FIGARO User Manual v20 – 13/09/2019

Instrument Control

Nomad has 3 main sections: “Hardware”, “Settings” and “Execution”. You should principally use “Execution” during your experiment with only the exceptions below in “Settings” and “Hardware”.

Hardware. To reconnect the Keyence laser open the menu “DIGIPORT_1”, right click on “LKG2152_H [ch.1]” and select “Connect”.

Settings. Here you find controls for individual actions to operate the instrument. You may also define the zero reference point for the sample height motor SHT1 and the sample angle motor SAN. More involved sequences of commands should be executed from the “Execution”.

Execution. This section of Nomad allows you to control the instrument using sequences of commands. Your local contact will show you how to configure appropriate sequences for your experiment. Common commands for a given experiment can be placed in the “Favourites” menu.

Scripts. You may create scripts to set up sequences of commands. To create a new script, click on Command → New Editor and then double-click or drag icons into the editor and modify the parameters. Then double-click on the name at the top, enter a name and press enter. Then click on Command → Save Editor and it will appear in the list of scripts. To run a script or sequence of commands, double-click or drag the appropriate icons into the launch pad and then click the Play button at the bottom right of the screen. Horizontal and vertical blue scroll bars indicate progress. Further actions or scripts may be added to the launch pad while it is running. To empty the launch pad after it has stopped click on Command → Rewind Command List then Editor → Erase Launch Pad. To create a script that is similar to an existing one, modify the parameters accordingly, double-click on the name at the top, enter a name and press enter. Then click on Command → Save Editor and it will appear as an additional editor in the list.

Digital spy. You can see the instrument configuration remotely at http://nomad.ill.eu/?figaro using Firefox or Chrome (not Internet Explorer) – its appearance should improve in the next update.

Re-starting NOMAD. If the software crashes or gets very slow you can close all the windows and then kill the server. First, click on a terminal (small icon of a computer) and enter “cmo stop ns”. Wait for the appropriate prompt, close the terminal and click on the light blue NOMAD icon. The server takes 30–40 seconds to start, so give it time. At this point the software will work but the detector image will be missing, so click on the small detector image on the right screen. It is safest next to run a ‘Theta’ file that has the frame overlap mirror and chopper setting options selected: simply save one of your existing ‘Theta’ files with both options ticked and then run it. It is a good practice to re-start the NoMad server at the beginning of each experiment. The program tends to become instable when running for long.

Starting an Experiment

Good practice is to level the sample table using a spirit level, this should correspond to a SAN offset of 0.075. PHI should be aligned with the sample holder and reset to 0. The SHT2 motor should *never* be reset and its high stop is at 16.70. To be on the centre of rotation of the SAN goniometer the interface should be 186 mm above the baseplate. This corresponds to an SHT1 offset of –245 (for SHT2 = 0, assuming it has not been misconfigured, which can be checked by hitting the high stop). The best guess for the SHT1 offset is “height above 10-mm baseplate minus 431”. “Theta” is configured to point at the sample even if it is not placed in the centre of the sample table. The horizontal sample offset in “Theta” should be negative if the sample is more towards the reactor.

Starting a Solid/Liquid Interface Experiment involving a Horizontal Sample Offset

(1) Configure ‘Theta’ files with the horizontal offset = –121.
(2) Make 'angle1' and 'angle2' editors with 'Theta' and 'san-relative = theta' (refl up) or ‘= –theta’ (refl. down) or use the standard macro Userfiles -> Standard-files -> 2AnglesSL
(3) Hit the high stop of SHT2 and redefine it as 16.70 if different.
(4) Set the SHT1 offset as 'height above 10-mm baseplate minus 431' (last known offset = –250).
(5) Configure the sample changer table with every cell at SAN = -0.62 (refl. up) or 0.62 (refl. down) and SHT2 = 0. The str positions should be 460.4, 375.4, 290.4, 205.4, 120.4 and 35.4 for Position 1-6, respectively.
(6) Level the table (not the sample holder!) using a spirit level and set the SAN to 0 (san offset should be 0.075).
(7) If you are reflecting down the following steps 8a-b can be done by using the Macro in Userfiles -> Standard-files -> AligSLInitial.
(8) Run ‘sample-changer’, run a ‘Theta’ file with the frame overlap mirror and chopper options selected, and run ‘san-relative = –theta’.
(9) Do a broad SHT1 scan from –4 to 1, refine it and reset SHT1 to 0 at the maximum (refl. up) or 86 to 91, Reset SHT1 to 87.265 at the maximum (refl. down).
(10) The following step 10a can be done by executing the macro Userfiles -> Standard-files -> AligSLDown
(10a) Drive DAN = –0.62 and S4B = 19 and do a calsan in the range 0.25 to 0.55 (refl. up) or DAN = 0.62 and S4T = 19 and do a calsan in the range –0.55 to –0.25 (refl. down); refine if necessary.
(11) Calsan will give you the SAN and SHT2 values for the sample changer table and drive there.
(12) If you don’t use the standard align macro from above. You can then make an ‘align’ macro so that future alignments are easy: use the SHT2 scan option with the range 0.4 to 2.8 (refl. up) or –2.8 to –0.4 (refl. down).

Important note. You must always run 'sample-changer' before each 'angle' script because of the SAN-relative movement, e.g. ‘sample-changer2, angle1, count, sample-changer2, angle 2, count’ not ‘sample-changer2, angle1, count, angle 2, count’.
Safe detector count rates for S/L experiments are 12k for angle 1 and 14k for angle 2 while using 20 A FO.

Data Reduction

Starting LAMP. Go to a different workspace and click on the “LAMP” icon at the bottom of the screen. A Terminal opens, type in “lamp”. When the lamp window arrives, enter the username “X” then press enter or click on “OK”. Select Lamp/Layout → ‘Extend to classical lamp’, which will allow you to enter commands at the bottom-right. Near the top left, click on the ‘data’ box and then ‘FIGARO’ and then ‘online’.

Starting COSMOS. Select Tools → ‘Reflectometry with cosmos’ and the COSMOS window will appear. Your local contact will set up the appropriate directories in the IO tab and explain the Normalisation and Calculation features. Once you write data into workspaces you may view them in “Superplot” either by clicking on “Superplot” on the right side of the LAMP window or selecting Tools → ‘Overplotting (Superplot)’. Type ‘det’ in the Angle fields for S/L experiments and nothing for free liquid experiments.

Visualisation macros. The commands “w2=total(w1,1) will provide a projection of I,y in workbook 2 and “w3=total(w1,2)” will provide a projection of I,lambda in workbook 3. These commands may help in the determination of appropriate parameters to use in the data reduction software.

Printing. The instrument computer is usually configured so that if you press ‘print’ in LAMP then a hard copy is sent to the IN5 printer. If this option is not working you can configure this option

yourself by selecting Options → 'Plot' in the main window and entering 'lc1-in5' in the bottom box.

Computers

There are three computers in the FIGARO cabin. User files may be stored in the appropriate directory on these computer but please *do not* leave any raw data as the file size is too large; any raw data found will be deleted as soon as possible.

Webcam

Type "figarocam2" in the address bar of Firefox on the Windows 7 PC, click on the link "Setup", log in with the user name "figaro" and the password "figfig", and then click on the link "View Video".

STARTING OLD ADSORPTION TROUGH EXPTS ON FIGARO

1. Open two file managers, and move the contents of both *nomadSettings* → *Theta* and *nomadScans* to an appropriate directory in *user-files* → *2018* the previous local contact may have done this already.
2. Log into the experiment at the top right of the main NOMAD window.
3. Settings → Axis → SHT1 → More Options: change offset to –248.
4. Turn off the beam, open the interlock and clear the sample area; drive SHT1 to –115 and SAN to horizontal to facilitate the exchange of sample environments.
5. Remove the previous equipment, install the troughs, level SAN and PHI, and change their set-points to zero.
6. Position the Keyence laser manually so that the beam goes appropriately through a quartz window.
7. Switch on the Keyence device and the anti-vibration table.
8. Hardware → DIGIPORT_1 → LKG152_H [ch. 1] → right click: reconnect (if red).
9. Settings → Sample Environment: disconnect any unused items (e.g. pumps and baths).
10. Settings → Axis → SHT2 → high stop (can enter 20) and redefine as 16.70 if necessary.
11. Settings → Sample Environment → Setting → Trough → More Options: enter the appropriate STR values with a height target of 0.000 and a tolerance of 0.030 for all six positions (no need to play anything); the troughs are 98 mm apart; for trough 6 STR = 4.5 (with the windows on the right).
12. Drive STR to a trough position (or run a trough number without the alignment ticked).
13. Make 'angle1-d2o', 'angle2-acmw' and 'angle2' Theta files with the appropriate slits and horizontal sample offset (–60 if the small troughs on the right side are used), use a vertical sample offset of –6.4 and an S4B beam block of 6 and S4T out at 19; make corresponding 'angle1-db' and 'angle2-db' files for the direct beams with S4B at 19.
14. Pour 50 mL of D_2O into a large trough or 25 ml into a small trough and play **Userfiles -> Standard-files -> AligFreeLiquid. If you don't want to use the standard macro then play** 'angle1', then scan SHT1, with the range –3 to 3 and 31 scans of 0.5 s driving to the optimum, then change the SHT1 set-point to 0, Then, do an SHT2 scan with a range of –1.2 to 1.2 with 31 points and scans of 2 s.
15. Zero the Keyence laser at the optimum of SHT2.
16. Remove D2O and run the macro **Userfiles -> Standard-files -> DirectBeamsFreeLiquid**
    a. If you don't want to use the standard macro then: Make a 'sample change' Theta file with STR = 4.5 and SHT1 = –230 running in parallel.
    b. Run 'angle1-direct-beam'.
    c. Configure 'favorites' in Execution by dragging 'theta', 'trough', 'count' and 'forloop'.

d. To start the attenuator drive ATW to any value < 5 and then turn OSC on with a range of –21 to 21.
e. Record direct beams (including the angle 1 calibration), and for each angle set SAN = –COA (collimation angle); Make sure the horizontal offset is 0 for the direct beams; the maximum count rate is 5k at angle 1 and 12k at angle 2 for 7% dλ/λ.
f. When the direct beams are finished run 'angle1', then turn OSC off, then ATR = –21, then ATW = 44, then SAN = 0, then SHT 2 = 0 (again the order is important).
g. Move the direct beam Theta files from *nomadSettings* → *Theta* to an appropriate directory in *user-files* → *2018*.

Always run *angle1* or *angle2* then *trough* (with ticked alignment) before each *count* !!

Safe detector count rates free liquids experiments are 5k for angle 1 D2O, 9k for angle1 ACMW and 14k for angle 2.

STARTING LANGMUIR TROUGH EXPTS ON FIGARO

1. Open two file managers, and move the contents of both *nomadSettings* → *Theta* and *nomadScans* to an appropriate directory in *user-files* → *2018*; the previous local contact may have done this already.
2. Log into the experiment at the top right of the main NOMAD window.
3. Settings → Axis → SHT1 → More Options: change offset to –245.
4. Turn off the beam, open the interlock and clear the sample area; drive SHT1 to –115 and SAN to horizontal to facilitate the exchange of sample environments.
5. Remove the previous equipment, install the Langmuir trough, level SAN and PHI, and change their set-points to zero.
6. Connect the Langmuir trough with the sensor in its final position, calibrate the area, speed and pressure, and select 'external control' on the software.
7. Drive STR to 250, then position the Keyence laser manually so that the beam goes appropriately through the quartz window.
8. Switch on the Keyence device and the anti-vibration table.
9. Hardware → DIGIPORT_1 → LKG152_H [ch. 1] → right click: reconnect (if red).
10. Settings → Sample Environment: disconnect any unused items (e.g. pumps and baths) and connect the Langmuir trough and bath if wanted.
11. Settings → Axis → SHT2 → high stop (can enter 20) and redefine as 16.70 if necessary.
12. Settings → Sample Environment → Setting → Trough → More Options: enter STR = 250 with a height target of 0.000 and a tolerance of 0.030 for all six positions (no need to play anything).
13. Make 'angle1-d2o', 'angle1-acmw' and 'angle2' Theta files with the appropriate slits, use a vertical sample offset of –8 and an S4B beam block of 6 and S4T out at 19; make corresponding 'angle1-db' and 'angle2-db' files for the direct beams with S4B at 19.
14. Make a 'sample change' Theta file with SHT1 = –230.
15. Pour 500 mL of D_2O into the large trough or 125 ml into the medium insert or 35 ml in the small insert and play **Userfiles -> Standard-files -> AligFreeLiquid. If you don't want to use the standard macro then play** 'angle1', then scan SHT1, with the range –4

to 4 and 41 scans of 0.5 s driving to the optimum, then change the SHT1 set-point to 0, Then, do an SHT2 scan with a range of –2 to 2 with 31 points and scans of 2 s.

16. Zero the Keyence laser at the optimum of SHT2.
17. Remove D2O and run the macro **Userfiles -> Standard-files -> DirectBeamsFreeLiquid**
    a. If you don't want to use the standard macro then: Make a 'sample change' Theta file with STR = 4.5 and SHT1 = –230 running in parallel.
    b. Run 'angle1-direct-beam'.
    c. Configure 'favorites' in Execution by dragging 'theta', 'trough', 'count' and 'forloop'.
    d. To start the attenuator drive ATW to any value < 5 and then turn OSC on with a range of –21 to 21.
    e. Record direct beams (including the angle 1 calibration), and for each angle set SAN = –COA (collimation angle); Make sure the horizontal offset is 0 in the direct beams; the maximum count rate is 5k at angle 1 and 12k at angle 2 for 7% dλ/λ.
    f. When the direct beams are finished run 'angle1', then turn OSC off, then ATR = –21, then ATW = 44, then SAN = 0, then SHT 2 = 0 (again the order is important).
    g. Move the direct beam Theta files from *nomadSettings* → *Theta* to an appropriate directory in *user-files* → *2018*.

Always run *angle1* or *angle2* then *trough* (with ticked alignment) before each *count* !!

Safe detector count rates free liquids experiments are 5k for angle 1 D2O, 9k for angle1 ACMW and 14k for angle 2.

FIGARO Troubleshooting – 13/09/2019

Choppers

The choppers can stop if, for example, there is a problem with the vacuum or cooling systems. At this point the interlock to the FIGARO sample area will fail. To restart the choppers, first shut down and restart the choppers computer (there is no password). Then open the "NSC019" software on the desktop. Once it has loaded, click on the 'calibrate' button towards the right. The choppers will then spin slowly in order to determine the phases of the pickups in a procedure that can take up to 5 minutes, so give it time. If after 10 minutes there is no progress you can restart the computer and restart the calibration procedure. Once the 4 numbers for the speeds of each chopper at the top go yellow you can control them. Change the speed of chopper 1 to '200' and click on "accept" below. Then for each of choppers 2, 3 and 4 click on "AS – S" and "accept". Once the actual speeds of each chopper (the second row of numbers from the top) are 200 then you can take over from NOMAD. However, to do that you will need to restart NOMAD, the procedure of which is described above. If you re-boot the choppers PC please note that there is no password: just hit enter when the log in box appears (username = "figarochopper"; password = blank).

Power Cut

If there is a power cut, the electronic racks for the motors may need to be reset: just turn all switches off and then on again. The zero positions of the motors of slits 2, 3 and 4 will then be lost and need to be redefined. If the instrument was not moving (most likely), the values can be reset in "Settings" according to the last known configuration, which can be determined using the data parameters of the last recorded data file in LAMP – or from an equivalent file if the last file did not save. If the instrument was moving, you will need to drive the motors to hit their limit switches and redefine them as: S2T (low) = –4.228, S2B (*high*) = 13.526, S2L (low) = –4.790,

S2R (low) = –3.551, S3T (low) = –8.291, S3B (low) = –9.058, S3L (low) = –5.295, S3R (low) = –4.694, S4T (low) = –1.657 and S4B (low) = –2.975; please note that all of these are the low limits except for S2B which is the high limit. Also, if the slit 4 beam stop afterwards does not seem correctly positioned at low Thetas simply try some different values and then update the corresponding Theta files accordingly. After these steps have been carried out, you can check that the count rate for a known configuration (e.g. angle 1 of a sample with D_2O) is not unreasonable, bearing in mind that adsorption at interfaces can decrease the count rate compared with pure D_2O.

Interlock

Please close the gate to the sample area gently to prevent the catch from breaking. If the procedure for the interlock is operated in the wrong order it can get stuck. If this happens, stay very calm and do the following steps slowly: (1) key to vertical, (2) press green door button, (3) wait for closure, (4) go through whole opening procedure in the correct order, and (5) go through whole closing procedure in the correct order. If this does not work and it is out of hours the local contact will need to request out-of-hours support for mechanical services, which is accessible only by him or her calling or visiting the reactor control room.

Out-of-hours support

Local contacts: please do not hesitate to call these services if the instrument is not working properly. The phone numbers are on the intranet. You should not feel bad about disturbing them as they are being paid for the service. It will not cause an investigation and they may be able to advise you efficiently. For mechanical, electronic and software problems that are not described above, they should be the first call to make out-of-hours before contacting the instrument responsible.

Current bugs

- The chopper resolutions 2.1%,4% and 6% are out of order at the moment
- Do not use the 16A FO. The loss in transmission due to its shallow angle equals the gain in chopper transmission for typical settings