

Bruker Timed Based Experiment For HTSL Pump Transit Volume Calibration

This pulse sequence should be used to record independent single scan data over a time sensitive period for a pump volume calibration. The formula for calculating the transit volume is below.

$$\left(\frac{FID\# \times Acqu.Time}{60\text{sec}} \right) \times Flowrate = TransitVolume$$

Pulse Sequence

(This is a modified T1 kinetics exp't)

```
;kinet.jsh
;avance-version
;Kinetics measurement (no decoupling)

#include <Avance.incl>

"d11=30m"

1 ze
  d8
2 d1
  p1 ph2
  go=2 ph31
  d11 wr #0 if #0
  lo to 1 times 14
exit

ph1=0 2
ph2=0 0 2 2 1 1 3 3
ph31=0 0 2 2 1 1 3 3

;p11 : f1 channel - power level for pulse (default)
;p1 : f1 channel - 90 degree high power pulse
;d1 : relaxation delay; 1-5 * T1
;d8 : kinetics delay
;d11: delay for disk I/O [30 msec]
;l4: 14 = number of experiments
;NS: 1 * n
;DS: 0
;td1: number of experiments

;this pulse program produces a ser-file (PARMOD = 2D)
```

Acquisition Parameters:

Use *ased* to change these acquisition parameters:

```
ns = 1;
ds = 0;
aq = 4.5;
d8 = 30 milliseconds;
L4 = 20;      L4 represents your number of scans.
td1 = 32.
d1 = (6 seconds - aq - d8 - d11)
```

On the last parameter, set the td1 value to be a power of two, but it probably would work fine with it set to 20. The parameter si1, however, needs to be a power of two (i.e., 32). Using 32 for td1 will give you 12 "empty" fid's in the 2D dataset but I don't think that should be a problem.

If you use the Bruker command "ased" to set up the experiment it will parse all the relevant variables. Note that if you are running one-scan spectra the interpulse delay can be set with either d8 or d1 or the sum of both.

Processing Procedure:

Before acquiring, ensure the parameter "mc2" is set to "qf".

1. Do rsr 1 to read the first fid
2. Process and phase the first fid, then use the "save as 2d" option
3. Go back to the 2d and check in *edp* that "pk" is set for phasing in *f2* - also check that the weighting functions are set correctly (e.g. *wdw2* = em, *lb2* = ca 0.1 - 1.0 depending on aq)
4. Use *xf2* to carry out the FT in the *f2* dimension
5. Setting the 2d display mode to [serial] should allow you to scroll through the spectra, or you can use *rsr 1*, *rsr 2*, etc., to read the individual rows.

Note: Timing Inconsistency (expt)

Many times there are 5 or 6 second delays that are not accumulative with the number of scans. A simple way to figure out what it is on your system is set L4 to one scan and subtract the aq and d1 time. This seems to be a wait/compiling delay built into the programming.

This experiment was written under the assumption that an initial scan would be done manually, then the kinetics experiment started. Thus, the delays execute prior to the first pulse, so the delay you're seeing is the fact that d8 and d1 occur at the beginning of the experiment. When running one-scan spectra, the interpulse delay can be set with either d8 or d1 or the sum of both.

A special thanks to Dr. John Harwood of the UIC NMR Facility for the development of the pulse sequence.

Here is an alternate pulse sequence from Rev.1

“d0=3u”

“dll=30ms”

ze

d1

p1 ph1

go=2 ph31

d11 wr #0 if #0 zd

d1 mc #0 to 2 F1QF(id0) newer way to loop 2D increments

lo to 2 times td1

exit

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

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

p11 : f1 channel – power level for the pulse (default)

p1 : f1 channel – 90 degree high power pulse

d0 : incremented delay for 2 D O [3us]

d11 : delay for disk I/O [30ms]

d1 : relaxation delay

td1 : number of experiments