

Sample volume: 550 µl, 4.2 cm in tube
Mix very well

Lift On
Place tube-with-rotor in magnet
Lift Off

First spectrum in new month: find example file '2103name' and move this in spectrum-window
File-New 2103name and change to 2103yourname, use this file-name-code for 400-liq
YYMMname

Next spectrum -find your Last spectrum
○-□ nmrafd open this Folder to see file-names
○-□ 2103yourname open this Folder to see all your measurements
drag last number in Topspin -and

>i increase experiment number

>h rpar h* read 1H-parameters
choose h1
Copy All

>lk gives lock-solvent list
Choose the solvent - ok

>atma automatic Tune and Match-1H-frequency (is to improve sensitivity, and for correct pulses)

For shimming you can choose Topshim or Shim manually using shim-knobs (shim is improve line-shape)

>ts (TopShim)
start
when Topshim: completed
Close

or

(>rb read standard shimfile, if shims are bad) (rsh bbo)
On-axis - Z1 **turn knob to get higher lock level**
On-axis - Z2 **turn knob to get higher lock level**
When highest level is reached:
stand by

(if peaks are double: mix the sample better and repeat shimming)

> rga receiver gain adjust standard ns=16
>zg zero go > rz rga and zg

Click title (Fill in title e.g. compound name)
Click spectrum

>tr (transfer data from spectrometer to computer)
wait 4 sec.

>ft or >ef (fourier transform or exponential-noise reduction + ft)
> ap automatic phase correction

If enough scans:

> halt

>efp (ft + noise red + use last phase-values to phase correct) *

take out tube: Lock OFF, Lift On, take tube, Lift OFF

To measure **COSY**

>i to increase the experiment-number (copy Title)
>h gives list of 1H-parameters
Click Cosy
Copy All
Title enter title (paste Title)
click Spectrum
(>atma if ' atma' is done for your 1D-1H spectrum, skip it)
>rga receiver gain adjust
Standard >ns = 1 (exp Time = 3 min) For low concentration : change number of scans
>zg zero go >xfb (see processing manual)

To measure **13C-APT**

First spectrum in new month:

File-New 2103name YYMMname use this file-name-code for 400-liq

Next spectrum -find your Last spectrum

○-□ nmrafd open this Folder to see file-names

○-□ filename open this Folder to see all your measurements

drag last number in Topspin -and

>i increase experiment number (copy Title)

>c gives list of 13C-parameters

Click APT

Copy All

Title , enter title (paste Title)

Lock and shim (skip if you did already a 1D-1H)

>atma tune 13C and 1H (NOTE: 13C-tuning is important !)

standard ns=256 (12 minutes) , when the concentration is low: ns = 1000 (24 min)

>zg zero go

>tr transfer

>efp if enough scans >halt >efp (see processing manual)

To measure **HSQC** , when 1H and 13C-apt is finished

>i increase expno (copy Title)

>c gives list of 13C-parameters

Click HSQC

Copy (or control- click-Title)

Title enter title (paste Title)

click spectrum

>atma skip if you did atma for the 13C-apt-spectrum

>rga

standard ns=1 (exp time = 6 min) when the concentration is low: increase ns=2 or ns=4

>zg

To Measure **31P**

>31p (rpar p*) choose 31P-param , lock and shim, atma , rga , zg , enter title