U400 Phosphorus 31 Standard
QUAD Probe or [1H/19F] - [15N-31P] Broadband Probe

31P Sensitivity, 10% TMPO in C6D6

Insert sample according to separate instruction sheet

jexp1 <>
LC Main Menu
LC Setup
LC Nucleus, Solvent
LC P31
LC Benzene
rts('QUAD') <>
d1=30 <>
nt =1 <>
at=0.8 <>
lb=1.00 <>
gain=20 <>
su <>

when set up is complete:

dg <>

Lock and Shim as per UVU407 and UVU409.

ga <>

when acquisition is complete:

f full <>
aph <>
LC HOLD, then release
RC HOLD, then release
LC Expand
LC HOLD, then release
nl <>
rl(0) <>
LC Full
axis='h' <>

join experiment 1
activate main menu display
activate setup menu
select nucleus, solvent menu
select nucleus
select solvent
retrieve shims
set preacquisition delay
set number of transients
set acquisition time
set line broadening
set receiver gain
perform experiment set up
display acquisition parameters
start acquisition (will wft when complete)
display full sweep width to screen
autophase
move first cursor to the left of single line
move second cursor to the right of single line
expand region inside cursors
move first cursor to center of line
select nearest line
set reference (in hertz)
display full spectrum
set axis to hertz
dscale <>
LC HOLD, then release
RC HOLD, then release
LC Expand
CC HOLD, then release
cdc <>
LC HOLD, then release
RC HOLD, then release
dsn <>
dsn:r1 <>

display scale in hertz
move first cursor to 1500ppm
move second cursor to -500ppm
expand region inside cursors
adjust vertical scale
correct drift correction (if necessary)
move first cursor to 1000ppm
move second cursor to 600ppm
display signal-to-noise
store signal-to-noise value in register one

Note that the proton decoupler was turned on and that the line appeared as a sharp singlet.

dm='nnn' <>
dmm='c' <>
lb='n' <>
su <>
turn decoupler off
set decoupler to continuous wave mode
set line broadening to "not used"
perform experiment set up

when set up is complete:

ga <>

when acquisition is complete:

aph <>
dscale <>
LC HOLD, then release
RC HOLD, then release
dsn <>
dsn:r2 <>
text('your name, advisor's initials') <>
pl pscale pap page<>
printon dg dg1 dgs printoff <>
svf('SNP31.fid') <>

autophase
display scale in hertz
move first cursor to 1000ppm
move second cursor to 600ppm
display signal-to-noise
store signal-to-noise value in register two
enter appropriate text
plot spectrum
print parameter groups
save fid in appropriate directory

Note that the signal-to-noise value, dsn, for the proton decoupled spectrum is stored in r1, and the signal-to-noise value for the coupled spectrum is stored in register 2 of the dgs data group.

It is almost always necessary to reference a phosphorus sample to an external reference. Please read the handout on External Referencing on the U400.