How to Use the AV300 Open Access NMR Spectrometer

The AV300 NMR spectrometer in room G21 has been set up to provide a number of popular NMR experiments in a straightforward and easy-to-use way. You need to know how to make up NMR samples, and to understand the NMR experiments on offer. You must be registered with the NMR Service and trained to use the spectrometer. You may not use the spectrometer if you have not been trained and registered with the NMR Service.

Here is an outline of what you do:

1) Prepare your sample.
2) Put the sample into the Sample Changer carousel.
3) Tell the computer about:
    a) yourself
    b) your sample
    c) the work you require.
4) Collect your samples and spectra after the work is completed.

Prepare your Sample

This is explained in another leaflet, and you should ask for a copy if you don't already have one. You should always make good quality samples, since a poor quality sample always produces a poor quality NMR spectrum. Before you start using the computer, you must put your sample tube into a spinner. Gently, and with a twisting movement, push the round end of the NMR tube into the top (wide) end of a spinner, until it just appears at the bottom. Then place the spinner with the NMR tube into the depth gauge. Hold the spinner and the depth gauge together with one hand and with the other hand gently rotate and push the tube down through the spinner until it touches the stop at the bottom of the depth gauge. Carefully, holding the NMR tube *above* the spinner, remove the tube and spinner from the depth gauge. *Clean* the length of tube below the spinner. Take care not to disturb the alignment. This is important because when the sample is in the magnet there is about 0.5mm clearance between the sample tube and the probe. Any dirt may bridge this gap and prevent the sample spinning, or even damage the probe. Put the sample into any holder in the Sample Changer carousel **except the one at the pick-up point**.

Tell the Spectrometer what NMR work you want it to do

You use the screen, mouse and keyboard to enter information about yourself and the NMR work you want into the computer. The computer places your request at the end of a Job Queue and the requests are then dealt with in order by the spectrometer. All data collected on the AV300 is stored on the computer's disk, and transferred automatically to an archive store. So that you can find your work in the archive, the system generates a data set name that identifies three things: you, your sample, and the nucleus that was observed. The computer requests information about all these things in turn. If you find the screen blank when you want to make an entry, move the mouse to switch it on.

To start making an entry into the Job Queue, click on the button labelled START in the window in the middle of the screen.

Window 1: The first question the computer asks is that you identify yourself by entering your User Code. This is the three-letter Analytical Services User Code that you received when you registered with the NMR Service. You should type all three letters into the box and then click the 'Continue' button. **Do not use a User Code belonging to someone else**, because User Codes are used to identify the budget from which charges for the NMR Service are taken. In addition, you may later find it difficult to find your data in the archive.

Window 2: Here, the computer asks you to enter your Sample Code. The computer will change all lowercase letters to uppercase, and will not allow you to use any punctuation characters such as / : ( ] '. **Do not include your initials or the nucleus** in a Sample Code, since the computer will automatically attach both of these to the Sample Code to make the data set name. **Do not use Sample Codes more than once,** since a new data set will automatically destroy an earlier one with the same name. You *may* use a Sample Code again if you are observing a different nucleus, since the nucleus is made part of the data set name, or if you select a different observe region for ^{19}F and ^{31}P. However, neither the comment text nor the solvent you use is part of the data set name, so if you record spectra of a sample in different solvents, each one should have a new Sample Code.

The computer will detect and prevent some mistakes in a Sample Code entry, but not all. You are strongly recommended to use a simple numbering system, e.g. 0 to 999, with resubmissions of the 'same' sample designated by a letter suffix, A to Z. You should

be careful to avoid confusion between letter 'I' and figure '1', letter 'O' and figure '0', letter 'S' and figure '5', and letter 'Z' and figure '2'. Type your Sample Code into the box and click the 'Continue' button.

Window 3: Type into the box the number marked on the carousel holder where you have put your sample. Click on the 'Continue' button to move on to the next stage.

Window 4: This window shows you a list of the solvents that the system knows about and asks you to select one of them. Click on the solvent that you have used and then click the 'Continue' button. If you wish to make frequent use of a solvent that is not listed, **please tell me**, so that it can be added to the list.

Window 5: The available NMR experiments are displayed. Click on the experiment that you wish to do and then click on the 'Continue' button. If you wish to use an experiment that is not listed, **please tell me**, so that it can be added to the list.

Some work is offered as both 'Day' and 'Night' experiments. 'Day' experiments are limited to less than 20 minutes. If you select an experiment with the word 'Night' in its name, it will not be started until the first opportunity after 18:30 and it will then be allowed to run for up to three hours. If the required S/N (currently 100) is reached before three hours then the experiment will be stopped and printed out. After three hours, even if the S/N is not good enough, the experiment will be stopped and printed out. If the result is unsatisfactory then you should pass it on to the AV400 for more work.

If there is already an experiment running at 18:30, then this will be allowed to finish before the first 'Night' experiment is started. If there are a lot of 'Night' jobs queued and some remain to be done at 07:00, then they will be held during the day until 18:30 before starting. Saturday and Sunday all day count as night-time (so Friday 'night' lasts until 07:00 Monday).

Remember that ^{13}C is approximately *six thousand times* less sensitive than ^{1}H, so to get a spectrum you need more material. You should only enter a ^{13}C request for a 'Day' experiment if know you have at least 0.1 millimole of material dissolved in 0.7ml of solvent.

Window 6: If you would like a comment to be printed at the top of your spectrum, type it in this box. Any characters are allowed. If you don't want a comment, leave the box empty. Please note that this comment text is **NOT** used by the system to identify your sample or to index the data. Click on the 'Continue' button to move on to the next stage.

Window 7: A summary of everything that you have entered so far is displayed. **Please check it!** If you want to change or correct anything, click the 'Go Back' button. Each window has a 'Go Back' button, and will reappear in turn. You may re-enter anything that you wish to change. When everything is the way you want it, click the 'Finish' button. The starting window will reappear.

You have now entered everything required for your entry in the Job Queue. The computer will pass this information to the spectrometer. You must now wait for your sample to take its turn. The entire data collection, processing and plotting sequence is completely automatic. A simple ^{1}H spectrum takes about 4 minutes to complete after the sample has gone into the magnet. After the work is completed, the plot will appear on the printer and your sample will be returned to the Sample Changer. Please **wait** until the Sample Changer arm has stopped moving before removing your sample.

Take your samples and spectra away after the work is done. Do not leave your samples in the Sample Changer. Replace spinners in the blue box.

Spinners are very costly (ca. £100 each) and must NOT be taken away!

Please do **NOT** do your reprocessing on the spectrometer. It is a hazard to continuous spectrometer operation, and it blocks others from entering their NMR work. Reprocessing may be done on any computer, but there is only one computer that runs the spectrometer. ACD software is available at no cost for reprocessing on your own computer, and NMR data is available all the time, from anywhere, from the archive.

It is not unknown for the system to stop unexpectedly, or misbehave in some way. Please do not try to fix it yourself. Tell me immediately. If anything goes wrong while you are using the system, please try to remember what you were doing, and tell me about what happened. It may be possible to alter the settings, the entry program or these instructions to avoid the problem occurring in the future.

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Your User Code is: _ _ _ _ _ _ _ _ _ _ _ _ _