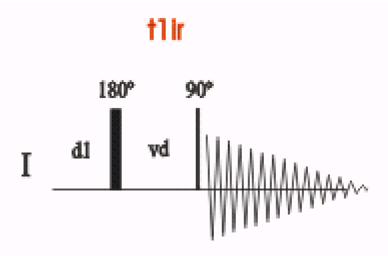
T1 Experiment

Introduction

When an NMR sample sits in the magnet, the applied static magnetic field B_0 will generate the equilibrium magnetization M_0 along +z axis. When a RF pulse is allied to the sample, the net magnetization will be rotated away from +z axis. T1 relaxation (longitudinal or spin-lattice) is the process by which the net magnetization goes back to its initial maximum value ($M_{z,eq}$) parallel to B_0.

The inversion-recovery experiment measures T_1 relaxation times of any nucleus. If the net magnetization is placed along the -z axis, it will gradually return to its equilibrium position along the +z axis at a rate governed by T1. The equation governing this behavior as a function of the time t after its displacement is:

$$M_z(t) = M_{z,\mathrm{eq}}\left(1 - 2e^{-t/T_1}\right)$$

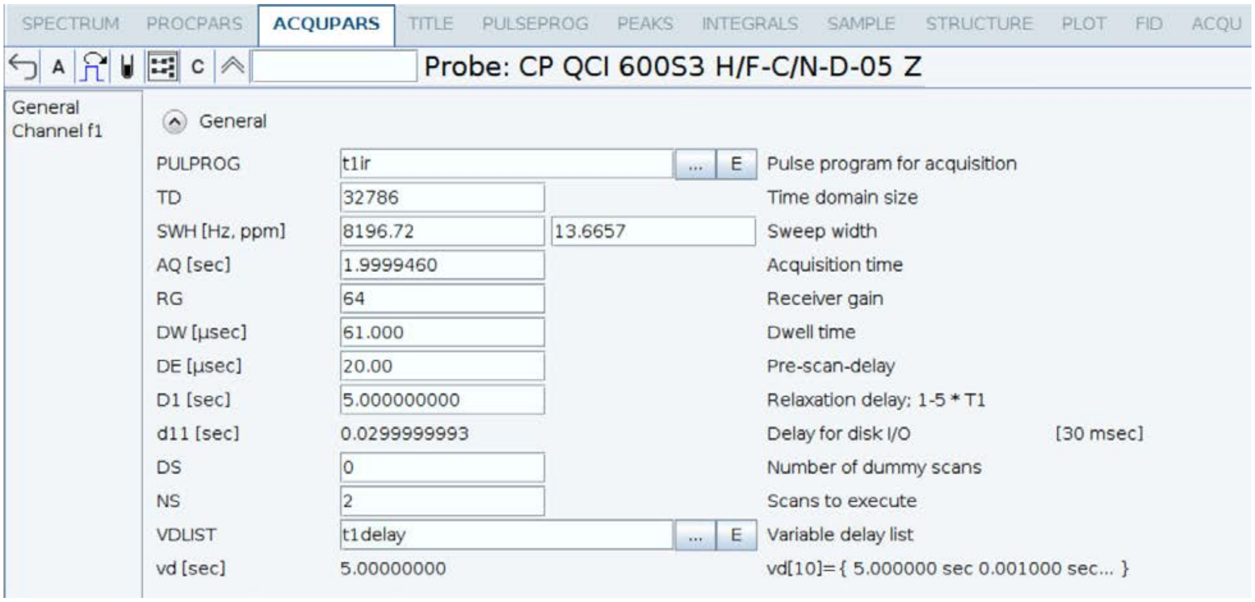


The basic pulse sequence consists of an 180^0 pulse that inverts the magnetization to the -z axis. During the following delay, relaxation along the longitudinal plane takes place. Magnetization comes back to the original equilibrium z-magnetization. A 90^0 pulse creates transverse magnetization. The experiment is repeated for a series of delay values taken from a variable delay list. A 1D spectrum is obtained for each value of vd and stored in a pseudo 2D dataset. The longer the recycle delay (d1) is, the more precise the T1 measurement is. Ideally d1 should be set to 5*T1. A rough estimation of the T1 value can be calculated from the null-point value by using T1=tnull/ln2.

Setting up proton T1 Experiment

1) To set up a T1 experiment, start with recording a normal proton spectrum to adjust the spectral sweep width **SWH**, acquisition time **aq** and other parameter if necessary.
2) Create new dataset and load "**Proton_T1"** parameter set. Update the parameters with the ones you obtained from last step. The recycle delay **D1** should be ~2-5*T1. Adjust **NS** accordingly to give sufficient S/N (**fig 1**).
3) Edit the "t1delay" by clicking on E at VDLIST line in **fig 1. Fig 2** is a good starting list.
4) Change the "TD" value for F1 dimension to the number in your VDLIST (**fig 3**)
5) Collect the pseudo 2D T1 dataset

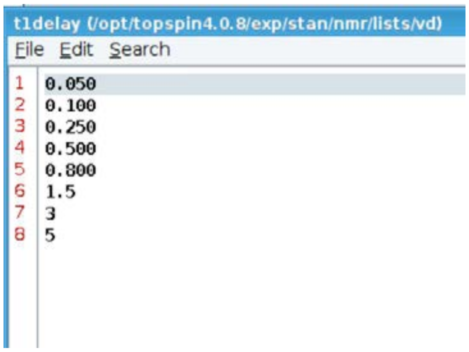
Fig 1. ACQUPARS display in “pulse program parameters” view



Probe: CP QCI 600S3 H/F-C/N-D-05 Z

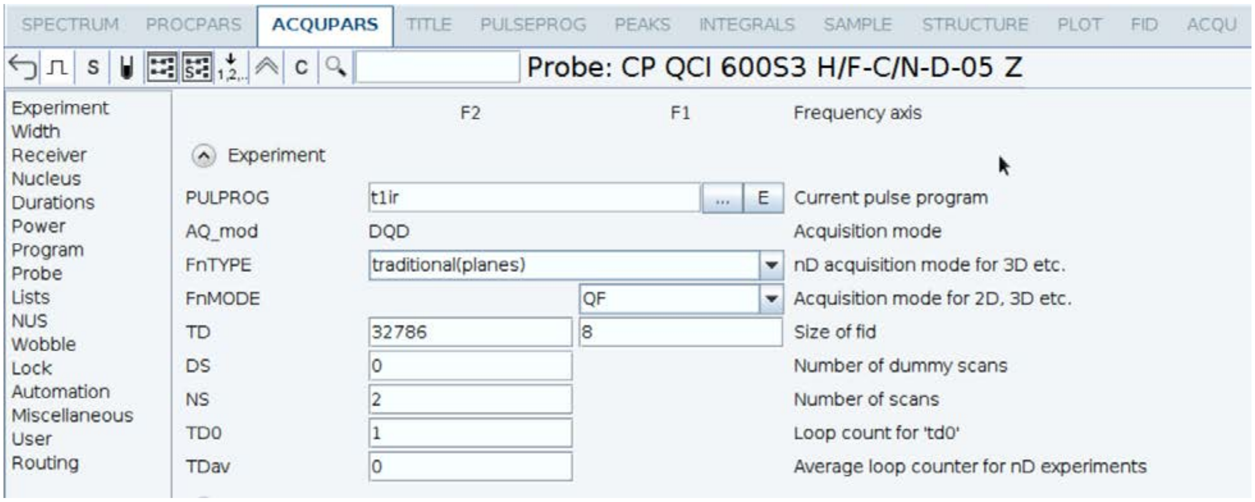
PULPROG	t1ir		Pulse program for acquisition
TD	32786		Time domain size
SWH [Hz, ppm]	8196.72	13.6657	Sweep width
AQ [sec]	1.9999460		Acquisition time
RG	64		Receiver gain
DW [µsec]	61.000		Dwell time
DE [µsec]	20.00		Pre-scan-delay
D1 [sec]	5.000000000		Relaxation delay; 1-5 * T1
d11 [sec]	0.0299999993		Delay for disk I/O [30 msec]
DS	0		Number of dummy scans
NS	2		Scans to execute
VDLIST	t1delay		Variable delay list
vd [sec]	5.00000000		vd[10]= { 5.000000 sec 0.001000 sec... }

Fig 2. An example of t1delay list with 8 delays



```
0.050
0.100
0.250
0.500
0.800
1.5
3
5
```

Fig 3. ACQUPARS display in “all acquisition parameters” view



Probe: CP QCI 600S3 H/F-C/N-D-05 Z

	F2	F1	Frequency axis
PULPROG	t1ir		Current pulse program
AQ_mod	DQD		Acquisition mode
FnTYPE	traditional(planes)		nD acquisition mode for 3D etc.
FnMODE		QF	Acquisition mode for 2D, 3D etc.
TD	32786	8	Size of fid
DS	0		Number of dummy scans
NS	2		Number of scans
TD0	1		Loop count for 'td0'
TDav	0		Average loop counter for nD experiments

Processing

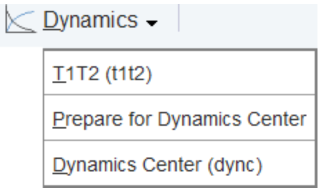
1) Process and adjust phase for the dataset. Use **rser n** (n is the number of total delays) to read out the last fid. Process and phase correct it. On the Adjust Phase toolbar, click **Save for spectrum**.

2) Go back to pseudo 2D T1 dataset by closing the 1D window

3) At the command prompt, type **xf2** to process only the F2 axis. Type **abs2** to baseline correct the rows.

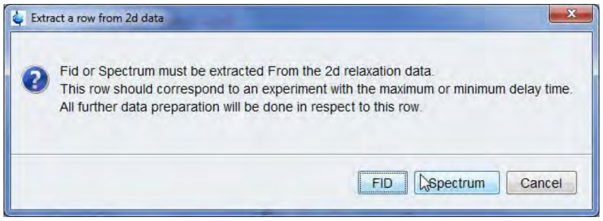
4) On the menu bar, click **Applications**.

5) On the **Dynamics** button, click the drop-down arrow to see more options and in the list, select **T1/T2 Module**.



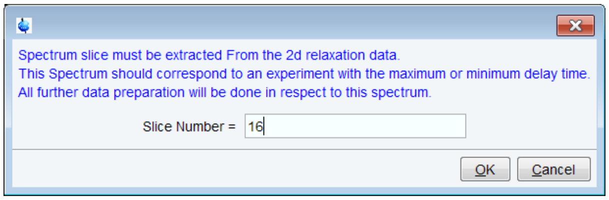
6) The flow buttons change to determine the T1 / T2 relaxation times. While executing the steps below, message windows will be displayed. Please read each message thoroughly and follow the instructions. On the Workflow button bar, click **Fid**

7) In the Extract a row from 2d data window, click **Spectrum**



Fid or Spectrum must be extracted From the 2d relaxation data.
This row should correspond to an experiment with the maximum or minimum delay time.
All further data preparation will be done in respect to this row.

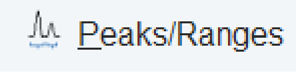
8) Enter Slice Number = **n** (the last one).



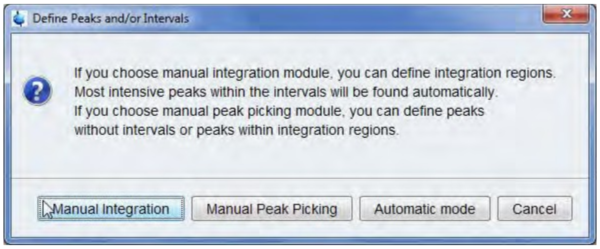
Spectrum slice must be extracted From the 2d relaxation data.
This Spectrum should correspond to an experiment with the maximum or minimum delay time.
All further data preparation will be done in respect to this spectrum.

Slice Number = 16

9) On the Workflow button bar, click **Peaks/Rang**



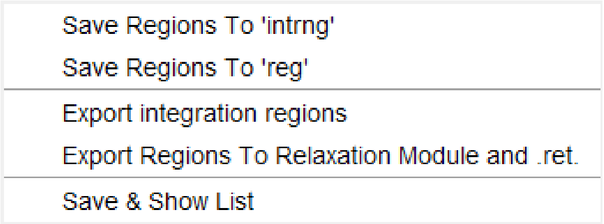
10) In the Define Peaks and/or Integrals window, click **Manual Integration**.



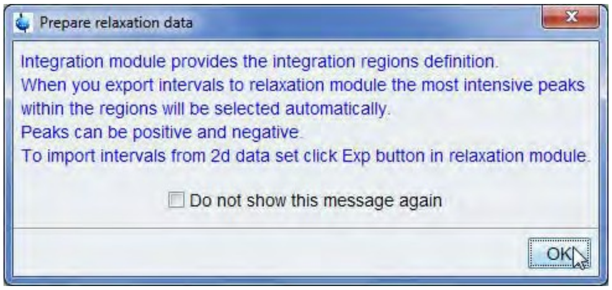
If you choose manual integration module, you can define integration regions.
Most intensive peaks within the intervals will be found automatically.
If you choose manual peak picking module, you can define peaks
without intervals or peaks within integration regions.

11) Define the regions by drawing an integral over the peaks of interest, On the Integration toolbar, click **Save/export integration regions**

12) In the list, select **Export Region To Relaxation Module**.



13) In the Prepare relaxation data window, click **OK**



Integration module provides the integration regions definition.
When you export intervals to relaxation module the most intensive peaks within the regions will be selected automatically.
Peaks can be positive and negative.
To import intervals from 2d data set click Exp button in relaxation module.

☐ Do not show this message again

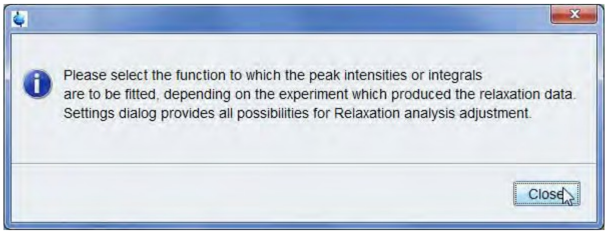
14) On the Workflow button bar, select **Relaxation**.

15) By default, the selected areas are peak-picked, and the first peak is displayed in the Relaxation window.

1.   .

16) On the Workflow button bar, select **Fitti**

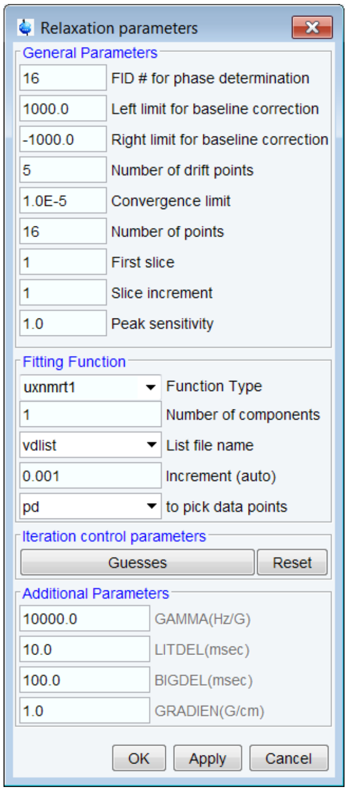
17) In the message window, click **Close**.



Please select the function to which the peak intensities or integrals
are to be fitted, depending on the experiment which produced the relaxation data.
Settings dialog provides all possibilities for Relaxation analysis adjustment.

18) In the Relaxation parameters window, click **OK** and select **Area** as Fitting type.

19) On the Workflow button bar, select **Calculation.** > Calculation



General Parameters

16	FID # for phase determination
1000.0	Left limit for baseline correction
-1000.0	Right limit for baseline correction
5	Number of drift points
1.0E-5	Convergence limit
16	Number of points
1	First slice
1	Slice increment
1.0	Peak sensitivity

Fitting Function

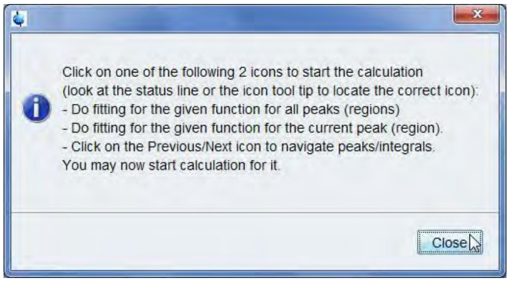
uxnmrt1	Function Type
1	Number of components
vdlist	List file name
0.001	Increment (auto)
pd	to pick data points

Iteration control parameters

Additional Parameters

10000.0	GAMMA(Hz/G)
10.0	LITDEL(msec)
100.0	BIGDEL(msec)
1.0	GRADIEN(G/cm)

20) In the message window, click **Close**.



Click on one of the following 2 icons to start the calculation
(look at the status line or the icon tool tip to locate the correct icon):
- Do fitting for the given function for all peaks (regions)
- Do fitting for the given function for the current peak (region).
- Click on the Previous/Next icon to navigate peaks/integrals.
You may now start calculation for it.

21) In the T1/T2 tools bar, click **Calculate fit for all peaks**

Brief Report

```
Region 1 from 7.797 to 7.650 ppm
T1   =     1.339s
Region 2 from 7.313 to 7.159 ppm
T1   =     1.294s
Region 3 from 6.860 to 6.413 ppm
T1   =   555.498m
Region 4 from 4.891 to 4.725 ppm
T1   =   644.916m
Region 5 from 3.414 to 3.321 ppm
T1   =     1.110s
Region 6 from 2.076 to 1.372 ppm
T1   =   398.815m
Region 7 from 1.171 to 0.552 ppm
T1   =   378.896m
```

22) On the Workflow button bar, select **Report** Report