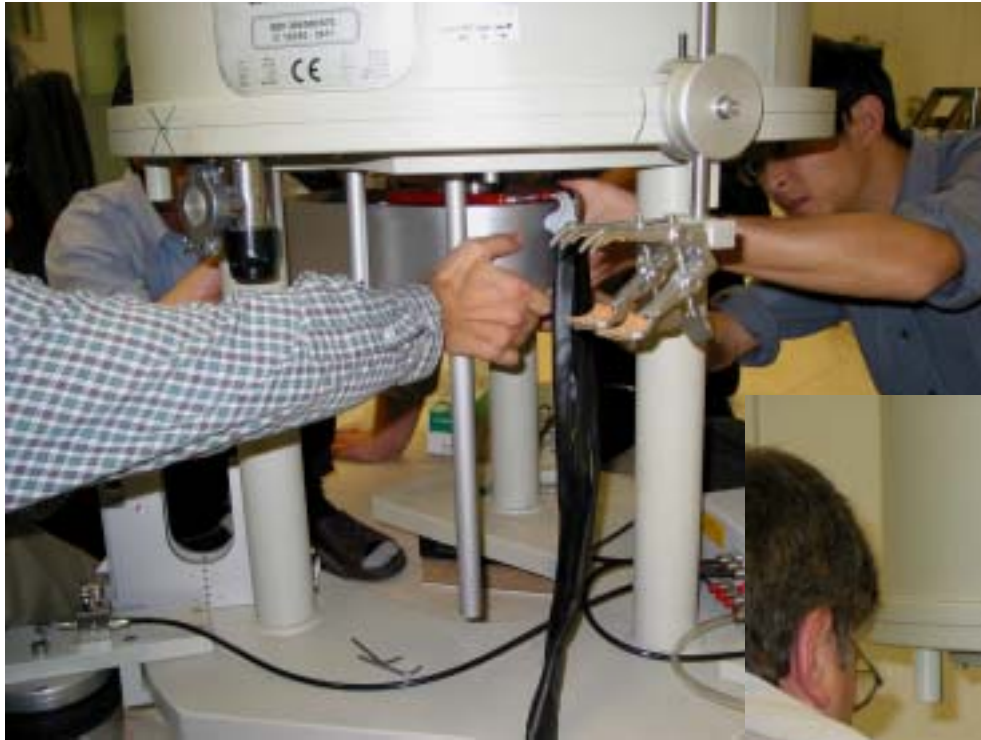
B1 Room



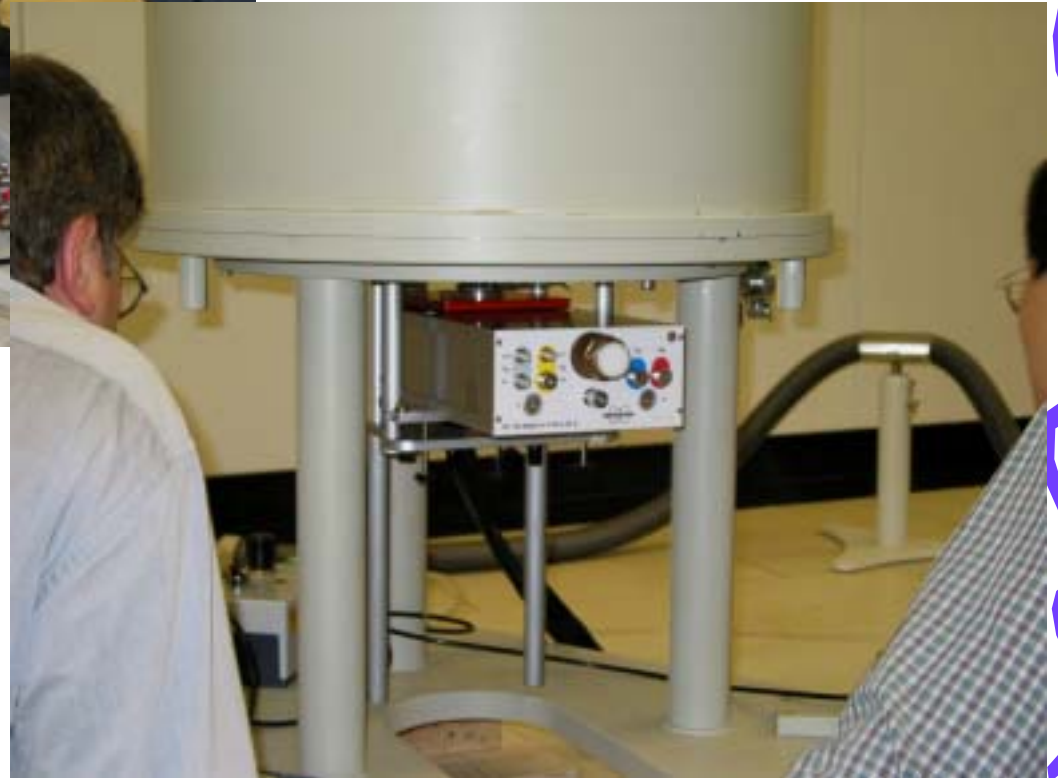
Probe 底部



好重喔! 至少需要二人一起裝……



終於裝好探頭了!!



500MHz CroyProbe



600MHz CroyProbe

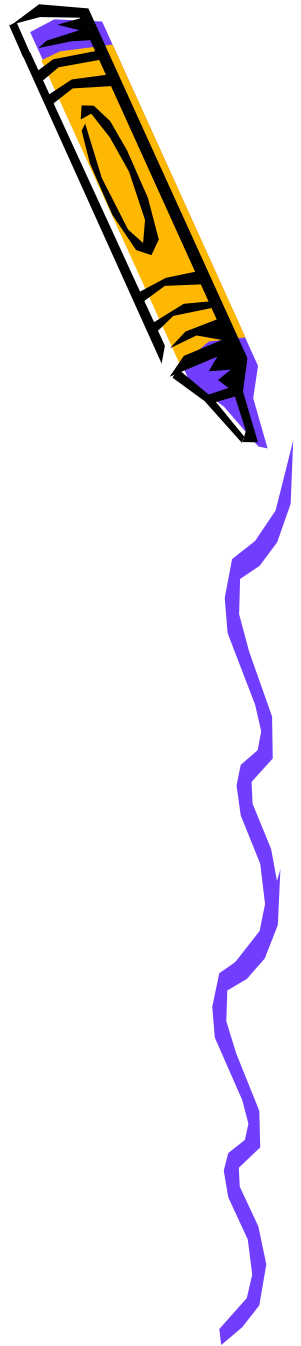


Someone is watching

Be Careful.....

請專心!!

請小心!!

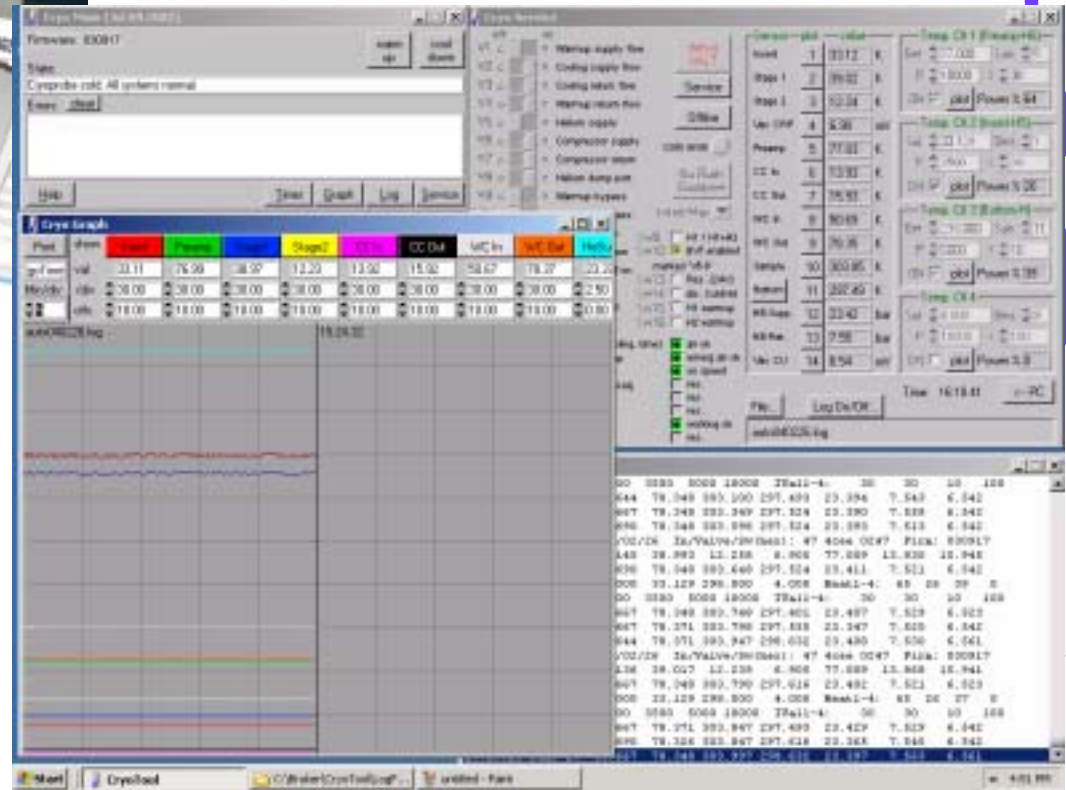


Cryo Tool

Computer Control

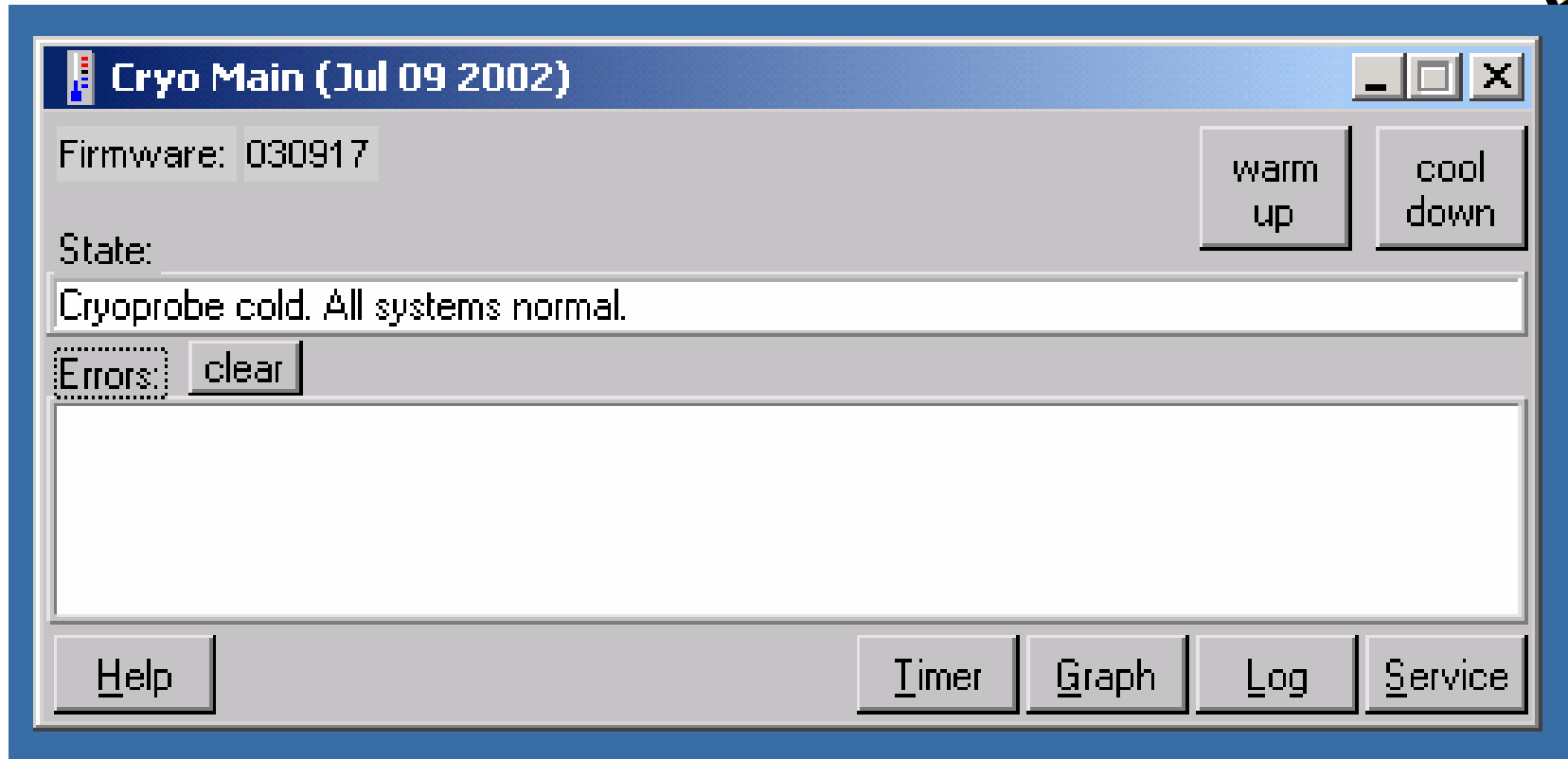


24 hours monitor

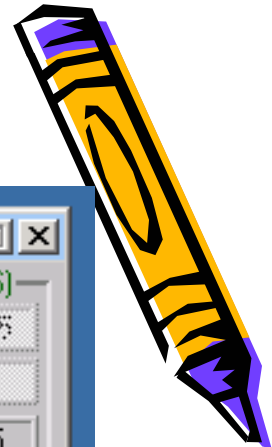


In case of error message

Stop your experiment immediately !!



Cryo Service



Cryo Service

off	on	
V1	<input type="checkbox"/>	Warmup supply flow
V2	<input type="checkbox"/>	Cooling supply flow
V3	<input type="checkbox"/>	Cooling return flow
V4	<input type="checkbox"/>	Warmup return flow
V5	<input type="checkbox"/>	Helium supply
V6	<input type="checkbox"/>	Compressor supply
V7	<input type="checkbox"/>	Compressor return
V8	<input type="checkbox"/>	Helium dump port
V9	<input type="checkbox"/>	Warmup bypass
V10	<input type="checkbox"/>	Cooldown bypass
V11	<input type="checkbox"/>	Probe Vacuum
V12	<input type="checkbox"/>	Coldbox Vacuum
V13	<input type="checkbox"/>	Emerg. Lift / T-on
V14	<input type="checkbox"/>	Emerg. T-off
V15	<input type="checkbox"/>	CRP vac. joint
V16	<input type="checkbox"/>	res.

Sw1	<input checked="" type="checkbox"/>	Support (air, water cooling, time)
Sw2	<input checked="" type="checkbox"/>	Vacuum roughing pump
Sw3	<input checked="" type="checkbox"/>	Vacuum turbo pump
Sw4	<input type="checkbox"/>	Warmup heaters (H3+H4)
Sw5	<input type="checkbox"/>	res. (AC socket)
Sw6	<input type="checkbox"/>	res. (AC socket)
Sw7	<input checked="" type="checkbox"/>	Compressor
Sw8	<input type="checkbox"/>	res. (free contact)

debug HALT

Service

Offline

com error

No Flush
Cooldown

Insert Max

Sensor	plot	value	
Insert	<input type="checkbox"/>	33.11	K
Stage 1	<input type="checkbox"/>	38.97	K
Stage 2	<input type="checkbox"/>	12.23	K
Vac CRP	<input type="checkbox"/>	6.98	mV
Preamp	<input type="checkbox"/>	76.99	K
CC In	<input type="checkbox"/>	13.90	K
CC Out	<input type="checkbox"/>	15.93	K
WC In	<input type="checkbox"/>	50.67	K
WC Out	<input type="checkbox"/>	78.37	K
Sample	<input type="checkbox"/>	304.00	K
Bottom	<input type="checkbox"/>	298.69	K
HE-Supp.	<input type="checkbox"/>	23.35	bar
HE-Ret.	<input type="checkbox"/>	7.51	bar
Vac CU	<input type="checkbox"/>	6.56	mV

Temp. Ctl 1 (Preamp-H6)
 Set Sen.
 P I
 ON Power %

Temp. Ctl 2 (Insert-H5)
 Set Sen.
 P I
 ON Power %

Temp. Ctl 3 (Bottom-H)
 Set Sen.
 P I
 ON Power %

Temp. Ctl 4
 Set Sen.
 P I
 ON Power %

Time: 16:17.03

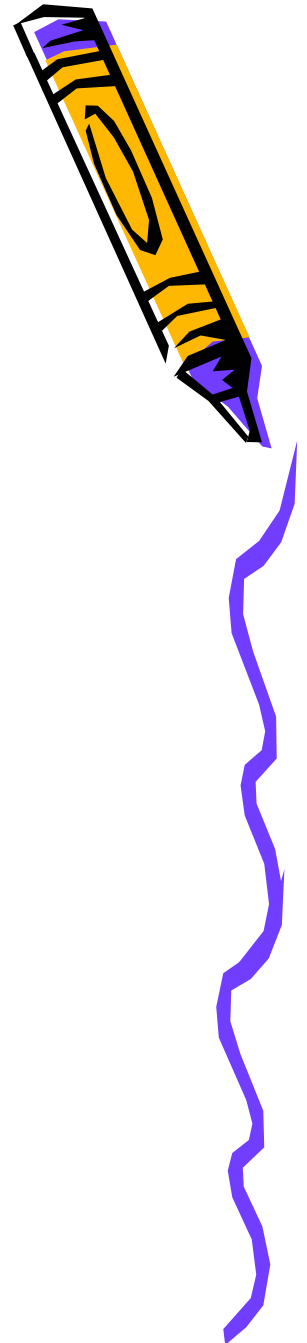
File...

auto040226.log

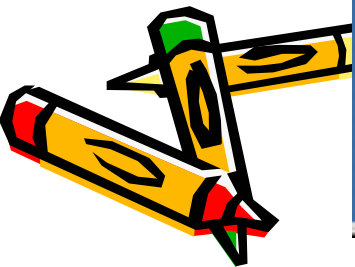
Cryo Graph



Cryo Log

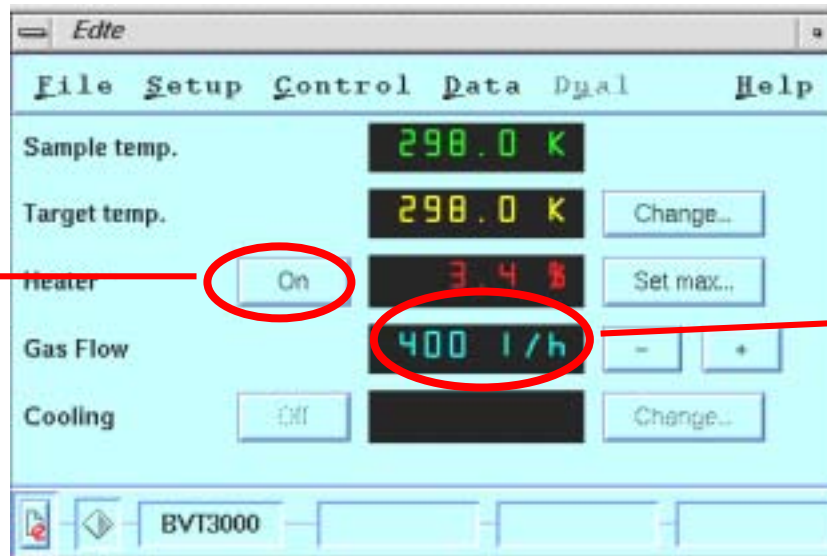


Cryo Log												
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	65	23	24	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
15:45.18	Sen8-14	50.529	78.371	300.913	297.493	23.369	7.520	6.542				
15:49.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	33.141	38.945	12.225	6.981	76.987	13.691	15.907				
	Sen8-14	50.529	78.371	301.062	298.570	23.393	7.502	6.561				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	65	23	25	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
15:51.18	Sen8-14	50.529	78.348	300.913	297.462	23.436	7.523	6.523				
15:54.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	33.124	38.969	12.208	6.905	77.009	13.725	15.893				
	Sen8-14	50.529	78.371	300.913	297.401	23.383	7.513	6.523				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	64	23	40	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
15:55.18	Sen8-14	50.529	78.371	301.062	298.570	23.384	7.539	6.561				
15:56.18	Sen8-14	50.529	78.348	300.913	297.462	23.390	7.534	6.542				
15:57.18	Sen8-14	50.552	78.371	301.062	298.570	23.407	7.572	6.561				
15:58.18	Sen8-14	50.529	78.371	300.913	297.524	23.373	7.516	6.523				
15:59.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	32.995	39.017	12.165	6.905	77.009	13.799	15.798				
	Sen8-14	50.552	78.348	300.913	297.555	23.411	7.521	6.542				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	64	25	38	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
16:00.18	Sen8-14	50.552	78.371	301.062	298.663	23.396	7.570	6.561				
16:01.18	Sen8-14	50.575	78.348	300.913	297.585	23.360	7.549	6.542				
16:02.18	Sen8-14	50.575	78.371	301.062	298.663	23.398	7.529	6.561				
16:04.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	33.113	38.945	12.237	6.981	76.964	13.873	15.911				
	Sen8-14	50.644	78.348	303.399	298.601	23.422	7.565	6.561				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	64	26	39	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
16:05.18	Sen8-14	50.644	78.348	303.100	297.493	23.394	7.543	6.542				
16:06.18	Sen8-14	50.667	78.348	303.349	297.524	23.390	7.559	6.542				
16:08.18	Sen8-14	50.690	78.348	303.598	297.524	23.393	7.513	6.542				
16:09.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	33.145	38.993	12.258	6.905	77.009	13.830	15.945				
	Sen8-14	50.690	78.348	303.648	297.524	23.411	7.521	6.542				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	65	26	39	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
16:11.18	Sen8-14	50.667	78.348	303.748	297.401	23.407	7.529	6.523				
16:12.18	Sen8-14	50.667	78.371	303.798	297.555	23.347	7.520	6.542				
16:13.18	Sen8-14	50.644	78.371	303.947	298.632	23.408	7.530	6.561				
16:14.18	REPORT:	2004/02/26	In/Valve/Sv(hex):	47 4cee 0247	Firm:	030917						
	Sen1-7:	33.136	39.017	12.239	6.905	77.009	13.868	15.941				
	Sen8-14	50.667	78.348	303.798	297.616	23.402	7.521	6.523				
	Set1-4:	77.000	33.129	298.000	4.000	Heat1-4:	65	26	37	0		
	FVall-4	10000	3500	5000	10000	IVall-4:	30	30	10	100		
16:15.18	Sen8-14	50.667	78.371	303.847	297.493	23.429	7.529	6.542				



Simple Outline

- Hardware Checking
 - Make sure the system is normal
- Temperature Setting



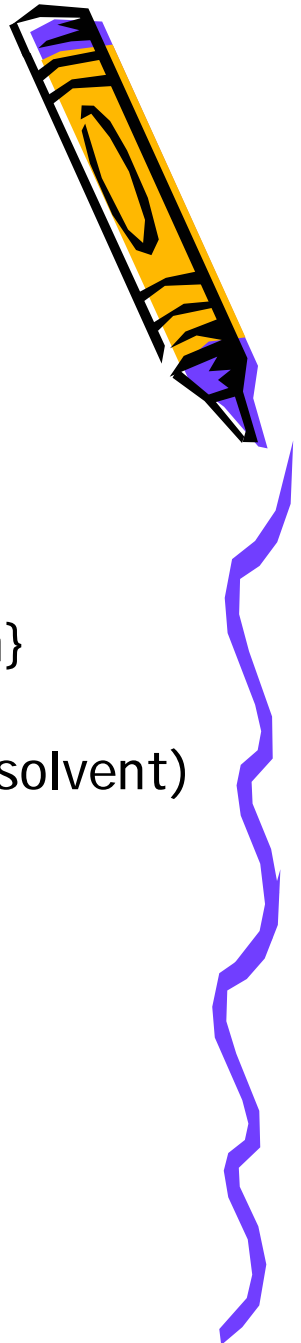
**Always
ON !!!!**

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

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



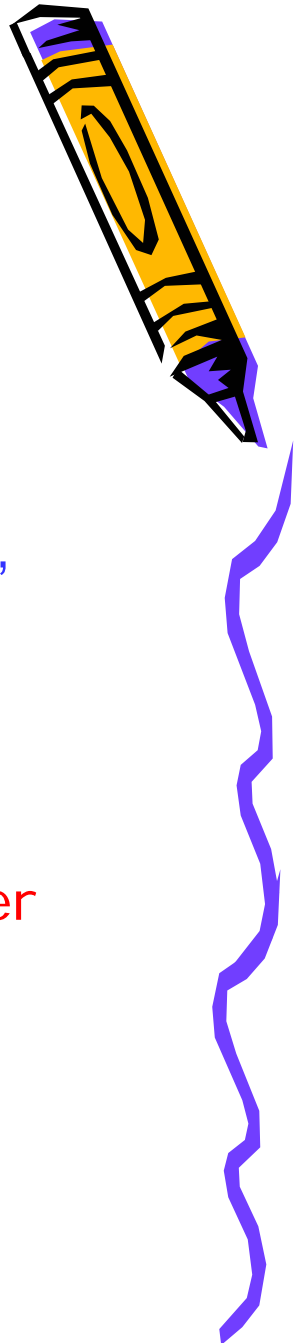
- Check spinner (sample holder with **yellow line**) position
- Lift gas (make sure there's lift gas) {click botton}
- Load sample
- Lift off (now the sample should go down **slowly**) {click botton}
- Lock the solvent ("lock" {command line}, and click on your D-solvent)
- Wobble (**Don't need to change cable anymore!!!**)



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
(d1+aq has to be longer than 1 sec)

Please read the Pulse and Power Recommendation from Bruker



- How to optimize condition?

- 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

p1 (high power, ex: 0dB), **p1** (90o pulse at p1)

p28 (trim pulse, ex:1m)

Others

cnst4 (J H-N , ex: 90Hz)

d24 (1/4JH-N)

o1 (for 1H)

o3 (for 15N)

1 sw, 1td (for F1 dimension, N)

2 sw, 2 td (for F2 dimension, H)

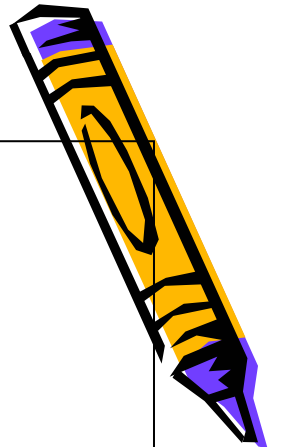
d1

ns(=1*n)

ds(>=16)

rg

Users need to adjust parameters in “red” (meaning of the parameter in “green”)



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

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

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

Experiment (3)

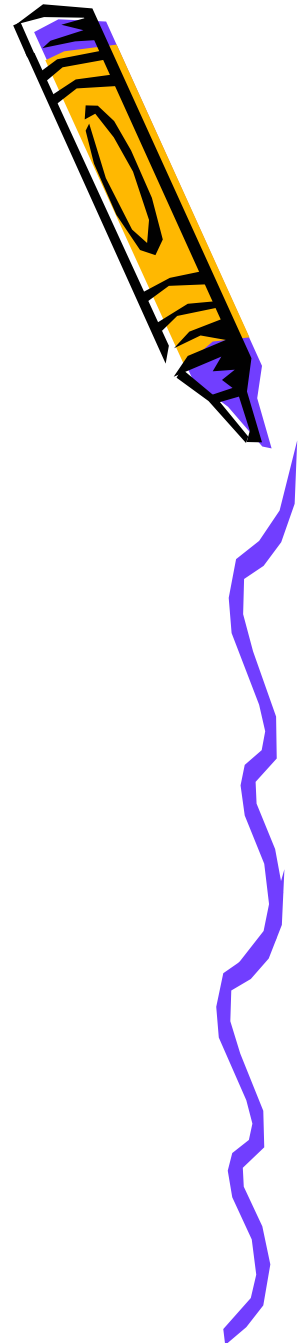
Experiment Name: **2D 15N-1H CRINEPT**

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

Standard Parameter Set: **CRP_2D_15N_CRINEPT**

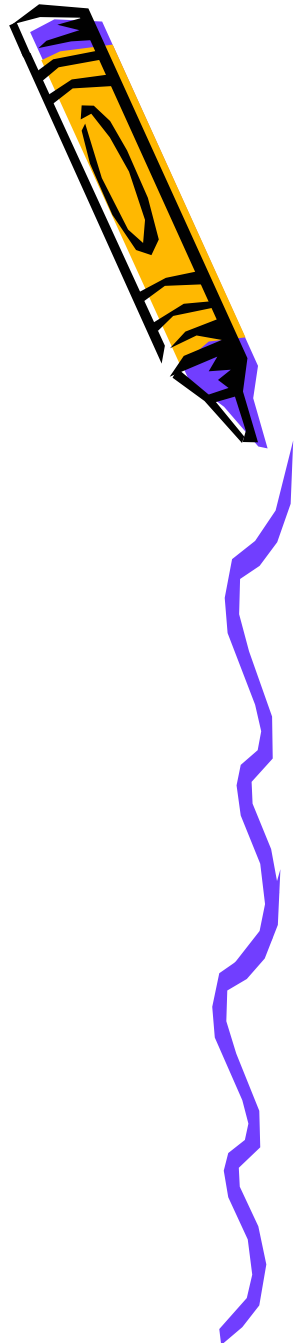
Pulse Program: crineptgpph

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



Are you ready?

Let's go!!



2004 NMR Users Training Course Lab Session



指導人員	游春愛	方珮如	張七鳳
儀器名稱	AV600	AV500	DRX600
2:00-3:30	林芳民、黃依川 駱啓仁、唐子鈞 林達顯、朱峰儀	黃偵雅、王誌慶 方羿喬、林書玄 陳昱任、盧弘捷	葉意茹、陳彥善 侯珈禎、何怡萱 潘韻如
3:30-5:00	白明道、吳昆峰 樊書傑、吳俊明 曹雅萍、魏淑怡	劉育男、薛光隆 林素卿、徐駿森 何孟儒、黃愈志	邱垂霖、黃保宜 鄭錫聰、黃一哲 林谷峰

