**31P NMR Acquisition Procedures**

**For PSC & NSC Bruker 400 NMR Spectrometers**

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**NMR Background:**

**Phosphorus-31 NMR** spectroscopy (NMR stands for [nuclear magnetic resonance](http://en.wikipedia.org/wiki/Nuclear_magnetic_resonance)) is an analytical technique. Solution 31P-NMR is one of the more routine NMR techniques because 31P has an [isotopic abundance](http://en.wikipedia.org/wiki/Isotopic_abundance) of 100% and a relatively high [magnetogyric ratio](http://en.wikipedia.org/wiki/Magnetogyric_ratio). The 31P nucleus also has a spin of ½, making spectra relatively easy to interpret. Phosphorus is commonly found in [organic compounds](http://en.wikipedia.org/wiki/Organic_compound) and coordination complexes (as [phosphines](http://en.wikipedia.org/wiki/Phosphines)), making it useful to measure 31P NMR spectra routinely.

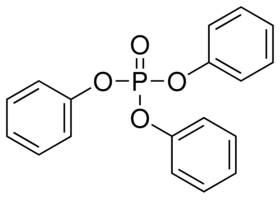
With a magnetogyric ratio 42.5% of that for 1H, 31P NMR signals are observed near 161.976 MHz on a 9.40 Tesla magnet (used for 400 MHz 1H NMR measuements). Chemical shifts are referenced to 85% [phosphoric acid](http://en.wikipedia.org/wiki/Phosphoric_acid), which is assigned the chemical shift of 0, with positive shifts to low field/high frequency.[[1]](http://en.wikipedia.org/wiki/Phosphorus-31_NMR_spectroscopy#cite_note-hoffman-1) Due to the inconsistent [nuclear Overhauser effect](http://en.wikipedia.org/wiki/Nuclear_Overhauser_effect), integrations are not useful.[[1]](http://en.wikipedia.org/wiki/Phosphorus-31_NMR_spectroscopy#cite_note-hoffman-1) Most often, spectra are recorded with protons decoupled.

The ordinary range of chemical shifts ranges from abut δ250 to -δ250, which is much wider than typical for 1H NMR.

**NMR Reference Standard Used for Setup:**

**31P Sensitivity - 0.0485M Triphenylphosphate**

Reference standard sample - 31P Sensitivity, 5 mm Ø, 0.0485 M Triphenylphosphate (TPP) in Acetone-d6.

[](http://www.google.com/url?sa=i&rct=j&q=&source=images&cd=&cad=rja&docid=wqKqHOQK1YlYPM&tbnid=UM_omnHl1P4cTM:&ved=0CAUQjRw&url=http%3A%2F%2Fwww.sigmaaldrich.com%2Fcatalog%2Fproduct%2Faldrich%2F241288%3Flang%3Den%26region%3DUS&ei=ZKe3Ufu_KpPu9ASJkIGoCw&bvm=bv.47810305,d.eWU&psig=AFQjCNE6wwXrW9CfSF-pNtG5dG2oss-ILA&ust=1371076741931934)

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**31P NMR Operation Procedures:**

Assuming you are familiar with routine 1H and 13C

LC=Left click; DC=double left click

Black and bold command need to be typed and followed with enter commond.

**PSC Data Directory: C:\Bruker\TopspinX.X\data\nmr\‘userid’**

**NSC Data Directory: /opt/topspin2.1/data/‘userid’**

|  |  |  |
| --- | --- | --- |
| **Step** | **Command** | **Notes** |
| **1** | Login to the computer | **DO NOT** share your user ID with unauthorized user. |
| **2** | Start Topspin | PSC: Use Topspin x.x as stated in proton acquisition procedure;  NSC : Use Topspin x.x as stated in proton acquisition procedure. |
| **3** | **Open an old spectrum and type ‘new’** | *Open Dataset window.*  Type in a *dataset name* in the appropriate box  Type in a title for the experiment |
| **4** | **bsmsdisp <**etr**>** | Unlock (make sure lockon-off button is not highlighted)  Unspin (make sure spin button is not highlighted). |
| **5** | **ej <**etr**>** | *Eject sample from the magnet.*  Remove spinner turbine from the top of the magnet;  Do not lean on magnet;  Take out D2O sample from the spinner turbine;  Position your sample using the sample depth gauge;  Make sure the eject air is still ON (should hear the eject air), place the turbine with your sample on top of the magnet at the magnet upper barrel opening. |
| **6** | **ij <**etr**>** | *Insert sample into the magnet.*  *Double check that the sample status on the bsmsdisp window showing* **‘down’**.  Close BSMSDISP display window. |
| **7** | **rpar** <etr> | read an appropriate parameter set for expt.  *For proton: rpar ‘Proton’;*  *NSC: For P31, rpar “duz\_P31” or duz\_P31CPD from user pull down menu.*  *PSC: For P31, rpar P31 or P31CPD from regular data set.*  */opt/topspin2.1/exp/stan/pp/nmr/par/user* |
| **8** | **lockdisp** <etr> | *Lock display window opens up* |
| **9** | **rsh** <etr> | s*him file list opens up.* LC on the file “lastbestbbo” or some other files with the same solvent. |
| **10** | **lock** <etr> | LC on the appropriate lock solvent. Wait until “lockin:finished” message appears |
| **11** | **atma <**etr**>** | Automatic probe tuning and match starts.  Wait till “atma:finished” message appears. For P31 NMR, experiments, please confirm that this is finished as it tunes both P31 and proton channels. |
| **12\*** | topshim 2h<etr> | automatic shimming starts.  Wait until the “topshim completed” message appears. NOT applicable for 100% deuterium solvent on NSC NMR. |
| **13** | Blank here. | Blank here. |
| **14** | **ased** <etr> | AquProc setup mode. |
| **15** | **getprosol** <etr> | *Read corresponding probe parameters.* |
| **16** | **rga** <etr> | *Automatic receiver gain optimization starts.*  Wait for the “rga:finished” message to appear |
| **17** | **expt** <etr> | Calculate the time to finish the exp;  Adjust “ns” if the acquisition time is too long;  DO NOT take other user’s time. |
| **18** | **zg** <etr> | *Start data acquisition.*  Wait till the “acquisition:finished” message to appear. |
| **19** | **efp** <etr>  **apk** <etr>  **abs** <etr>  command ‘**duzproc**’ does all three together. | Simple data processing |
| **20** | **bsmsdisp <**etr**>** | Unlock (make sure lockon-off button is not highlighted  Unspin (make sure spin button is not highlighted. |
| **21** | **ej <**etr**>** | Remove the spinner turbine from the top of the magnet. Take out your sample from the spinner turbine. Position the D2O sample in the turbine using the sample depth gauge. Making sure the eject air is still ON (should hear the eject air), place the turbine with the D2O sample on top of the magnet at the magnet upper barrel opening. |
| **22** | **ij <**etr**>** | *the D2O sample is inserted into the magnet.* Wait until the sample status on the bsmsdisp window button showing down. Then close BSMSDISP display window. |
| **23** | **lock d2o** <etr> | Locks d2o sample. Wait until “lockn:finished” message appears |
| **24** | **Transfer your data** | Please process this within 2 weeks’ time window as old data may be deleted without notice. |
| **25** | **exit** <etr> | Topspin terminates all active programs. Please **DO NOT** just click the crossbar as it does not always terminate all active commands.  Log out PC.  Fill out the log book  Record all error messages from the PC screen to logbook if instruments are malfunctioning.  Dr.Zhenming Du |