$^1$H{$^{31}$P} – Phosphorus Decoupled Proton NMR on the UI400 or U500

Decoupling $^{31}$P from 1H on the UI400 or U500 is easily accomplished with the standard QUAD probe. This handout outlines a procedure to acquire $^1$H{$^{31}$P} spectra, where the phosphorus decoupling can be selective or non-selective. If you want to run this experiment on the U400 or VXR500 a cabling change is required. Check with Vera Mainz before attempting to run this experiment on any instruments EXCEPT the UI400 or U500.

Preliminaries:

- **Insert, Lock and Shim** as usual.
- If you are doing variable temperature work AND want to acquire $^1$H{$^{31}$P} spectra, check with Lab staff as the temperature change might affect the efficiency of the decoupling.

Acquire Preliminary Spectra:

NOTE: Even if you’ve acquired this data before, you need to run them again.

jexp1

Setup and run a standard $^1$H experiment.

jexp2

Setup and run a standard $^{31}$P experiment. Make sure you can see all the $^{31}$P peaks you plan to decouple.

1) If you want to decouple all $^{31}$P peaks at the same time, place the left cursor at the center of the region of interest and type movetof <rtn>. Record tof=____________________.

2) If you plan to acquire selectively decoupled $^1$H{$^{31}$P} spectra, place the cursor on the center of a $^{31}$P peak of interest, type movetof. Record tof=____________________.

If you have more than one $^{31}$P peak to decouple, you need to reload the FILE-$31$P-REFN fid and wft for each frequency. After the wft, place the cursor on the center of the $^{31}$P peak of interest, type movetof. Record tof=____________________.

Continue to repeat this step for each $^{31}$P peak of interest.

NOTE: You need to reload the reference $^{31}$P spectrum each time before you execute another movetof command!!

$^1$H{$^{31}$P} Decoupling Setup – decoupling all $^{31}$P peaks at the same time:

jexp3

Setup and run a standard $^1$H experiment.

mp(1,3) move the 1H parameters from exp1 to exp3

xdec sets up for $^1$H{$^{31}$P} decoupling

Enter nucleus to be decoupled (e.g. P31): P31 <rtn>
Check that dn=P31

dof=________

set the tof value from the $^{31}\text{P}$ spectrum, above; if you have multiple values, you can do them one at a time or in an array.

nt=1, if possible, but the minimum number of scans needed
ga

NOTE: To acquire a $^{31}\text{P}$-coupled and decoupled array (so you can compare/plot them easily) set

dm=’nnn’, ’nny’
gain=’y’

Turns off the autoscale gain function for arrays
dssa or dssh
display arrayed data stacked vertically or horizontally
pl(’all’) pscale page
plot stacked data

$^{1}\text{H}\{^{31}\text{P}\}$ Decoupling Setup – decoupling $^{31}\text{P}$ peaks one at a time:

Use the same setup as above, with the following changes:

jexp3
join experiment 1
mp(1,3)
move the 1H parameters from exp1 to exp3
xdec
sets up for $^{1}\text{H}\{^{31}\text{P}\}$ decoupling

Enter nucleus to be decoupled (e.g. P31): P31<rtn>

Check that dn=P31

dof=________

set the tof value from the $^{31}\text{P}$ spectrum, above; if you have multiple values, you can do them one at a time or in an array.

dm=’nny’
dmm=’c’
dmf=250
dpwr=20 (This may change depending on how close your peaks are to each other and the JPP coupling constant. Check with Lab staff if you have problems.)

nt=1, if possible, but the minimum number of scans needed
ga

Example: Bis(diphenylphosphino)methane monoxide (dppmO) – Ph$_2$P-CH$_2$-(Ph$_2$)P=O (data from Inorg. Chem. v/9(7), 1980, 1982-1987):

$^{2}\text{J}_{\text{P-CH}} = <0.5 \text{ Hz}; \quad ^{2}\text{J}_{\text{P(O)CH}} = 12.5 \text{ Hz}$

$\delta \ ^{1}\text{H}$: CH$_2$ = 3.06 (doublet) (in my data, the doublet at ~ $\delta$ 3.22 is the CH$_2$ from dppmO; the triplet at ~ $\delta$ 3.74 is the CH$_2$ from dppmO$_2$)

$\delta \ ^{31}\text{P}$: P-CH$_2$ = -28.4 (in my data, found at -26.7ppm)

$\delta \ ^{31}\text{P}$: P(O) CH$_2$ = 27.7 (in my data, found at 33.3ppm)

$\delta \ ^{31}\text{P}$ dppmO$_2$: P(O) CH$_2$ = 24.2 (in my data, found at 28.6ppm)
dppmO – Ph₂P-CH₂-(Ph₂)P=O
All $^{31}$P Peaks Decoupled

$^{31}$P{¹H} Spectrum dppmO and dppmO₂
**dppmO – Ph₂P-CH₂-(Ph₂)P=O**

$^1$H Spectra showing $^{31}$P Peaks Decoupled One at a Time