

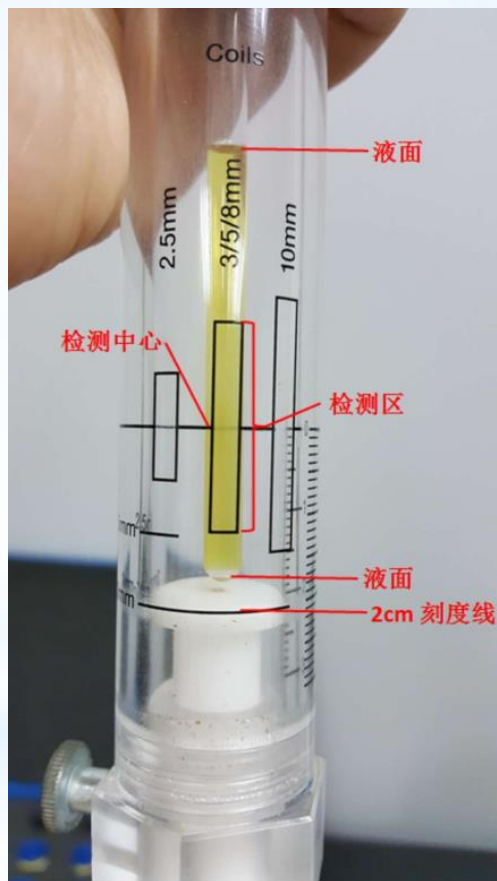
核磁共振实验技术（小分子）

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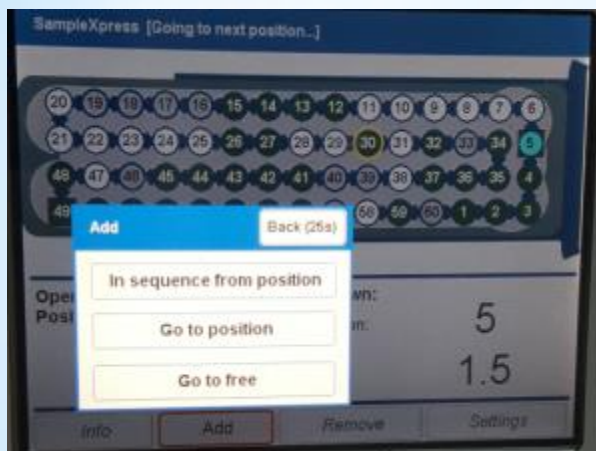
2018.10.09

400MHz液体核磁共振谱仪操作流程

- (1) 将身上的手机、卡、钥匙等放到桌子收纳箱内；
- (2) 将核磁管依次放入转子内，用量规调整每一个样品的高度，如下图所示：



(3) 放样，如图所示



Add→In sequence from position



选择图标为灰色的序号放样，每次放样结束，等指示灯灭再放下一个样品

Icon实验设置界面简单介绍

The screenshot displays the Icon software interface, which is used for managing and scheduling experiments. The interface is divided into two main sections: the Experiment Queue and the Pending Experiments section.

Experiment Queue: This section lists the experiments currently in the queue. The table below shows the data for the experiments listed in the queue.

Exp. #	Type	Status	Disk	Name	No.	Solvent	Experiment	Pr.	Par.	Total/Deg.	Time	User	Start Time
24	2	Queued	D:\NMR\400\G2T3	LY-42	10	CDCl3	chiral N-PROTON	1			00:00:00	new	11:18 Fri Nov 02 2017
25	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:04:00	new	11:18 Fri Nov 02 2017
26	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
27	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
28	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
29	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
30	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
31	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
32	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
33	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
34	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017
35	2	Queued	D:\NMR\400\G2T3	LY-42	11	CDCl3	chiral N-CL3CPD	1			00:00:00	new	12:14 Fri Nov 02 2017

Pending Experiments: This section lists the experiments that are pending or have been completed. The table below shows the data for the experiments listed in the pending section.

#	Date	Holder	Name	No.	Experiment	Load	ATM	Lock	Shut	Acq	Proc	User	Disk	Total/Deg	Remarks
132	2017-11-02 10:46:06	26	130217-26-5	10	PROTON	✓	✓	✓	✓	✓	✓	new	D:\NMR\400\G2T3\data	00:00:00	
133	2017-11-02 12:09:21	30	130217-30-5	10	PROTON	✓	✓	✓	✓	✓	✓	new	D:\NMR\400\G2T3\data	00:00:00	
134	2017-11-02 12:25:57:53	39	W04466	10	PROTON	✓	✓	✓	✓	✓	✓	new	D:\NMR\400\G2T3\data	00:00:00	
135	2017-11-02 13:37:21	18	W04443-2	10	CL3CPD	✓	✓	✓	✓	✓	✓	new	D:\NMR\400\G2T3\data	00:00:00	
136	2017-11-02 15:24:56	17	ZIP-44-20C	11	CL3CPD	✓	✓	✓	✓	✓	✓	new	D:\NMR\400\G2T3\data	00:00:00	

Annotations:

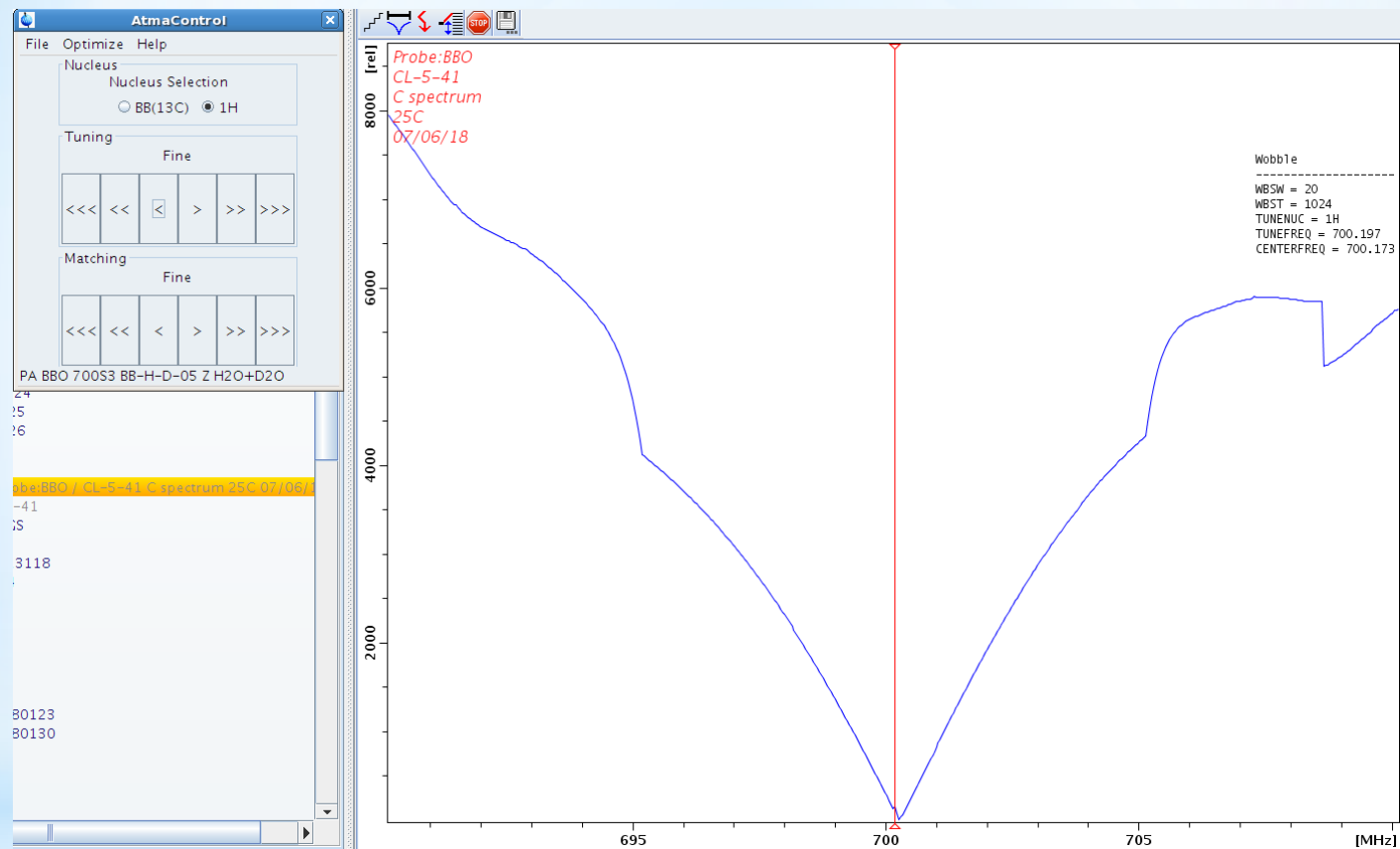
- 文件夹名字 (Folder Name): Points to the 'Disk' column in the Experiment Queue table.
- 子文件夹 (Sub-folder): Points to the 'Name' column in the Experiment Queue table.
- 血液试剂 (Blood Reagent): Points to the 'Solvent' column in the Experiment Queue table.
- 编辑实验 (Edit Experiment): Points to the 'Experiment' column in the Experiment Queue table.
- 序号 (Serial Number): Points to the 'No.' column in the Experiment Queue table.
- 实验序列选择 (Experiment Sequence Selection): Points to the 'Experiment' column in the Experiment Queue table.
- 关键参数修改 (Key Parameter Modification): Points to the 'Pr.' column in the Experiment Queue table.
- 开始时间设置 (Start Time Setting): Points to the 'Start Time' column in the Experiment Queue table.
- 提交实验 (Submit Experiment): Points to the 'Submit' button in the Pending Experiments section.
- 取消已排队的实验 (Cancel Queued Experiment): Points to the 'Cancel' button in the Pending Experiments section.
- 删除已完成实验 (Delete Completed Experiment): Points to the 'Delete' button in the Pending Experiments section.

锁场 (lock) :

Solvents table	
Δ Solvent	Description
Acetic	acetic acid-d4
Acetone	acetone-d6
C6D6	benzene-d6
CD2Cl2	dichloromethane-d2
CD3CN	acetonitrile-d3
CD3CN_SPE	LC-SPE Solvent (Acetonitrile)
CD3OD_SPE	LC-SPE Solvent (Methanol-d4)
CDCl3	chloroform-d
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)
D2O	deuteriumoxide
D2O_salt	deuteriumoxide with salt
Dioxane	dioxane-d8
DMF	N,N-dimethylformamide-d7
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
H2O+D2O_new	test for shifted water
H2O+D2O_salt	90%H2O and 10%D2O with salt
HDMSO	90%DMSO and 10%DMSO-d6
Juice	fruit juice
MeOD	methanol-d4
Plasma	blood plasma
Pyr	pyridine-d6
SDS1	sds1-d25
T_H2O+D2O+Me4NCl	(CD3)4NCl in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+NaAc	sodium acetate in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+Pivalate	pivalate-d9 in 90% H2O and 10% D2O, for NMR thermometer
T_MeOD	methanol-d4, for NMR thermometer
TFE	trifluoroethanol-d3
THF	tetrahydrofuran-d8
Tol	toluene-d8
Urine	urine

OKCancel

调谐 (atmm) :



匀场(topshim):

S TopShim [minimize] [maximize] [close]

Shim Report Service

SHIM

Dimension ☒ 1D ☐ 3D

Optimisation solvent's default

Optimise for 1H

Use Z6 ☐

TUNE

Before off

After off

Only ☐

PARAMETERS

☐

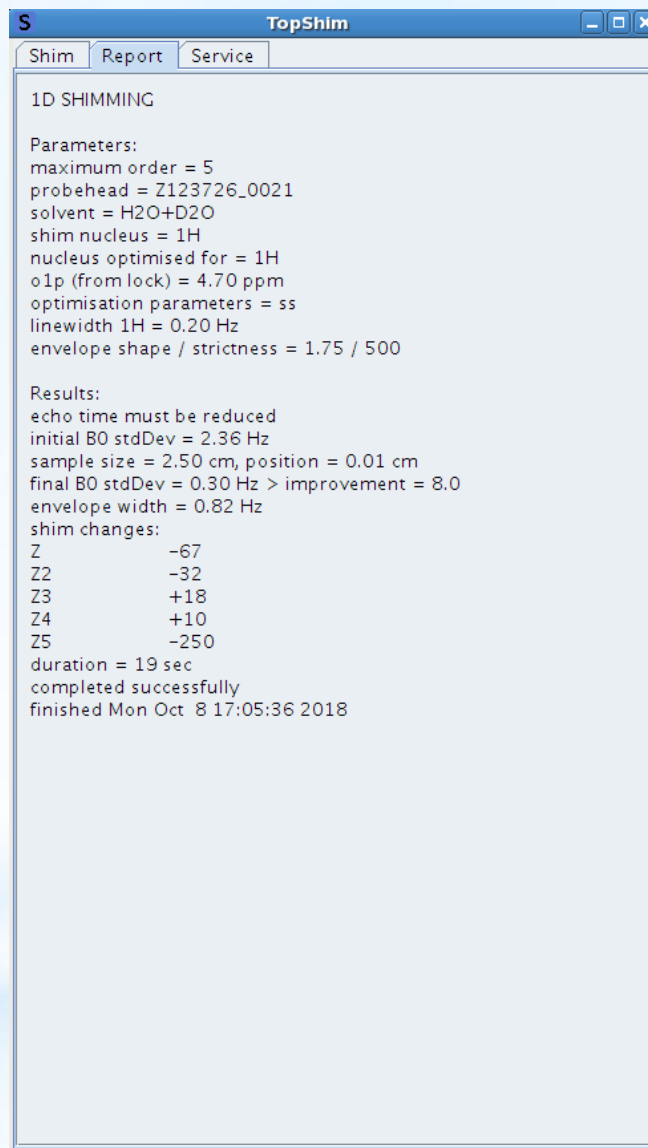
STATUS

running ...
solvent = H2O+D2O
target 1
start acquisition

CONTROL

Start Stop Help Close

匀场(topshim):



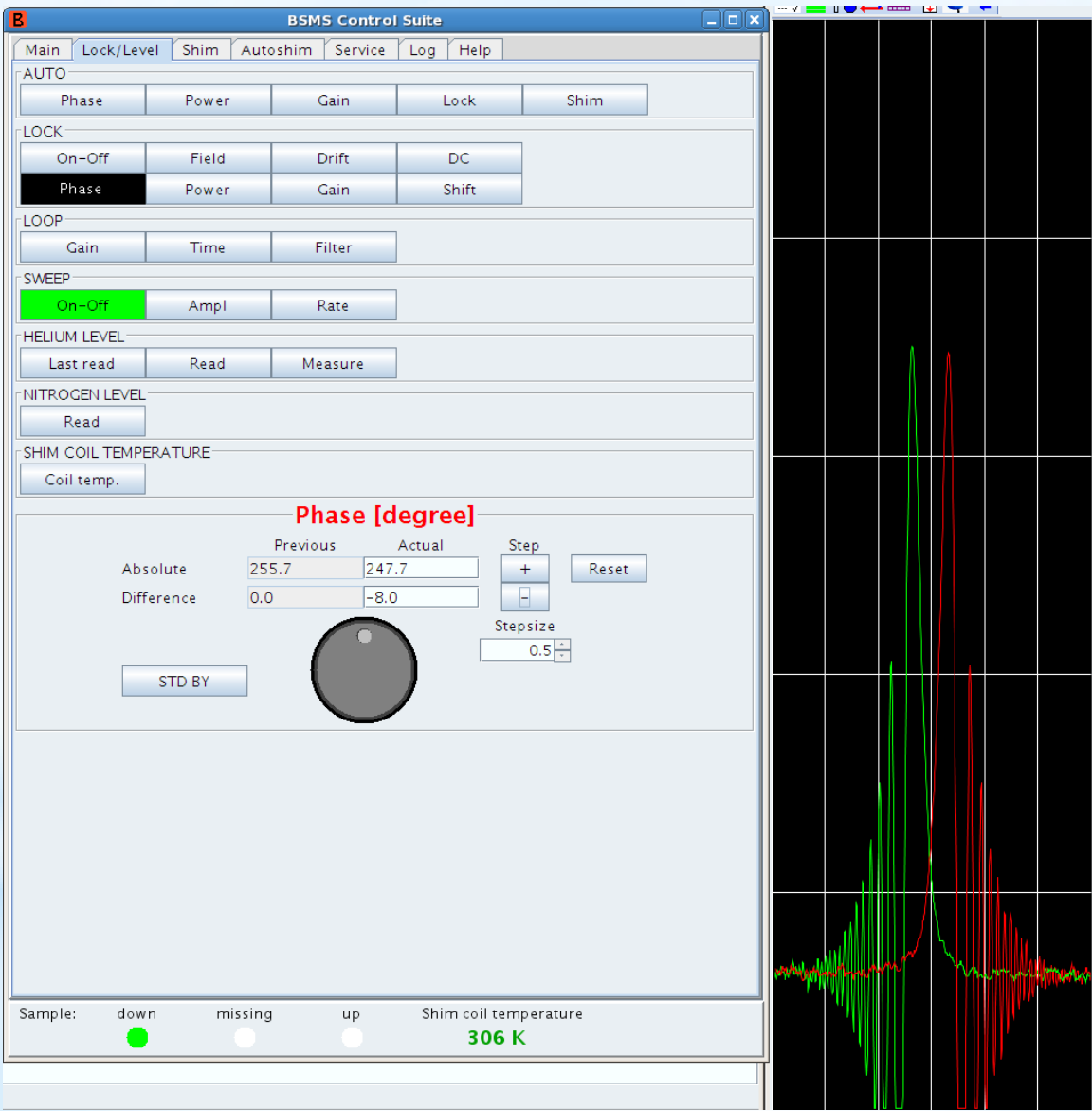
The screenshot shows a window titled 'TopShim' with three tabs: 'Shim', 'Report', and 'Service'. The 'Shim' tab is active, displaying the following text:

```
1D SHIMMING

Parameters:
maximum order = 5
probehead = Z123726_0021
solvent = H2O+D2O
shim nucleus = 1H
nucleus optimised for = 1H
o1p (from lock) = 4.70 ppm
optimisation parameters = ss
linewidth 1H = 0.20 Hz
envelope shape / strictness = 1.75 / 500

Results:
echo time must be reduced
initial B0 stdDev = 2.36 Hz
sample size = 2.50 cm, position = 0.01 cm
final B0 stdDev = 0.30 Hz > improvement = 8.0
envelope width = 0.82 Hz
shim changes:
Z          -67
Z2         -32
Z3          +18
Z4          +10
Z5        -250
duration = 19 sec
completed successfully
finished Mon Oct 8 17:05:36 2018
```

lock level:



一、一维实验

1.1 氢谱

1.2 碳谱

1.3 磷谱

1.4 DEPT实验

二、二维实验

2.1 异核相关谱

2.2 同核相关谱

2.3 NOE类相关谱

1.1 氢谱

zg30:

General

PULPROG

zg30

...

E

TD

65536

SWH [Hz, ppm]

14097.74

20.1346

AQ [sec]

2.3243434

RG

1

DW [μsec]

35.467

DE [μsec]

10.00

D1 [sec]

1.70000005

DS

4

NS

16

TD0

1

Pulse program for acquisition

Time domain size

Sweep width

Acquisition time

Receiver gain

Dwell time

Pre-scan-delay

Relaxation delay; 1-5 * T1

Number of dummy scans

1 * n, total number of scans: NS * TD0

Dimension of accumulation loop

Channel f1

SFO1 [MHz]

700.1743238

O1 [Hz, ppm]

4323.84

6.175

NUC1

1H

Edit...

P1 [μsec]

7.40

PLW1 [W, -dBW]

10

-10.00

Frequency of ch. 1

Frequency of ch. 1

Nucleus for channel 1

F1 channel - 90 degree high power pulse

F1 channel - power level for pulse (default)

1.1 氢谱

zg30:

```
;zg30
;avance-version (12/01/11)
;1D sequence
;using 30 degree flip angle
;
;$CLASS=HighRes
;$DIM=1D
;$TYPE=
;$SUBTYPE=
;$COMMENT=
;$RECOMMEND=y

#include <Avance.incl>

"acqt0=-p1*0.66/3.1416"

1 ze
2 30m
   d1
   p1*0.33 ph1
   go=2 ph31
   30m mc #0 to 2 F0(zd)
exit

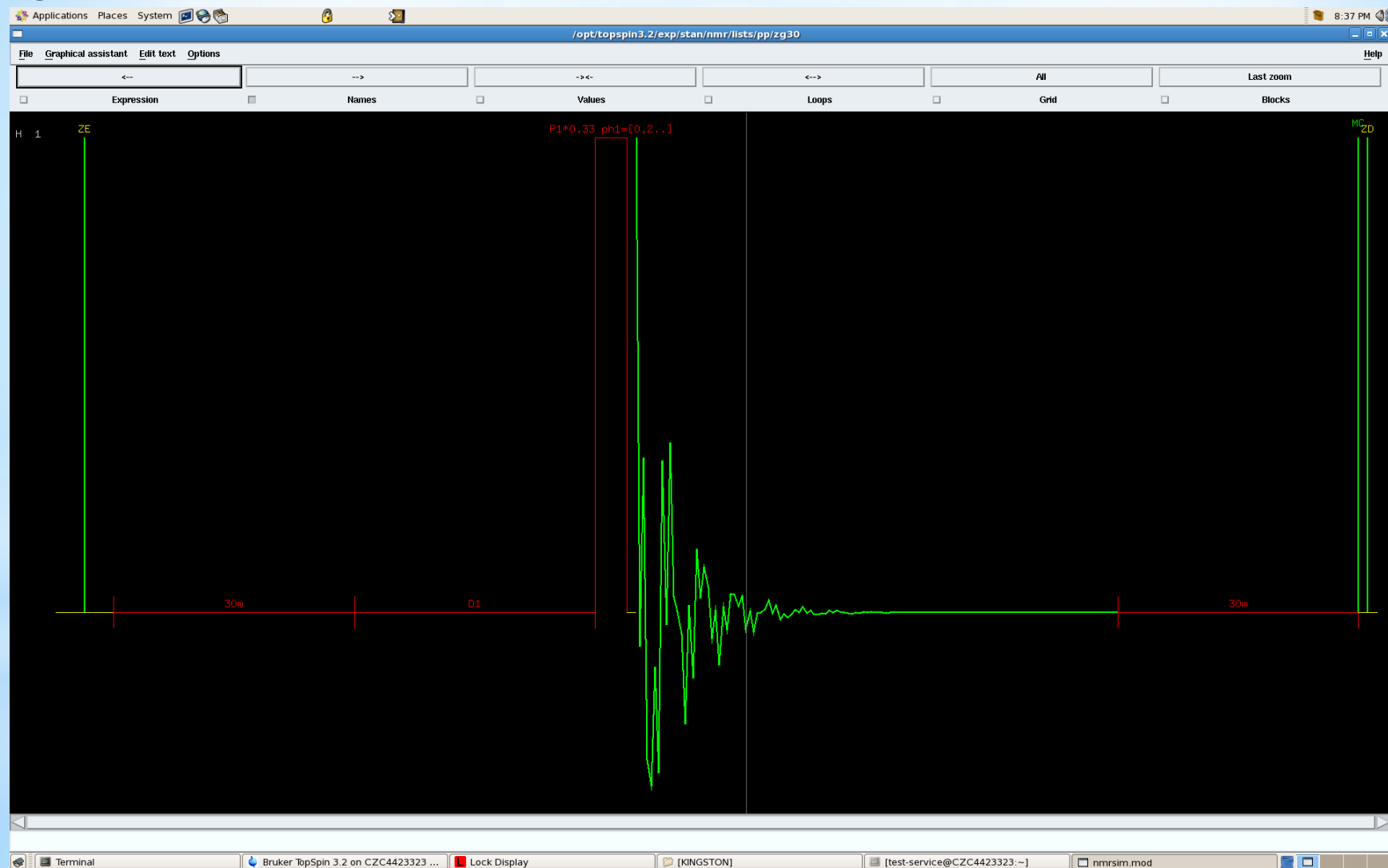
ph1=0 2 2 0 1 3 3 1
ph31=0 2 2 0 1 3 3 1

;p11 : f1 channel - power level for pulse (default)
;p1 : f1 channel - 90 degree high power pulse
;d1 : relaxation delay; 1-5 * T1
;ns: 1 * n, total number of scans: NS * TD0

;$Id: zg30,v 1.11.6.1 2012/01/31 17:56:41 ber Exp $
```

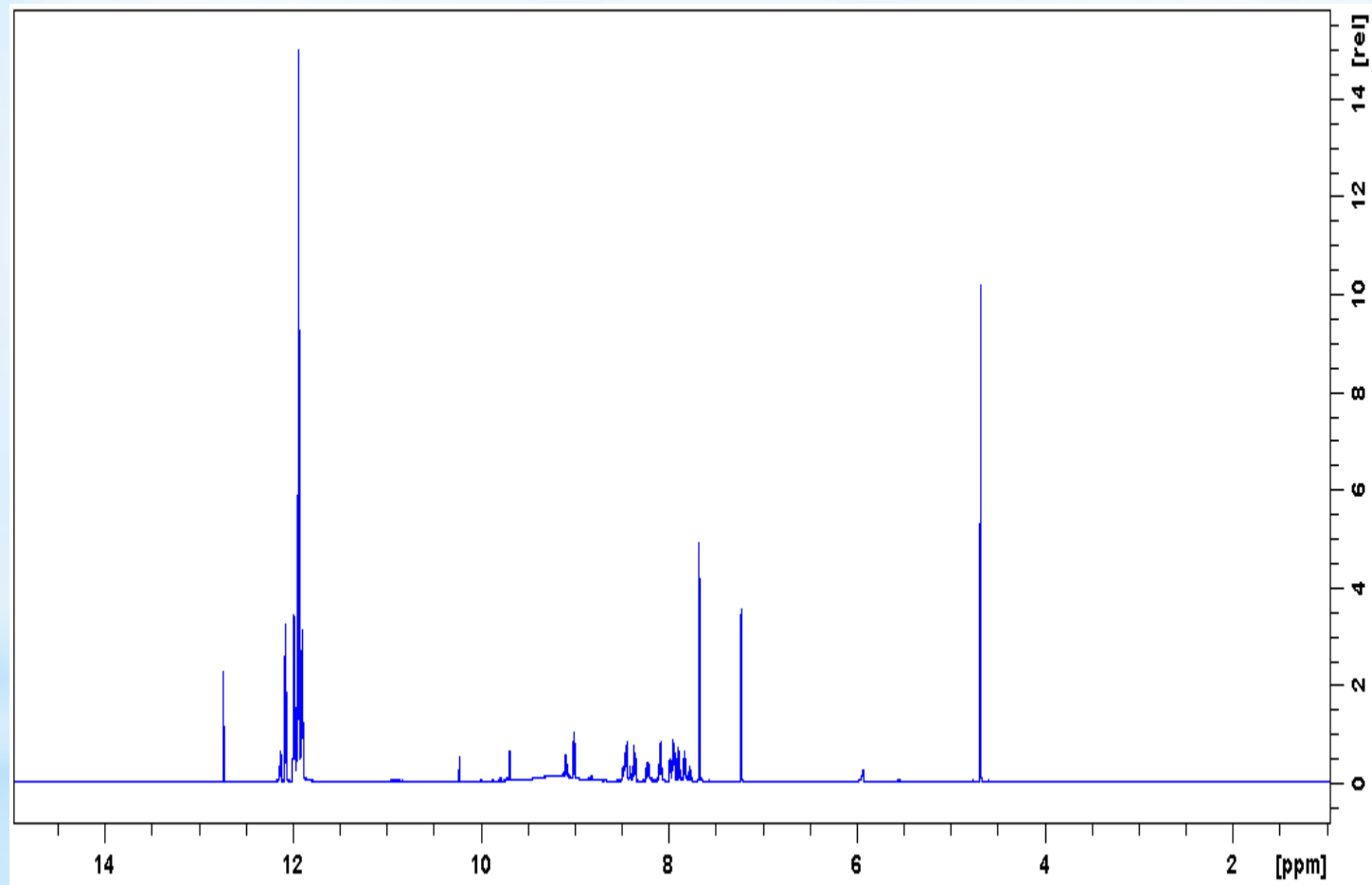
1.1 氢谱

zg30脉冲信息如下:



1.1 氢谱

zg30对应的谱图如下:



1.1 氢谱

压水峰的一维氢谱:

General

PULPROG

TD

SWH [Hz, ppm]

AQ [sec]

RG

DW [μsec]

DE [μsec]

D1 [sec]

d12 [sec]

D16 [sec]

DELTA1 [sec]

DS

NS

TD0

zgpgppewg

8192

14097.74

0.2905429

32

35.467

10.00

2.00000000

0.00002000

0.00020000

0.00102800

8

128

1

...

E

Pulse program for acquisition

Time domain size

Sweep width

Acquisition time

Receiver gain

Dwell time

Pre-scan-delay

Relaxation delay; 1-5 * T1

Delay for power switching [20 usec]

Delay for homospoil/gradient recovery

DELTA1=p11+d12+8u

4

8 * n, total number of scans: NS * TD0

Dimension of accumulation loop

Channel f1

SFO1 [MHz]

O1 [Hz, ppm]

NUC1

P1 [μsec]

p2 [μsec]

P11 [μsec]

PLW1 [W, -dBW]

SPNAM 1

SPOAL1

SPOFFS1 [Hz]

SPW1 [W, -dBW]

700.1737936

3793.60

1H

8.25

16.50

1000.00

10

Squa100.1000

0.500

0

0.00068063

Edit...

...

E

Frequency of ch. 1

Frequency of ch. 1

Nucleus for channel 1

F1 channel - 90 degree high power pulse

F1 channel - 180 degree high power pulse

F1 channel - 90 degree shaped pulse

F1 channel - power level for pulse (default)

File name for SP1

Phase alignment of freq. offset in SP1

Offset frequency for SP1

F1 channel - shaped pulse 90 degree

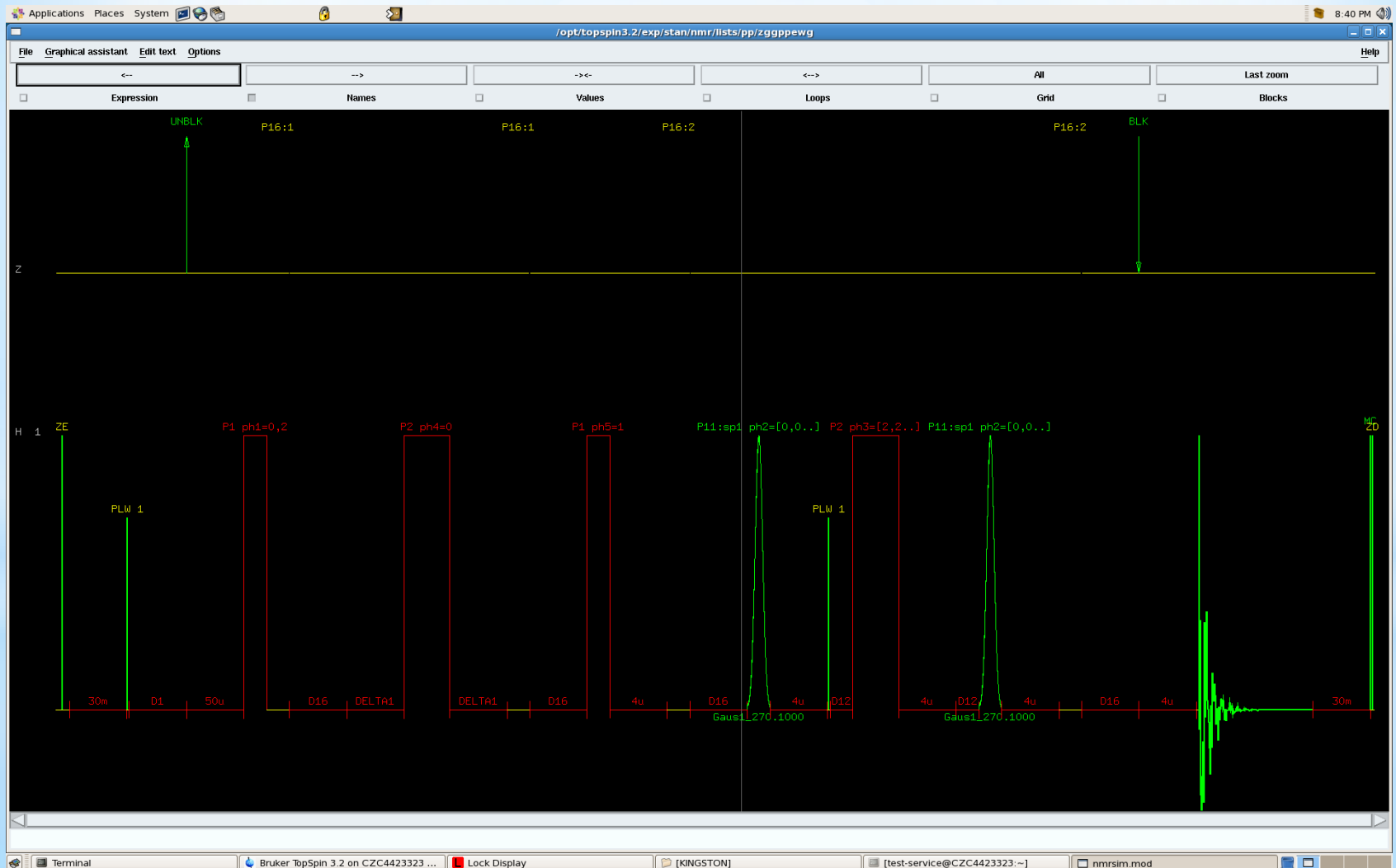
1.1 氢谱

压水峰的一维氢谱：

SPW1 [W, -dBW]	0.00060065	51.67	F1 channel - shaped pulse 90 degree
Gradient channel			
CPNAM 1	SMSQ10.100	... E	SMSQ10.100
CPNAM 2	SMSQ10.100	... E	SMSQ10.100
GPZ1 [%]	11.00		11%
GPZ2 [%]	31.00		31%
P16 [μsec]	1000.00		Homospoil/gradient pulse

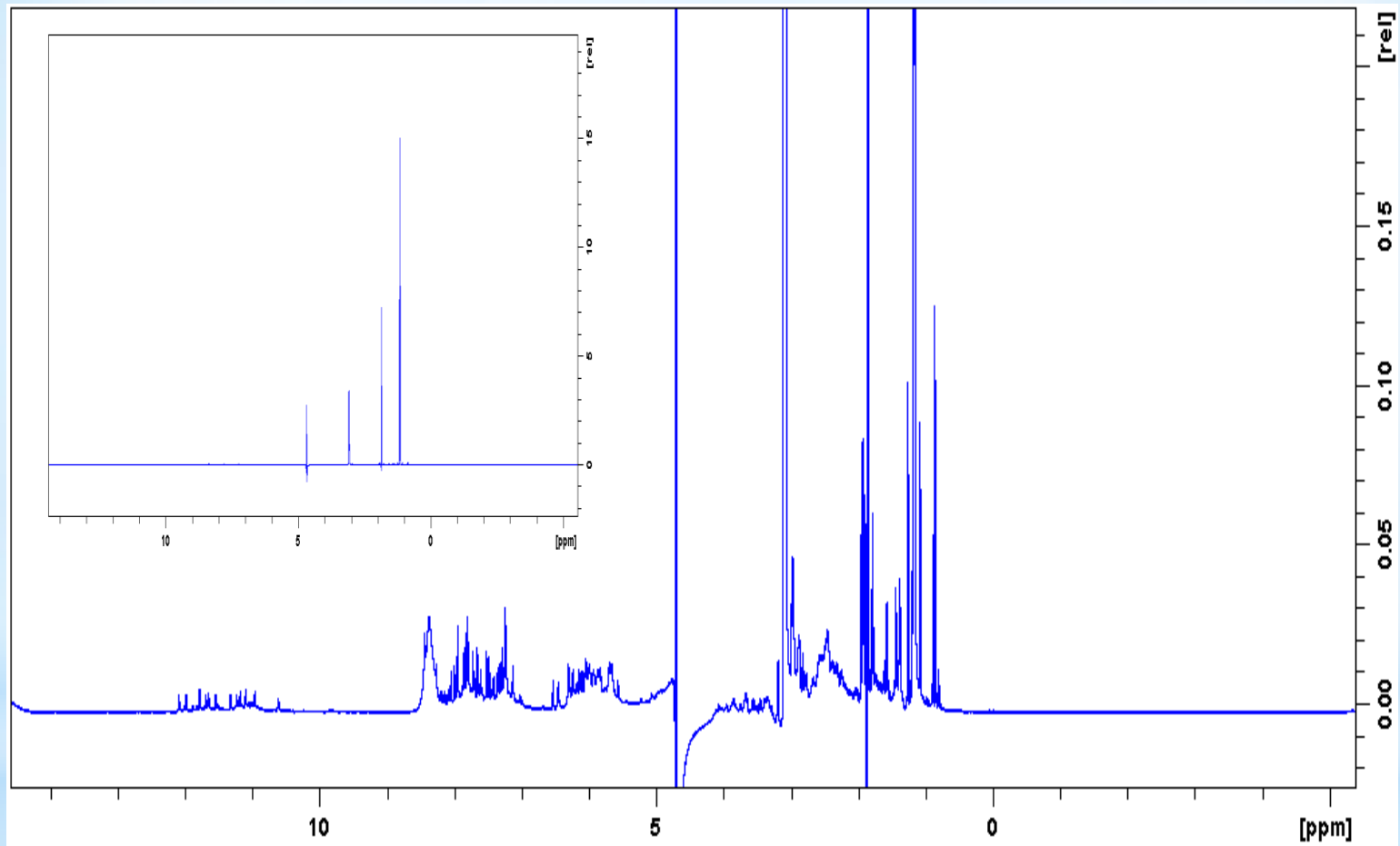
1.1 氢谱

压水峰的一维氢谱脉冲信息如下：



1.1 氢谱

压水峰的一维氢谱谱图如下：



1.1 氢谱

zgpr:预饱和单脉冲，（在单脉冲前加一个低功率脉冲）

General

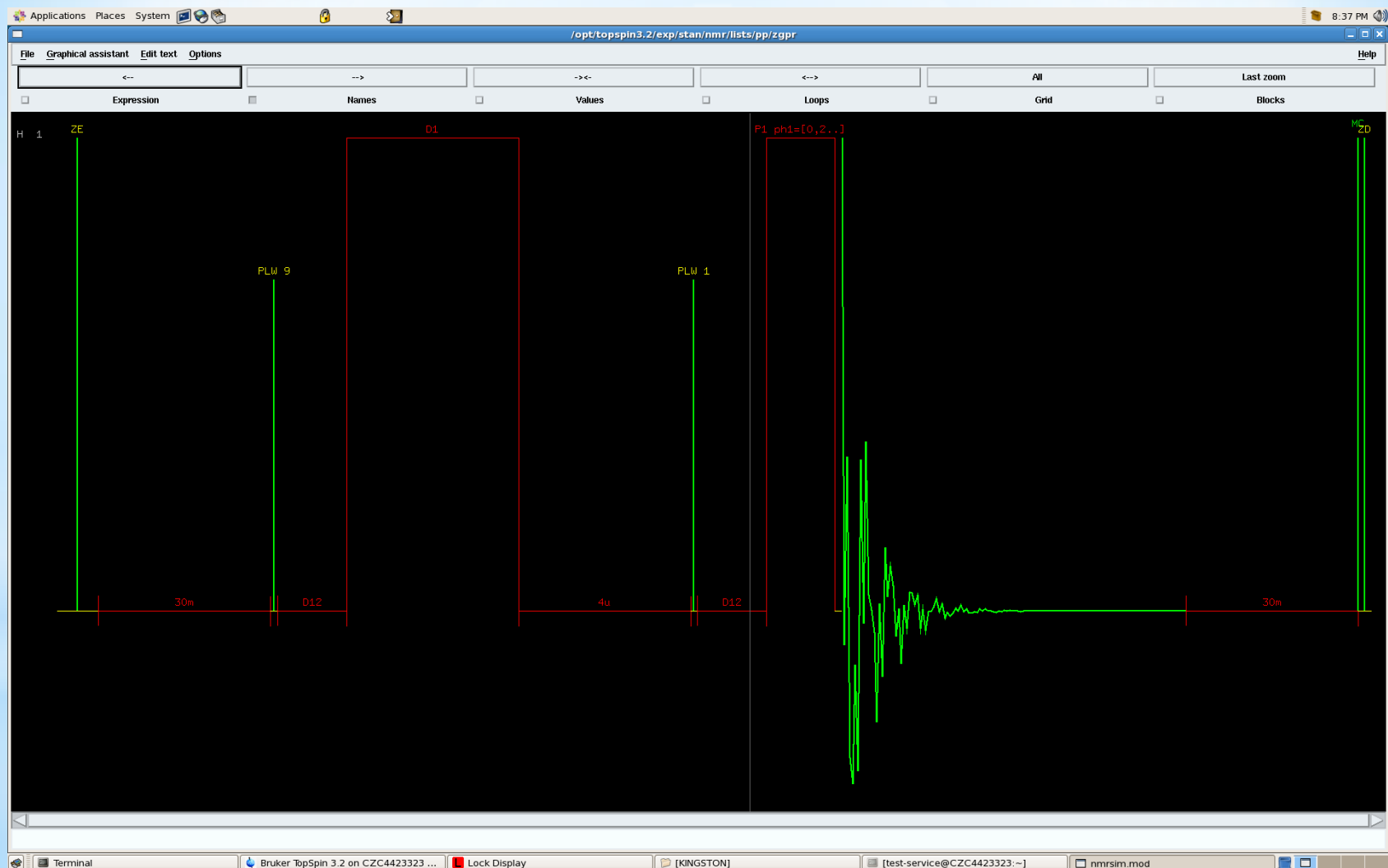
PULPROG	zgpr	...	E	Pulse program for acquisition
TD	65536			Time domain size
SWH [Hz, ppm]	14097.74	20.1347		Sweep width
AQ [sec]	2.3243434			Acquisition time
RG	16			Receiver gain
DW [μsec]	35.467			Dwell time
DE [μsec]	10.00			Pre-scan-delay
D1 [sec]	1.60000002			Relaxation delay; 1-5 * T1
d12 [sec]	0.00002000			Delay for power switching [20 usec]
DS	8			Number of dummy scans
NS	128			1 * n, total number of scans: NS * TD0
TD0	1			Dimension of accumulation loop

Channel f1

SFO1 [MHz]	700.1732908			Frequency of ch. 1
O1 [Hz, ppm]	3290.80	4.700		Frequency of ch. 1
NUC1	1H	Edit...		Nucleus for channel 1
P1 [μsec]	3.40			F1 channel - 90 degree high power pulse
PLW1 [W, -dBW]	10	-10.00		F1 channel - power level for pulse (default)
PLW9 [W, -dBW]	4.2025e-05	43.76		F1 channel - power level for presaturation

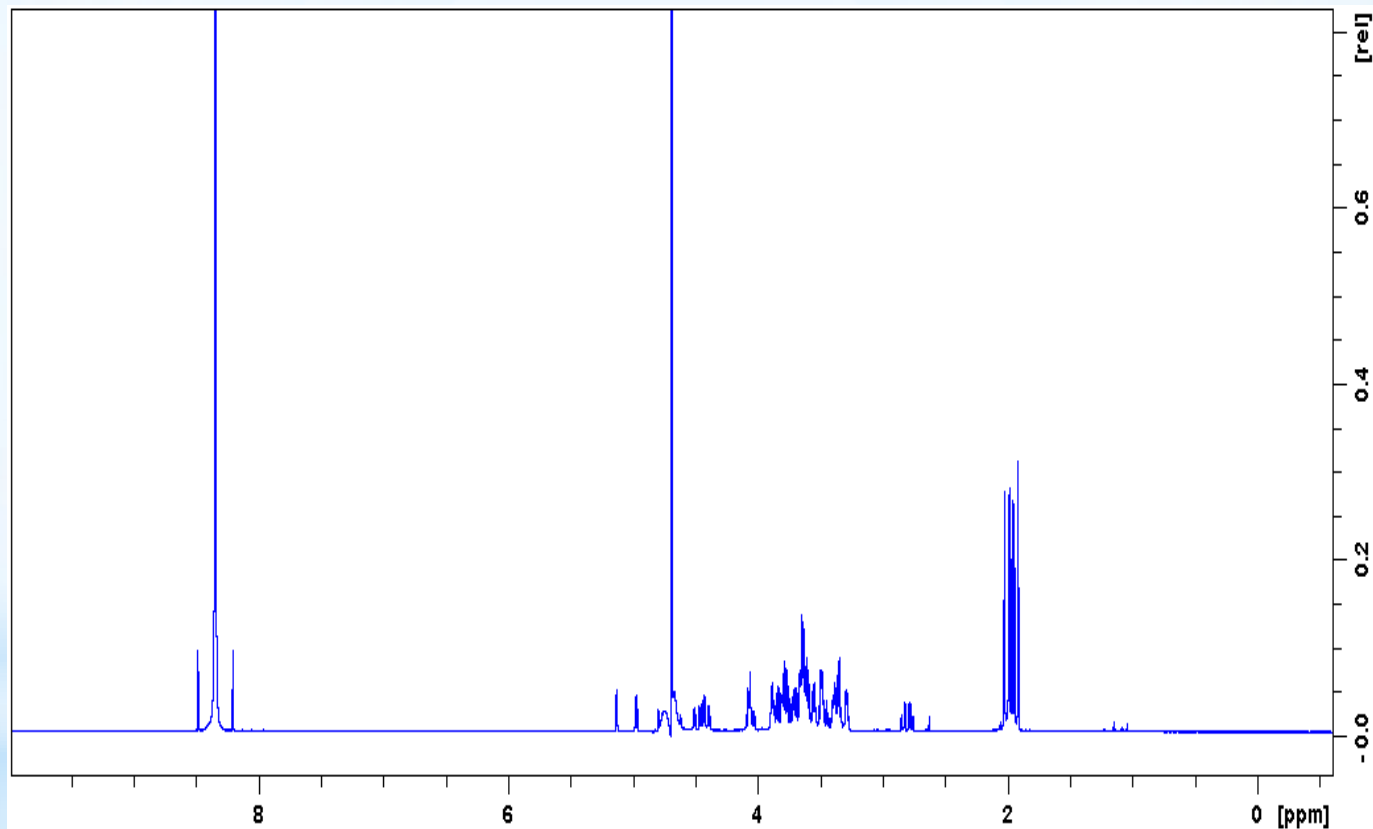
1.1 氢谱

zgpr:预饱和单脉冲，（在单脉冲前加一个低功率脉冲）



1.1 氢谱

zgpr预饱和单脉冲序列对应的谱图如下：

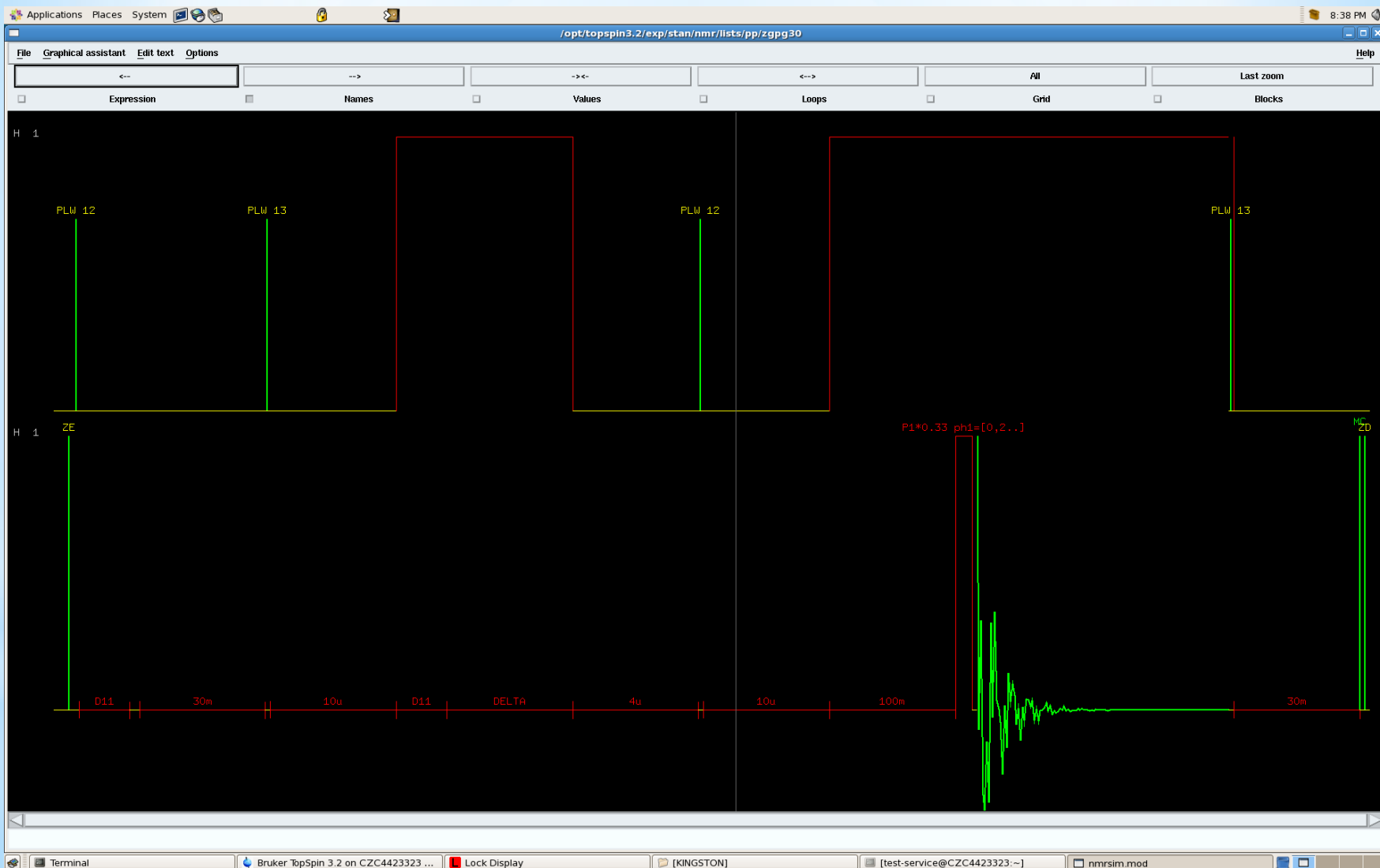


1.2 碳谱

zgpg30:

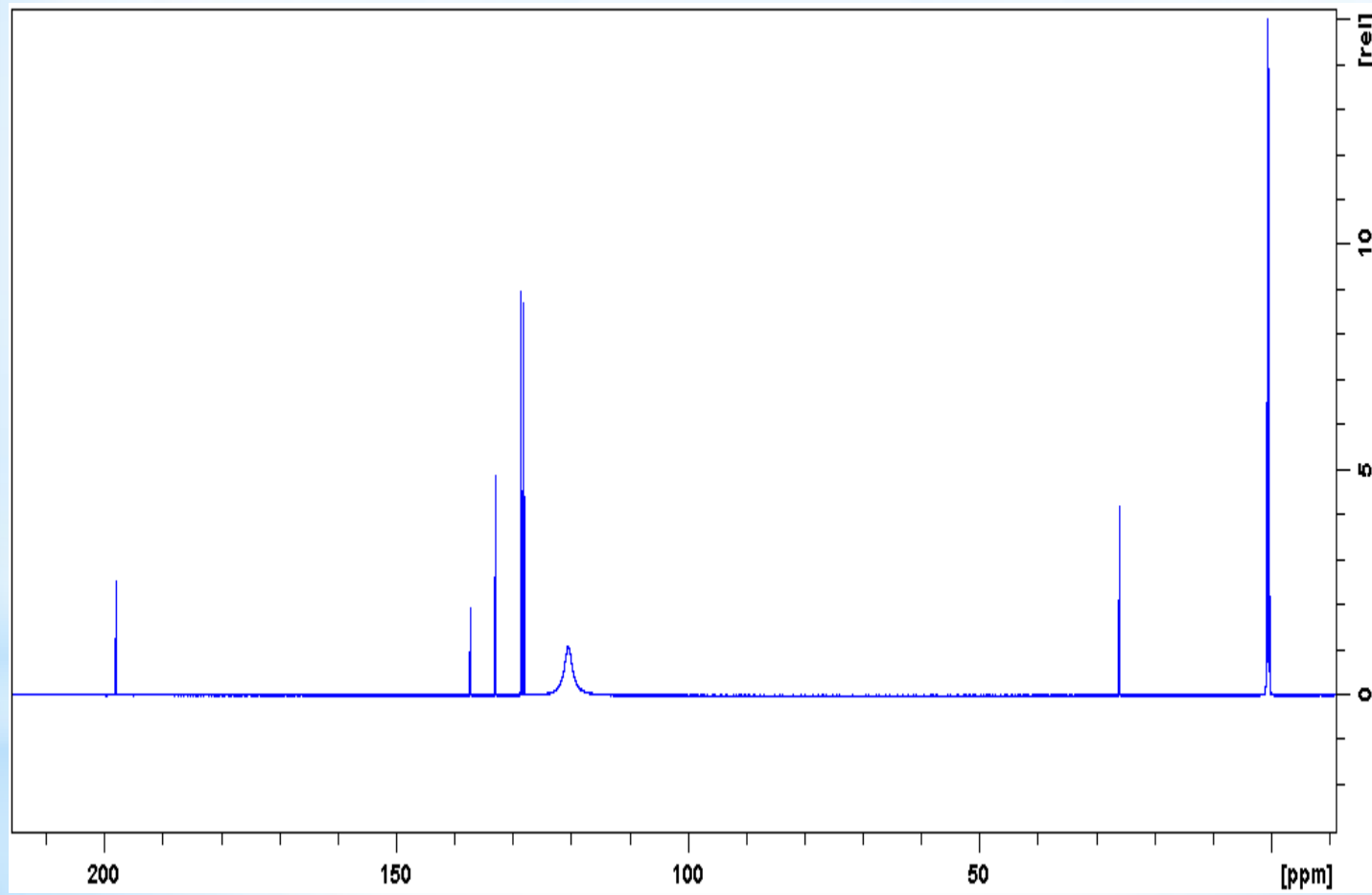
PULPROG	zgpg30		...	E	Pulse program for acquisition
TD	65536				Time domain size
SWH [Hz, ppm]	42613.64	242.02			Sweep width
AQ [sec]	0.7689557				Acquisition time
RG	32				Receiver gain
DW [µsec]	11.733				Dwell time
DE [µsec]	18.00				Pre-scan-delay
D1 [sec]	2.00000000				Relaxation delay; 1-5 * T1
d11 [sec]	0.03000000				Delay for disk I/O [30 msec]
DELTA [sec]	1.89999998				DELTA=d1-100m
DS	16				Number of dummy scans
NS	3200				1 * n, total number of scans: NS * TD0
TD0	1				Dimension of accumulation loop
⬆ Channel f1					
SFO1 [MHz]	176.0746120				Frequency of ch. 1
O1 [Hz, ppm]	16725.50	95.000			Frequency of ch. 1
NUC1	13C	Edit...			Nucleus for channel 1
P1 [µsec]	11.60				F1 channel - 90 degree high power pulse
PLW1 [W, -dBW]	100	-20.00			F1 channel - power level for pulse (default)
⬆ Channel f2					
SFO2 [MHz]	700.1728007				Frequency of ch. 2
O2 [Hz, ppm]	2800.68	4.000			Frequency of ch. 2
NUC2	1H	Edit...			Nucleus for channel 2
CPDPRG 2	waltz16	...	E		File name for cpd2
PCPD2 [µsec]	80.00				F2 channel - 90 degree pulse for decoupling sequence
PLW2 [W, -dBW]	10	-10.00			Power PLW2
PLW12 [W, -dBW]	0.085563	10.68			F2 channel - power level for CPD/BB decoupling
PLW13 [W, -dBW]	0.05476	12.62			F2 channel - power level for second CPD/BB decoupling

zgpg30:



1.2 碳谱

zgpg30脉冲序列对应的谱图：



1.2 碳谱

zgig:反门控去耦一维碳谱

General			
PULPROG	zgig		Pulse program for acquisition
TD	16384		Time domain size
SWH [Hz, ppm]	42613.64	242.017	Sweep width
AQ [sec]	0.1922389		Acquisition time
RG	32		Receiver gain
DW [μsec]	11.733		Dwell time
DE [μsec]	18.00		Pre-scan-delay
D1 [sec]	10.00000000		Relaxation delay; 1-5 * T1
d11 [sec]	0.03000000		Delay for disk I/O [30 msec]
DS	16		Number of dummy scans
NS	3000		1 * n, total number of scans: NS * TD0
TD0	1		Dimension of accumulation loop
Channel f1			
SFO1 [MHz]	176.0772529		Frequency of ch. 1
O1 [Hz, ppm]	19366.37	110.000	Frequency of ch. 1
NUC1	13C	Edit...	Nucleus for channel 1
P1 [μsec]	11.60		F1 channel - high power pulse
PLW1 [W, -dBW]	100	-20.00	F1 channel - power level for pulse (default)
Channel f2			
SFO2 [MHz]	700.1735709		Frequency of ch. 2
O2 [Hz, ppm]	3570.87	5.100	Frequency of ch. 2
NUC2	1H	Edit...	Nucleus for channel 2
CPDPRG 2	waltz16	... E	File name for cpd2
PCPD2 [μsec]	80.00		F2 channel - 90 degree pulse for decoupling sequence
PLW2 [W, -dBW]	10	-10.00	Power PLW2
PLW12 [W, -dBW]	0.091441	10.39	F2 channel - power level for CPD/BB decoupling

如果是定量碳谱，则d1取15s，p1取3.8