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

Decoupling 31P 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 join experiment 1

Setup and run a standard ^{1}H experiment.

jexp2 join experiment 2

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-31P-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 join experiment 1
mp(1,3) move the 1H parameters from exp1 to exp3
xdec **sets up for 1H{31P} decoupling**
Enter nucleus to be decoupled (e.g. P31): P31<rtn>

Check that dn=P31
dof=_____ set the tof value from the ^{31}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 P31-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

$^{1}H\{^{31}P\}$ Decoupling Setup – decoupling ^{31}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 1H{31P} decoupling**
Enter nucleus to be decoupled (e.g. P31): P31<rtn>

Check that dn=P31
dof=_____ set the tof value from the ^{31}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) – $Ph_2P\text{-}CH_2\text{-}(Ph_2)P{=}O$ (data from *Inorg. Chem. v19*(7), **1980**, 1982-1987):

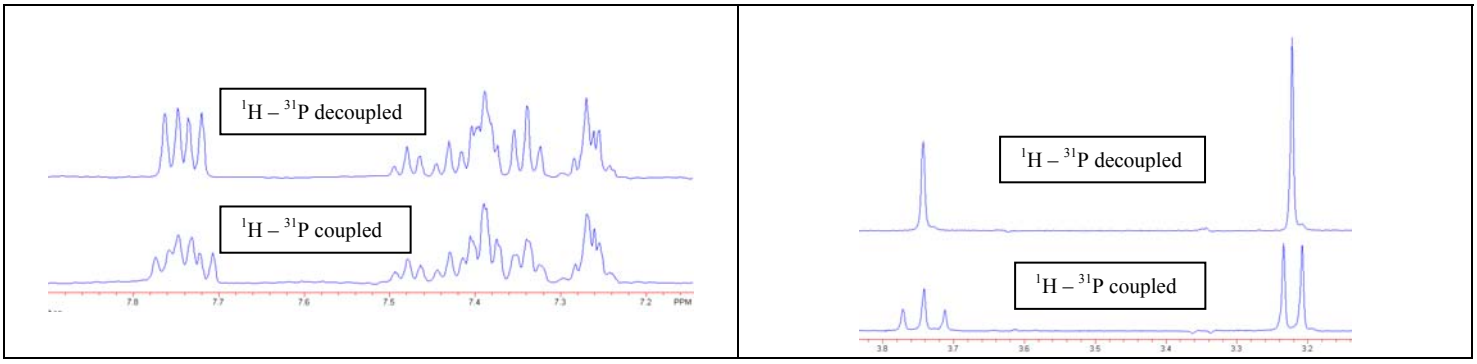
$^{2}J_{P\text{-}CH} = <0.5$ Hz; $^{2}J_{P(O)CH} = 12.5$ Hz

δ ^{1}H: $CH_2 = 3.06$ (doublet) (in my data, the doublet at ~ δ 3.22 is the CH_2 from dppmO; the triplet at ~ δ 3.74 is the CH_2 from $dppmO_2$)

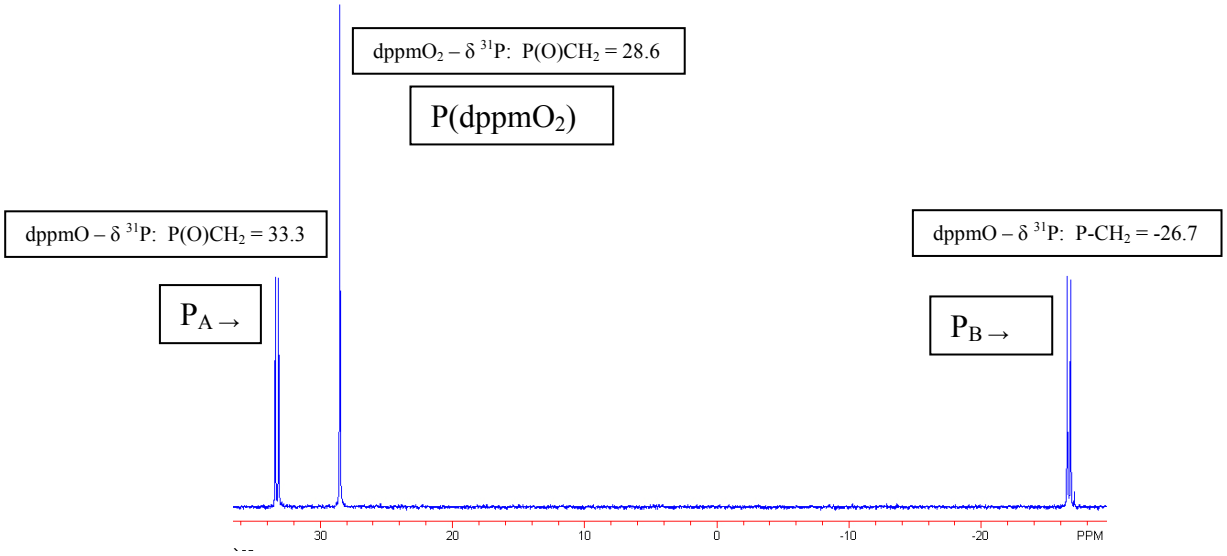
δ ^{31}P: $P\text{-}CH_2 = -28.4$ (in my data, found at -26.7ppm)
δ ^{31}P: $P(O)\ CH_2 = 27.7$ (in my data, found at 33.3ppm)
δ ^{31}P $dppmO_2$: $P(O)\ CH_2 = 24.2$ (in my data, found at 28.6ppm)

dppmO – Ph_2P-CH_2-(Ph_2)P=O

All ^{31}P Peaks Decoupled



$^{31}P\{^1H\}$ Spectrum dppmO and $dppmO_2$



dppmO – Ph_2P-CH_2-$(Ph_2)P$=O

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