KJM 5300

II

^{13}C-, ^{19}F- and ^{31}P one dimensional nmr spectroscopy

Version 1.0
TopSpin 1.3 patch level 10
DPX 200, DPX 300 and DRX 500 nmr instruments
Windows XP

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When you are going to do one or more of the following one dimensional nmr experiments ^{13}C ^{31}P and ^{19}F nmr experiments in manual mode you have already configured the TopSpin program, you have made the nmr sample with correct height and concentration in an appropriate deuterated nmr solvent, removed the standard sample from the magnet of choice, inserted/lowered your own sample into the magnet, locked the magnet´s main frequency to the solvent deuterium atoms, tuned and matched (the wobbing procedure) if using the DRX 500 instrument, read a shim file, shimmed the magnet field manually and started autoshim, performed rga and acquired your ^{1}H one dimensional nmr spectrum. You must know and understand all this before continuing using the instructions provided here.

You can perform one dimensional ^{13}C nmr experiments on all three instruments. But you cannot perform one dimensional ^{31}P and ^{19}F nmr experiments at all places. You have to figure out by looking at the probe which at any time is sitting in the magnet to decide if your wanted nucleus is possible to observe on your chosen instrument. The probe of the DPX 200 instrument is show to the left below here.

When looking at the print the red/orange field you realized that ^{13}C, ^{19}F and ^{31}P are all there ☺ When you decide to do an nmr experiment that differ from what was just run on the instrument the pneumatic selection unit switches hardware to the correct settings when you define your nuclei. When you look at the probe on the DPX 300 instrument you see the following nuclei, ^{13}C, ^{15}N and ^{31}P (picture below, next page). ^{19}F experiments cannot be performed on DPX 300 instrument even though the experiments are in the list that comes up when you type rpar in the command line.

DPX 300 probe.

On the DRX 500 nmr instrument there are approximately 10 different probes available. The two most often used probes are shown above here. The so called BBO probe is sitting in the magnet in the picture. Looking at the probe we see ^{2}H, ^{1}H and BB. ^{2}H (deuterium) is related the lock channel of the instrument and BB stands for broad band which means many nuclei. To go from one nuclei to another involves a lengthy tuning and matching procedure (wobbing). Numerical values to start from are written on a paper attached to the probe. On this nmr-instrument you must always wobb = tune and match on each sample and nucleus even if the nucleus is not changed when going from one sample to the next. See the first nmr manual used in this course about this. The other most used probe on this instrument is the TXI probe. ^{2}H, ^{1}H ^{13}C and ^{15}N is stated on this probe. One dimensional ^{15}N experiments are impossible to execute on the DPX 300 and DRX 500 instruments – the sensitivity is far too low. 2D ^{1}H-^{15}N experiments are possible to perform on DRX 500. ^{31}P experiments are possible to perform on the BBO probe on the DRX 500, ^{19}F is only possible if a special ^{19}F probe is used (no picture shown here) on the DRX 500 instrument.

Above are the two probes in the two 400 MHz instruments AVII400 and AVIIHD400 shown for comparison. ^{2}H, ^{1}H and BB on one instrument and ^{2}H, ^{1}H and BBF on the other. On these instruments which most of the time are not used in manual mode the following nuclei are observable: ^{1}H, ^{13}C, ^{15}N (only 2 D inversely detected experiments with very strong samples, half amount sample and half amount solvent), ^{19}F, ^{29}Si, ^{31}P and ^{195}Pt. These nuclei are observed routinely in full automation by the program ICONNMR which is actually running the TopSpin program. One program is running another program! See a separate document about this.

You have now figured out if you can do experiments on your selected nucleus on the instrument you are using.
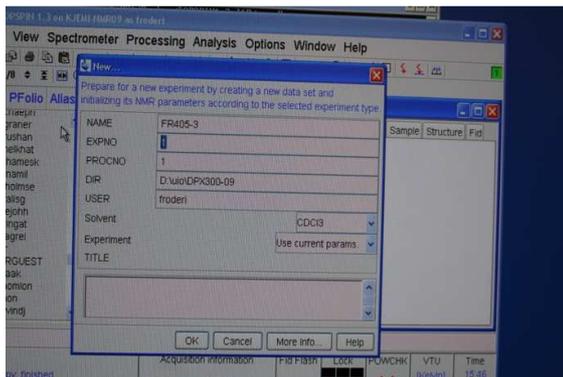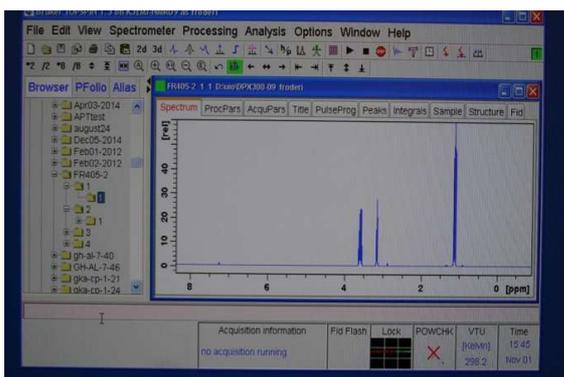
You have your completed ^{1}H experiment in the main part of the TopSpin program. The command new or the command edc brings up the experiment definition window.
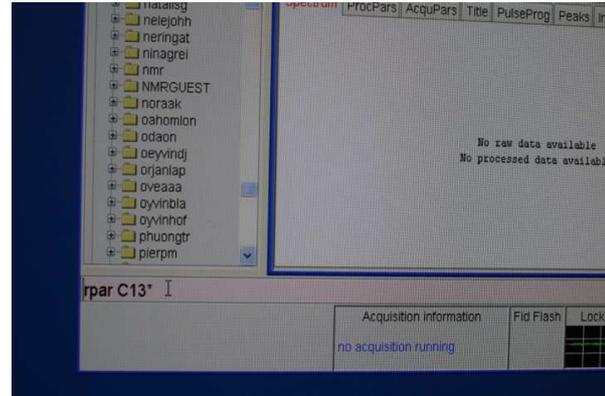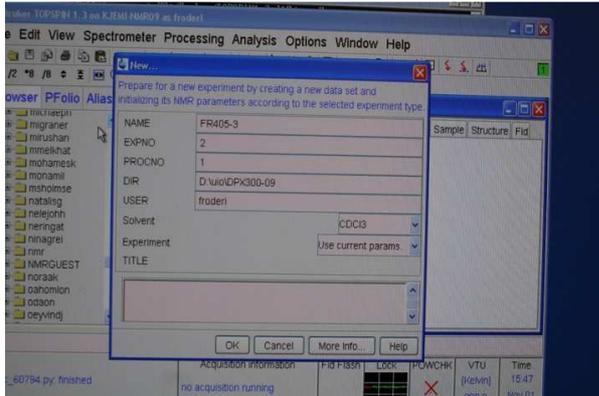
You change EXPNO from 1 to 2 and keep everything else the same. Click on OK. Then you type rpar C13* in the command line.
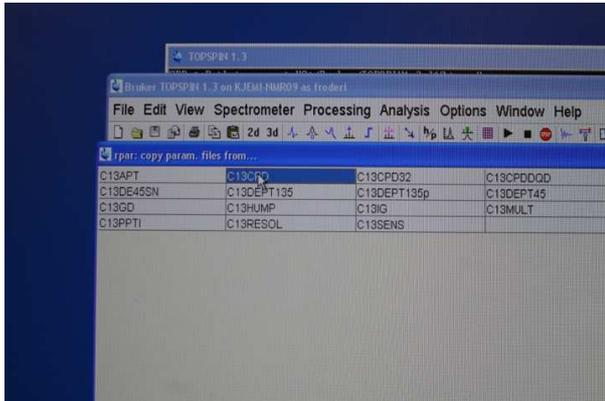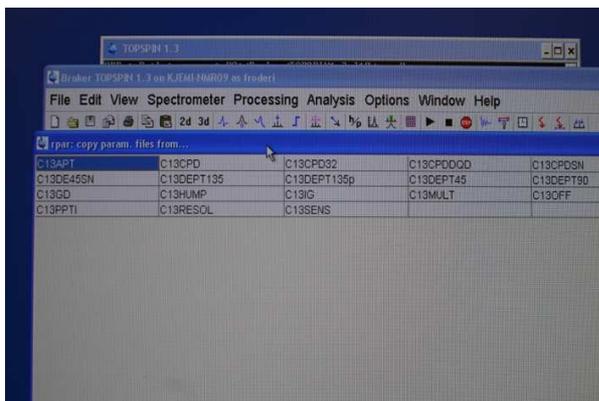

All the experiments containing C13 shows up. In KJM 5300 you shall use the following PAR files to obtain spectra: C13CPD, C13APT, C13DEPT135, C13DEPT90, C13DEPT45 and C13IG. Start with C13CPD.
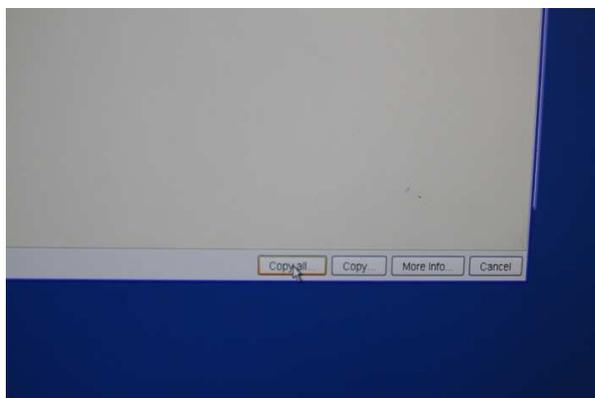

After selecting C13CPD click on Copy all.

If running out of time some of the ^{13}C experiments can be acquired in ICONNMR automation later in the KJM 5300 course. Check the following parameters and change them if needed. ns (number of scans), d1 (relaxation delay), expt (experiment time). Execute the command getprosol. Do the wobb procedure if you are on DRX 500. Then you do rga (receiver gain adjustment), zg (zero go) and wait for completion. If impatient you can use tr (transfer) to see the result before completed experiment. Use efp (fourier transformation) and apk (automatic phase correction). Use manual phasing if apk does not work properly (see the ^{1}H manual if you do not remember how). Calibrate the ppm scale (see the ^{1}H manual if you do not remember how). Adjust the height of the peaks. Find the exact ppm and or Hz values of all resonances (see the ^{1}H manual if you do not remember how). Print your spectrum (see the ^{1}H manual if you do not remember how).
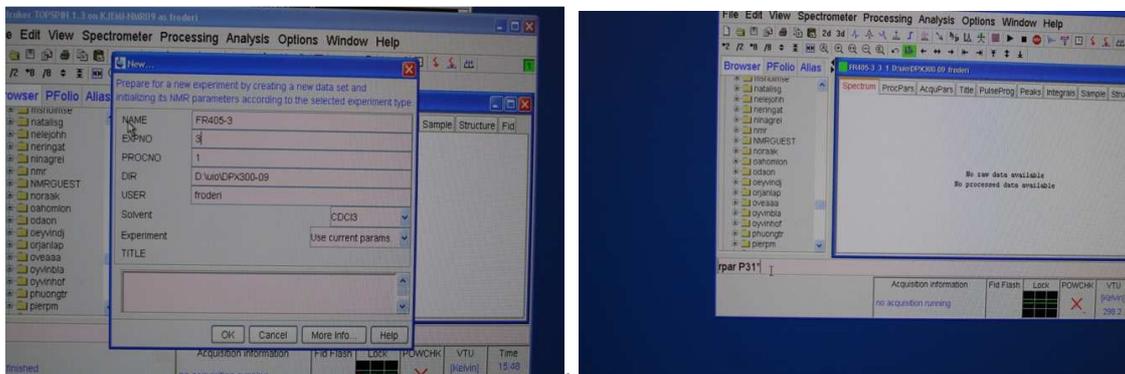

Use the command new or edc and change the EXPNO to 3.

Type rpar P31*. You have two experiments to choose from P31 and P31CPD. Choose P31CPD. Click Copy all. P31CP is similar to C13CPD – in both experiments the coupling effects to nearby protons are removed by irradiation on the protons. Check the following parameters and change them if needed. ns (number of scans), d1 (relaxation delay), expt (experiment time). Execute the command getprosol. Do the wobb procedure if you are on DRX 500, often impossible to do ^{19}F or ^{31}P experiments on the 500 due to probe limitations. Then you do rga

(receiver gain adjustment), zg (zero go) and wait for completion. If impatient you can use tr (transfer) to see the result before completed experiment. Use efp (fourier transformation) and apk (automatic phase correction). Use manual phasing if apk does not work properly (see the ^{1}H manual if you do not remember how). Calibrate the ppm scale (see the ^{1}H manual if you do not remember how). Adjust the height of the peaks. Find the exact ppm and or Hz values of all resonances (see the ^{1}H manual if you do not remember how). Print your spectrum (see the ^{1}H manual if you do not remember how).


Use the command new or edc and change the EXPNO to 4 (no picture about this is shown now- this procedure has been shown many times before and you shall remember how this is done at this stage). Type rpar F19*. Select either F19 or F19CPD. Choose F19CPD. Click Copy all. F19CP is similar to C13CPD – in both experiments the coupling effects to nearby protons are removed by irradiation on the protons. Check the following parameters and change them if needed. ns (number of scans), d1 (relaxation delay), expt (experiment time). Execute the command getprosol. Do the wobb procedure if you are on DRX 500, often impossible to do ^{19}F or ^{31}P experiments on the 500 due to probe limitations. Then you do rga (receiver gain adjustment), zg (zero go) and wait for completion. If impatient you can use tr (transfer) to see the result before completed experiment. Use efp (fourier transformation) and apk (automatic phase correction). Use manual phasing if apk does not work properly (see the ^{1}H manual if you do not remember how). Calibrate the ppm scale (see the ^{1}H manual if you do not remember how). Adjust the height of the peaks. Find the exact ppm and or Hz values of all resonances (see the ^{1}H manual if you do not remember how). Print your spectrum (see the ^{1}H manual if you do not remember how). .

At this stage in learning nmr you have discovered a problem. You cannot calibrate the ppm scale of F19 and P31 experiments. Internal standards of the form of capillary tubes (melted close on top and bottom) containing a sample with either a known ^{19}F or a known ^{31}P reference ought to be added to the nmr tube. The capillary is making the shimming a bit harder. In earlier times the capillary sometime broke in the nmr tube, this is usually not a problem

anymore since we have stopped using the old procedure of spinning the nmr tubes while doing experiments.
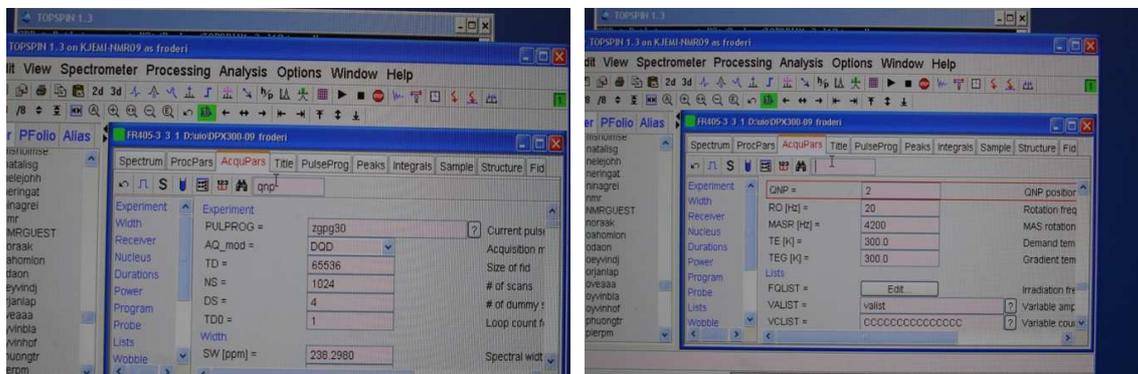
If you on DPX200 or DPX 300 see no FID (free induction decay) and no nmr signals after doing efp there might no ^{19}F or no ^{31}P in your sample. Or you have forgotten to use the command getprosol. In rare instances the so called qnp/QNP parameter in the par file is wrong. Click on AcquPar in the main Topspin window (above left). Type qnp where it is shown in the window and use also of course return/enter. Compare QNP with what is on the probe. Change accordingly and do the experiment again.

Consult the ^{1}H manual for how to leave the instrument. Inserting stand sample. Having a 1H experiment in the main TopSpin window. Locking on the solvent. Wait until the lock procedure is completed. Stop the program. Log out.