Procedure for Acquiring a $^{31}\text{P}\{^1\text{H}\}$ spectrum

**NOTE:** This protocol is for qualified users. Please see Dr. Chris Jao if you intend to use it.

1. Login to WindowXp using assigned loginname.
2. Double click TOPSPIN 1.2 icon on the desktop to start the program.
3. Turn lock off by pushing Lock ON-OFF on the BSMS box.
4. Push Lift ON-OFF button on the BSMS box to eject the standard (CDCl$_3$).
5. Put your sample in the spinner and adjust the position by fitting into the gauge.
6. **While you can hear the hissing sound of the lifting gas,** load sample on the top of the magnet and push Lift ON-OFF to insert your sample.
7. On the TOPSPIN command line, type "edc Enter" and choose "31P" for $^{31}\text{P}\{^1\text{H}\}$ experiment ($^{31}\text{P}$ spectrum with $^1\text{H}$ decoupled).

8. Adjust BB channel TUNING and MATCHING according to the values listed under $^{31}\text{P}$ (Tuning = 7997, Matching = 997)
9. On the TOPSPIN command line, type "wobb" to start tuning and matching process - make sure that the bottom right corner shows "tune nucleus: 31P".
10. Push and pull the last digit of the tuning and matching tabs on the probehead until the wobble curve is exactly in the middle and its

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**TUNING** – 4 digits (7997 for $^{31}\text{P}$)

**MATCHING** – 3 digits (997 for $^{31}\text{P}$)

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31P_acquisition.doc by Chris Jao

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minimum reaches the zero line (see wobble pictures on next page) or until the tuning and matching lights on the HPPR preamplifier box all turn green.

11. Click \( \text{stop} \) to stop the wobble procedure.
12. Click \( \text{click} \) to close the wobble window.
13. Lock and shim just like you normally will do for a \( \text{\textsuperscript{1}H} \) spectrum (see document "Basic Instructions for Bruker NMR AVANCE 400 Running TOPSPIN")
14. On the dataset window you just created in step 7, type "\text{rga Enter}".
15. Change the \( \text{ns} \) according to the \( \text{rg} \) value. The sensitivity of \( \text{\textsuperscript{31}P} \) is about 400 times compared to \( \text{\textsuperscript{13}C} \)’s.
16. Type "\text{zg Enter}" to start acquisition.
17. When the acquisition is done, take out your sample and put the standard labeled "\( \text{H}_3\text{PO}_4/\text{D}_2\text{O} \)" in.
18. Type "\text{edc Enter}" and choose "\text{31P}" as the experiment to create a new directory for acquiring \( \text{\textsuperscript{31}P} \{\text{\textsuperscript{1}H}\} \) for the \( \text{H}_3\text{PO}_4 \) sample.
19. Lock \( \text{D2O} \) and shim.
20. Acquire a \( \text{\textsuperscript{31}P} \{\text{\textsuperscript{1}H}\} \) spectrum of \( \text{H}_3\text{PO}_4 \) and calibrate the only peak to 0.0 ppm by clicking \( \text{\textcircled{4}} \).
21. Type "\text{sr Enter}" to get the value of spectrum reference frequency.
22. Go back to your sample directory, type "\text{sr Enter}" and key in the \( \text{sr} \) value from the \( \text{H}_3\text{PO}_4 \) experiment. This makes \( \text{H}_3\text{PO}_4 \) an external reference.
23. Process the data - efp, apk, abs.
24. Do a peak picking and print the spectrum.
25. Once you have finished acquiring \( \text{\textsuperscript{31}P} \) data, please do the wobble procedure again to change the BB channel back to \( \text{\textsuperscript{13}C} \) (Steps 26-32).
26. Eject your sample and put \( \text{CDCl}_3 \) standard sample in.
27. On the TOPSPIN command line, type "\text{edc Enter}" and choose "\text{13C}" to create a directory for preparing wobble procedure for \( \text{\textsuperscript{13}C} \).
28. Adjust BB channel TUNING and MATCHING according to the values listed under $^{13}$C (Tuning=7984, Matching=896).

29. On the TOPSPIN command line, type "wobb" to start tuning and matching process - make sure that the bottom right corner shows "tune nucleus: $^{13}$C".

30. Push and pull the last digit of the tuning and matching tabs on the probehead until the wobble curve is exactly in the middle and its minimum reaches the zero line (see wobble pictures above) or until the tuning and matching lights on the HPPR preamplifier box all turn green.

31. Click to stop the wobble procedure.

32. Click to close the wobble window.

33. Lock on CDCl$_3$. Read in standard shim file, i.e. "rsh cdcl3.shm Enter". Push STD BY button and close all TOPSPIN related windows.

34. To transfer data to the process station- Nmr400p or to save the data to a portable device, follow the instruction of "Protocol for Access NMR400p to Process NMR data".

35. Log out.

36. Turn off the monitor while leaving the computer on.