PROXIMA 2A Users Guide

- Remote Access
- MXCuBE
- ALBULA
- BeamCheck
- Sample Changer
- Centering Samples
- Characterisation
- Standard Data Collections
- Helical Data Collections
- Raster/Mesh/Grid Scans
- Data Processing
- X-ray Fluorescence Emission
- Energy Scans
- MAD Collections
- Manual Commands
  - Raster/Grid Scans
  - Area scans
  - X-ray Centering
  - (Annealing)
- Sample Changer Errors
  - Missing Samples
  - Collisions
- Before You Leave PX2-A

Please note: Recent modifications are marked in **MAGENTA**

PROXIMA 2A - Remote Access for Users

- Before the remote access session (>1 week):
  - Obtain your SUNSET project number and password (e.g. 20180123, wDvg2ke7Gt)
  - Ask the BL staff to contact the SOLEIL IT group to permit remote access for your project number
  - Test the connection to SOLEIL
    - Use a « decent » network connection (e.g. ADSL, not modem)
    - Download NoMachine
    - Screen resolution 2560 x 1440 (MXCuBE is optimised for this resolution)
    - Mouse with Left-Right buttons and a Scroll-wheel

- Start NoMachine
  - Check the connection parameters (Right-hand click & Edit Connection)
    - Name = PX2A (for example)
    - Protocol = NX
    - Host = nx-vip.synchrotron-soleil.fr
    - Port 4000

  - Open **Custom** session on **proxima2a-10**
    - **Custom** session on **proxima2a-10**
      - Independent display & mouse, which can run a separate instance of MXCuBE or X-terminal for processing

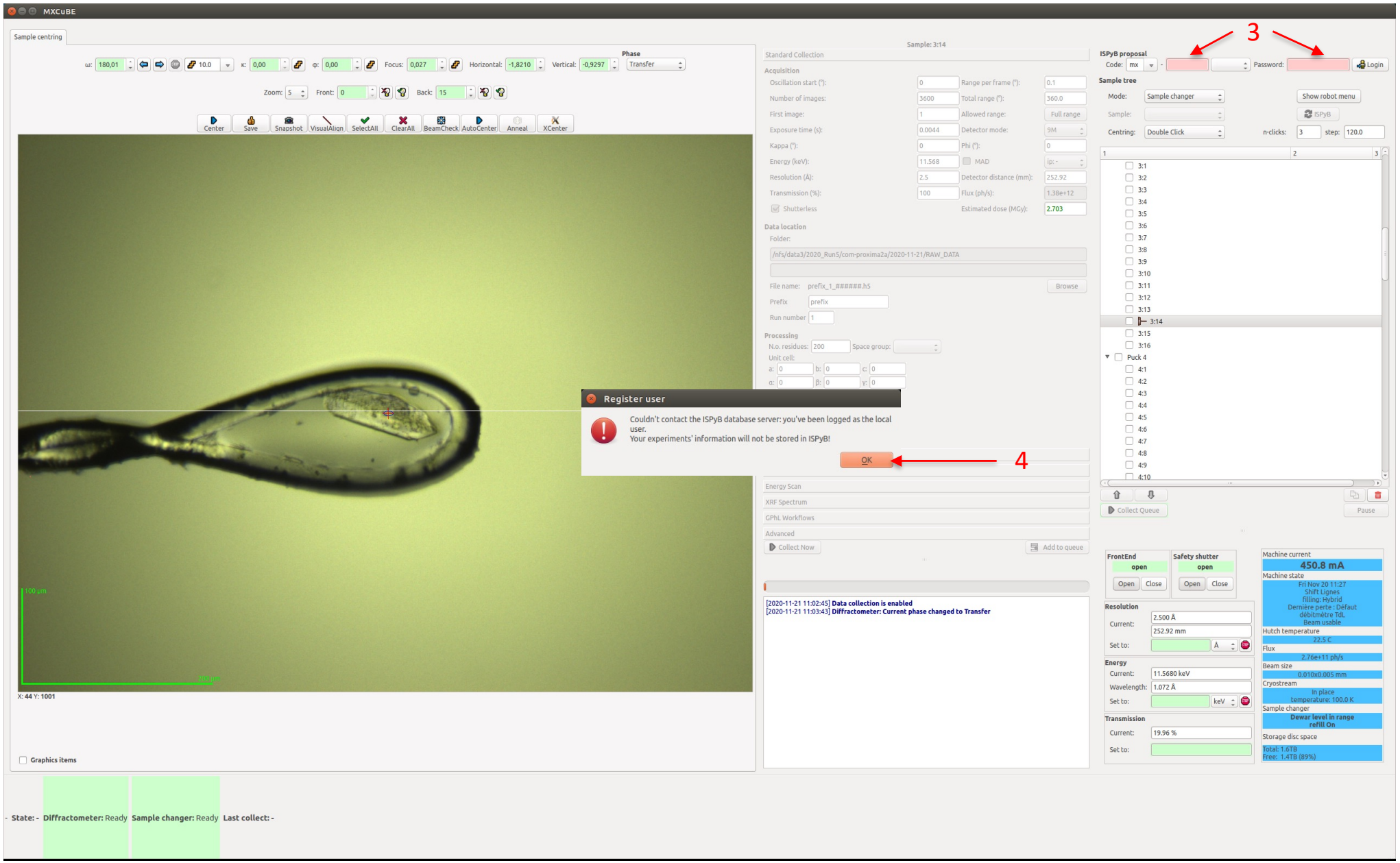
  - Open a terminal window from the side menu/icons
    - **`gnome-terminal &`** [launches gnome terminals]
    - **`mxcube`** [launches MXCuBE to control your experiment]
    - **`albula`** [to display diffraction images]
    - **`firefox –P &`** [to display webcams]
    - **`ssh –X process1`** [to process data on the 280-core server]

Starting MXCUBE

1

To start MXCuBE:

1) Either click on the MXCuBE icon,
2) Or open an X-terminal window (cntl-alt-t)
   - Type ”mxcube”
3) Enter “proposal id”, “password” and/or click “login”
4) If the message window “Couldn’t contact ISPyB database…” appears,
   - click “OK”



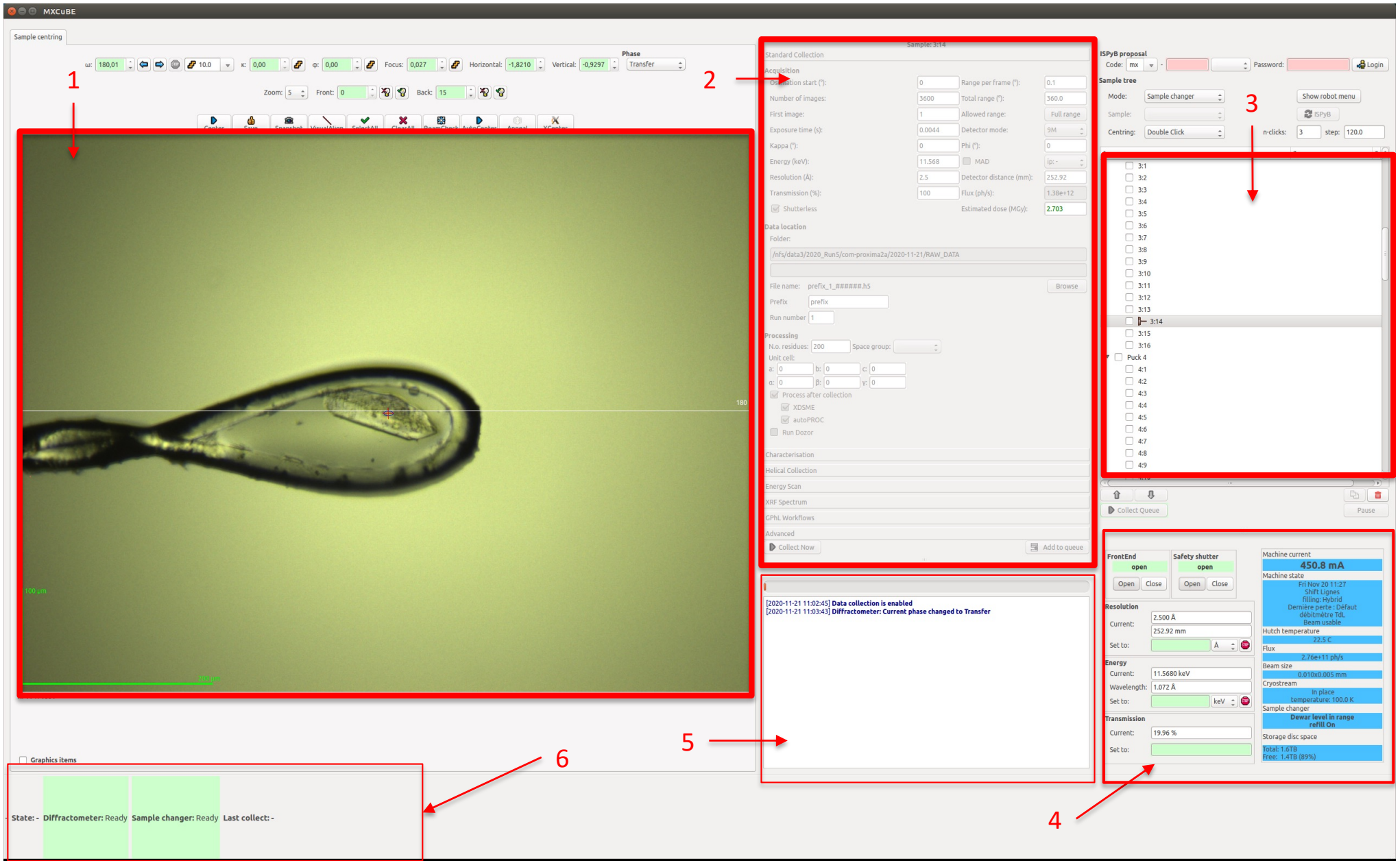
MXCUBE Overview

Description of frames:

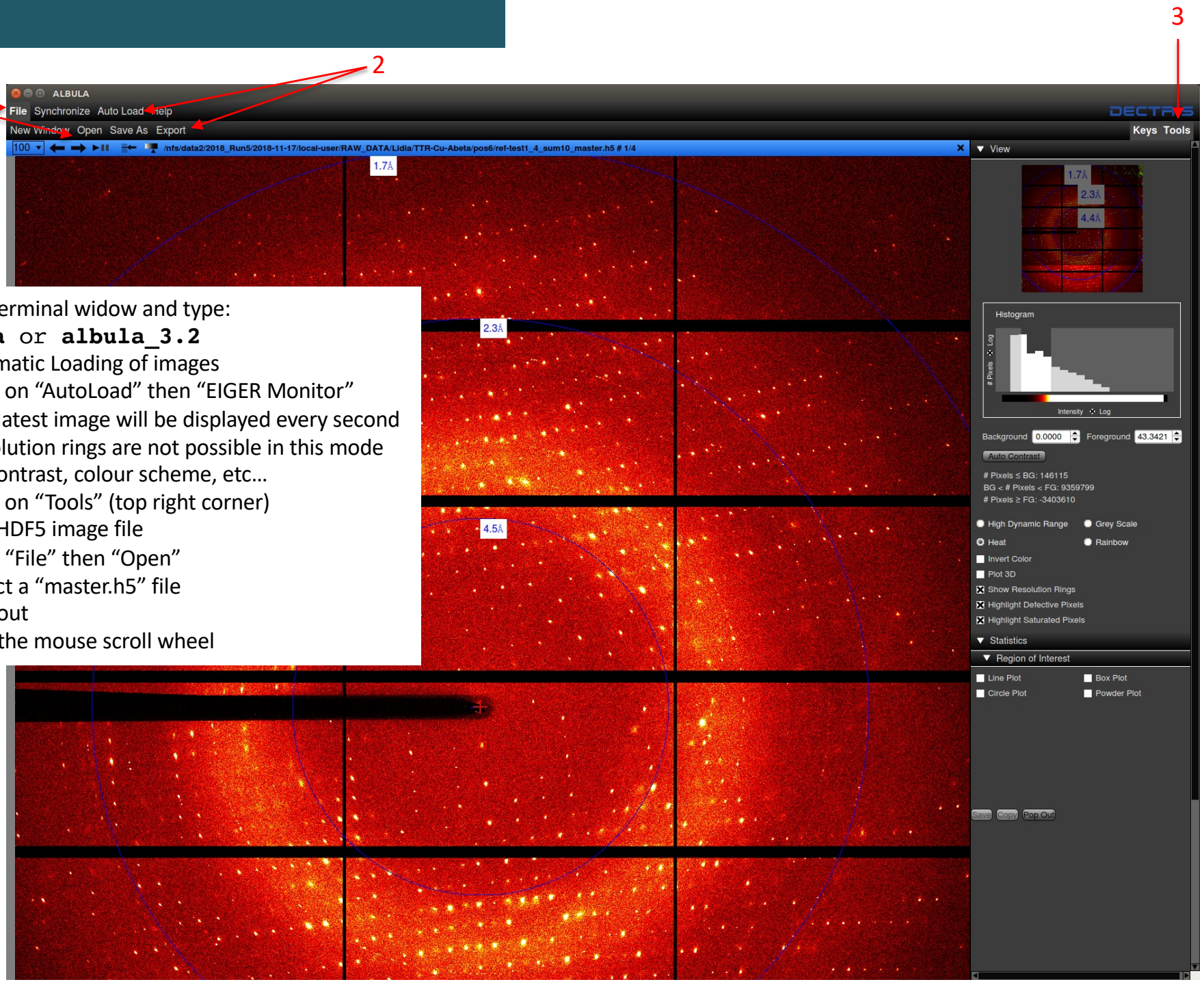
1) Sample Microscope frame
2) Data Collection Methods frame
3) Sample Tree frame:
   - the highlighted line shows puck 3: pin 14 is mounted

Description of frames continued:

4) Beamline parameters, Machine current, CryoStream temperature, Energy, etc…
5) Log messages: please check here for RED error messages
6) State frame for diffractometer, sample changer, etc…



ALBULA



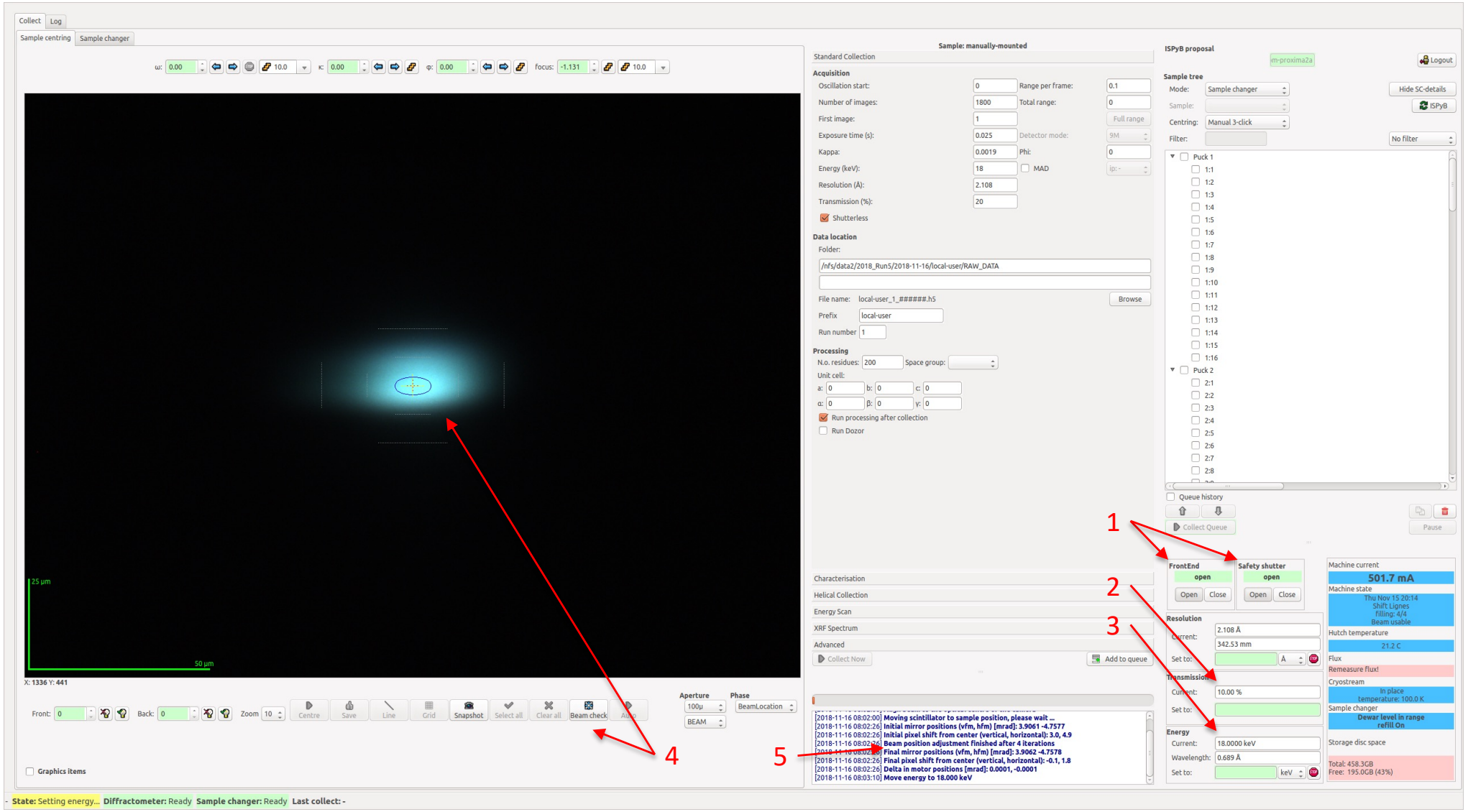
1) Open an X-terminal widow and type:
   `albula` or **`albula_3.2`**
2) To set Automatic Loading of images
   - Click on “AutoLoad” then “EIGER Monitor”
   - The latest image will be displayed every second
   - Resolution rings are not possible in this mode
3) To change contrast, colour scheme, etc…
   - Click on “Tools” (top right corner)
4) To open an HDF5 image file
   - Click “File” then “Open”
   - Select a “master.h5” file
5) To zoom in/out
   - Use the mouse scroll wheel

BeamCheck

BeamCheck should be done:

- At the start of each session
- Repeated every few hours
- After large changes in energy
- Potentially for very small or thin crystals

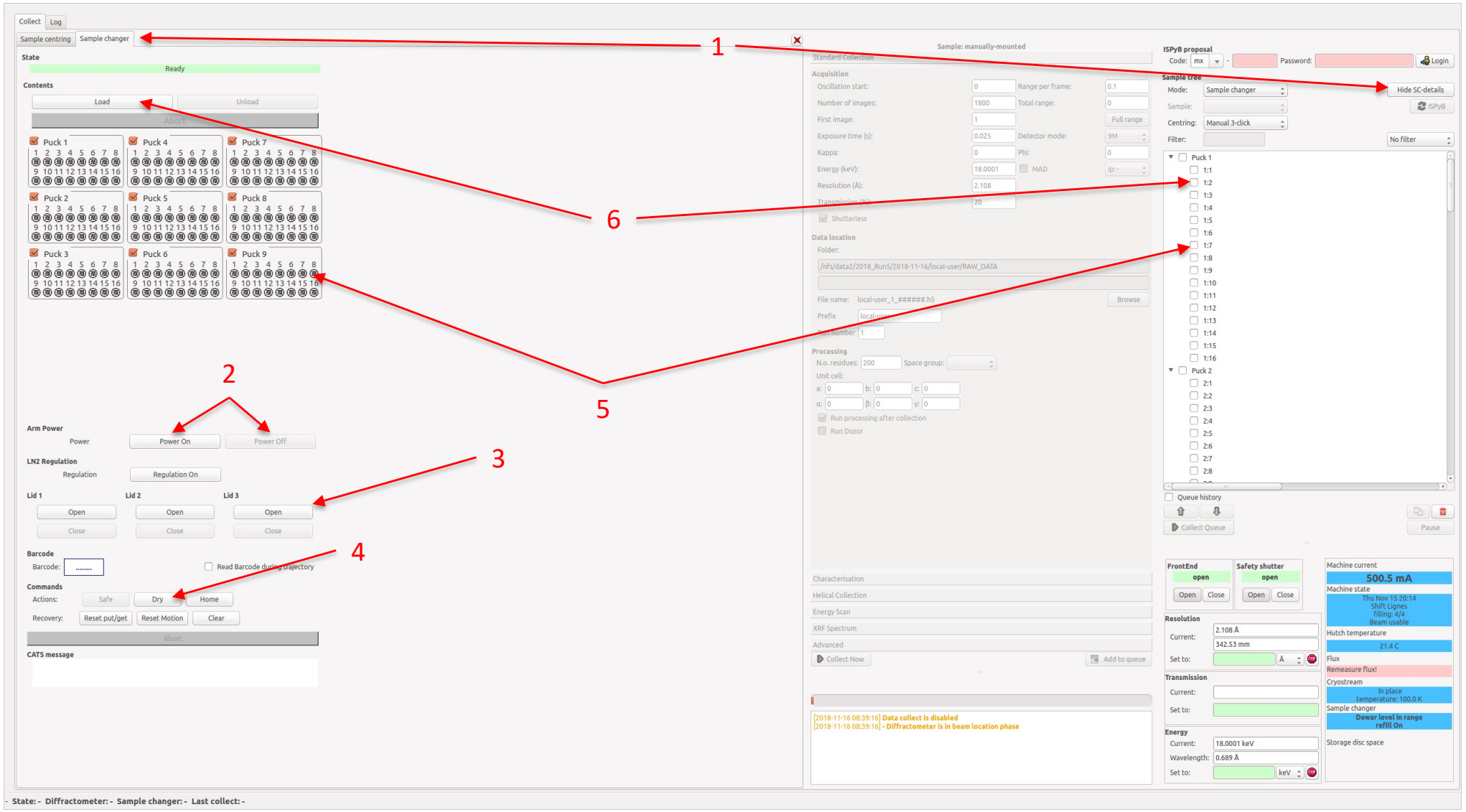
1) Open the “Front End” and “Safety Shutter”
2) Set the Transmission to **<u>100</u>**%
3) Set the Energy (default 12.65 keV)
4) Click on “BeamCheck”
    - Wait several seconds for the scintillator to be put in place (10 s)
    - A blue beam spot will flash up in the sample display
5) The shift in position is displayed in the log frame



Sample Changer

1) Click on “Show/Hide SC-details”, then on “Sample changer” tab
2) Click “Power On/Off” to switch ON/OFF the Robot Arm Power
3) Click on OPEN/CLOSE lids
    - If LID 2does not open, check that Robot Arm Power is ON
4) Click “Dry” to dry the Cryotongs
    - This should done every 8-16 mounts

5) Status of the pucks and pins are displayed in the
    - Sample Tree – highlighted in GREY
    - Sample changer Contents – T-shaped icon
6) Two ways to load/unload pins
    - Sample Tree: Right-Click to MOUNT/UNMOUNT
    - Sample changer-Contents: Click LOAD/UNLOAD

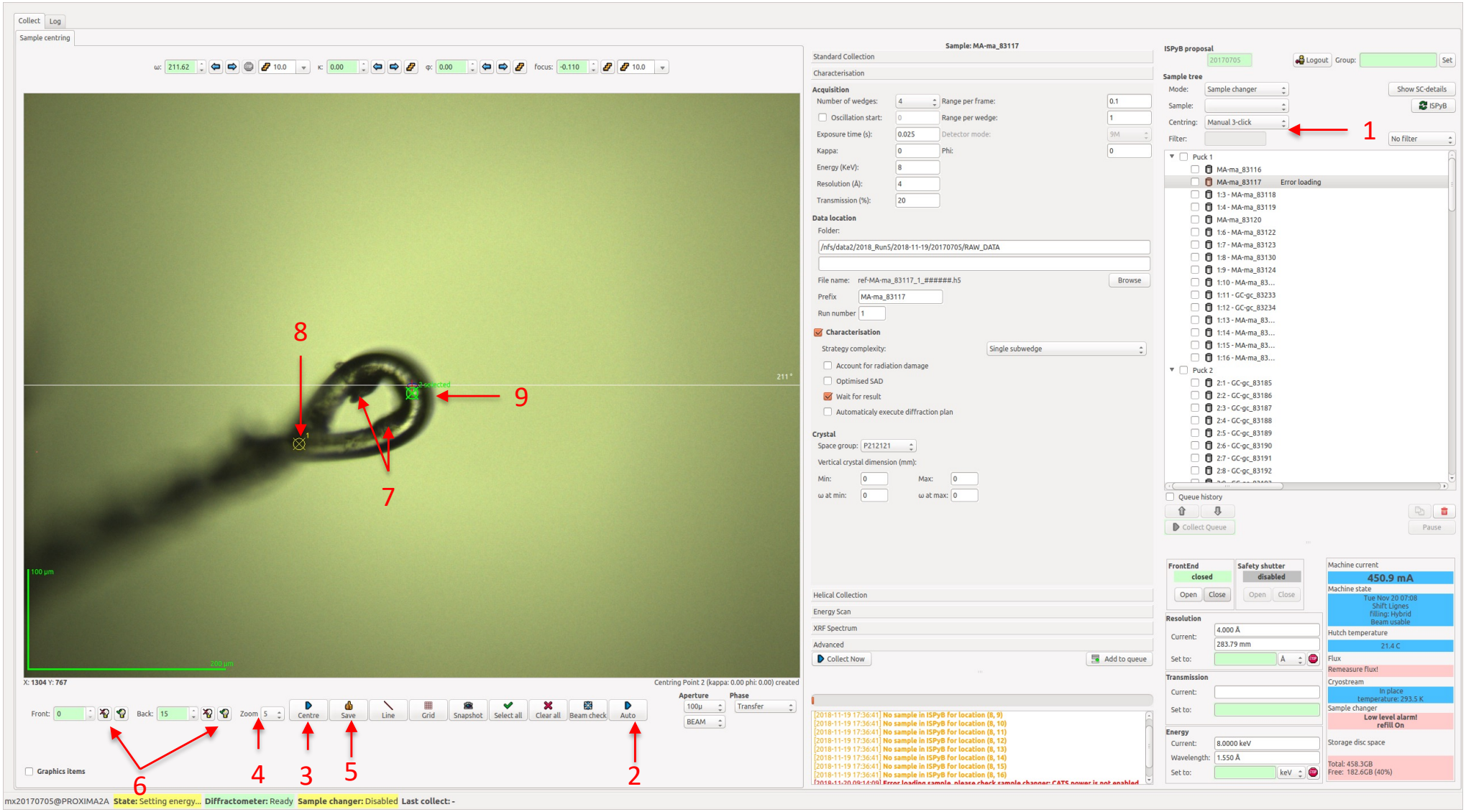


Sample Centering

1) Check “Centring mode” = “3-Click”
2) Click “Auto” to centre the loop
3) Click “Centre” to launch 3-click centring
   - Click on the crystal at 3 omega angles
4) Change the zoom accordingly
5) Click “Save” to record a centred position

Other functionalities:

6) Adjust the front and back-lighting if necessary
7) Double-click anywhere to translate to that position
8) Double-click on a YELLOW centred position to move directly to it
9) A green saved position is “activated” and recorded for the data collection

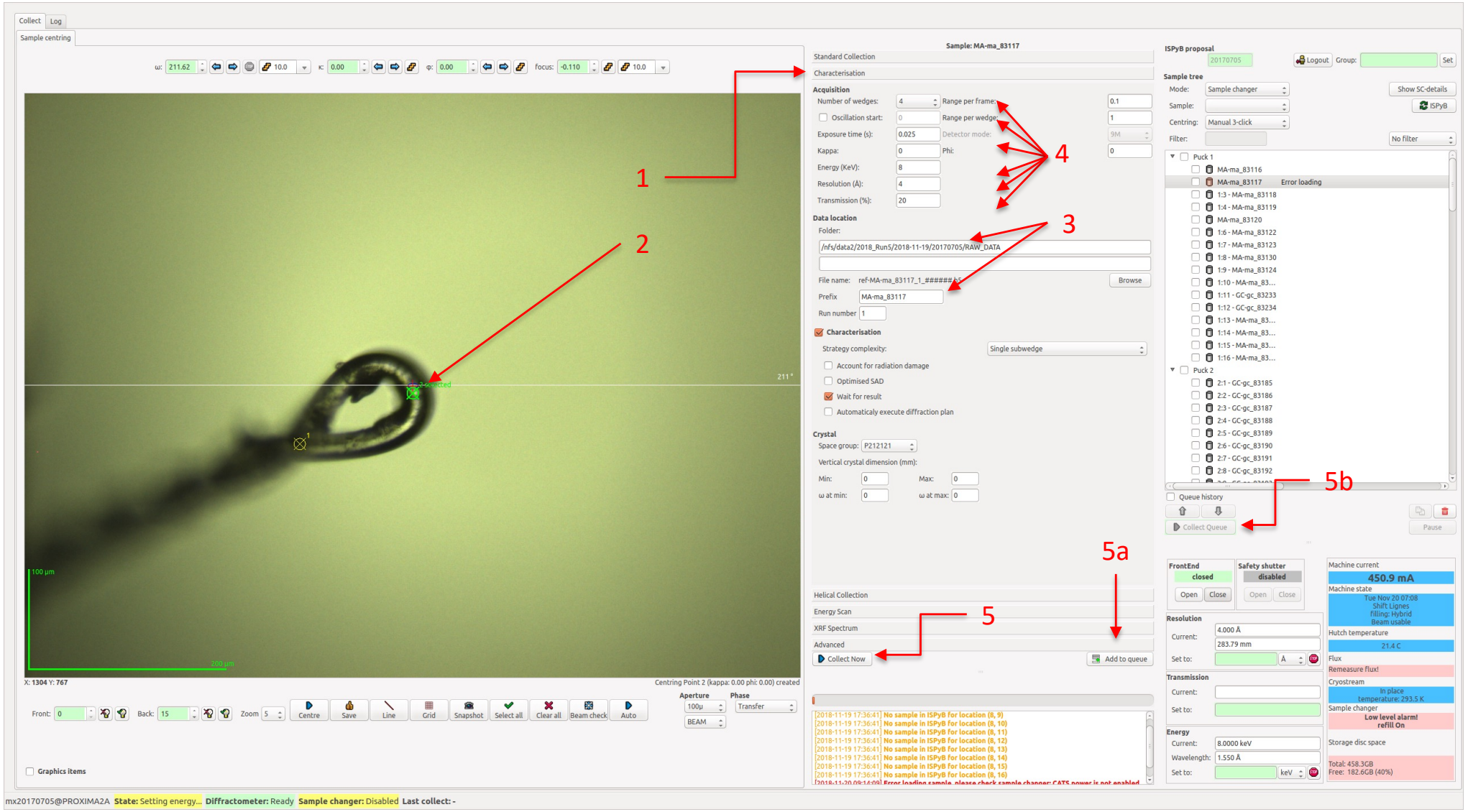


Characterisation

1) Click CHARACTERISATION
2) Click on a saved position
3) Enter directory & prefix
   - **NO blanks or special characters!**

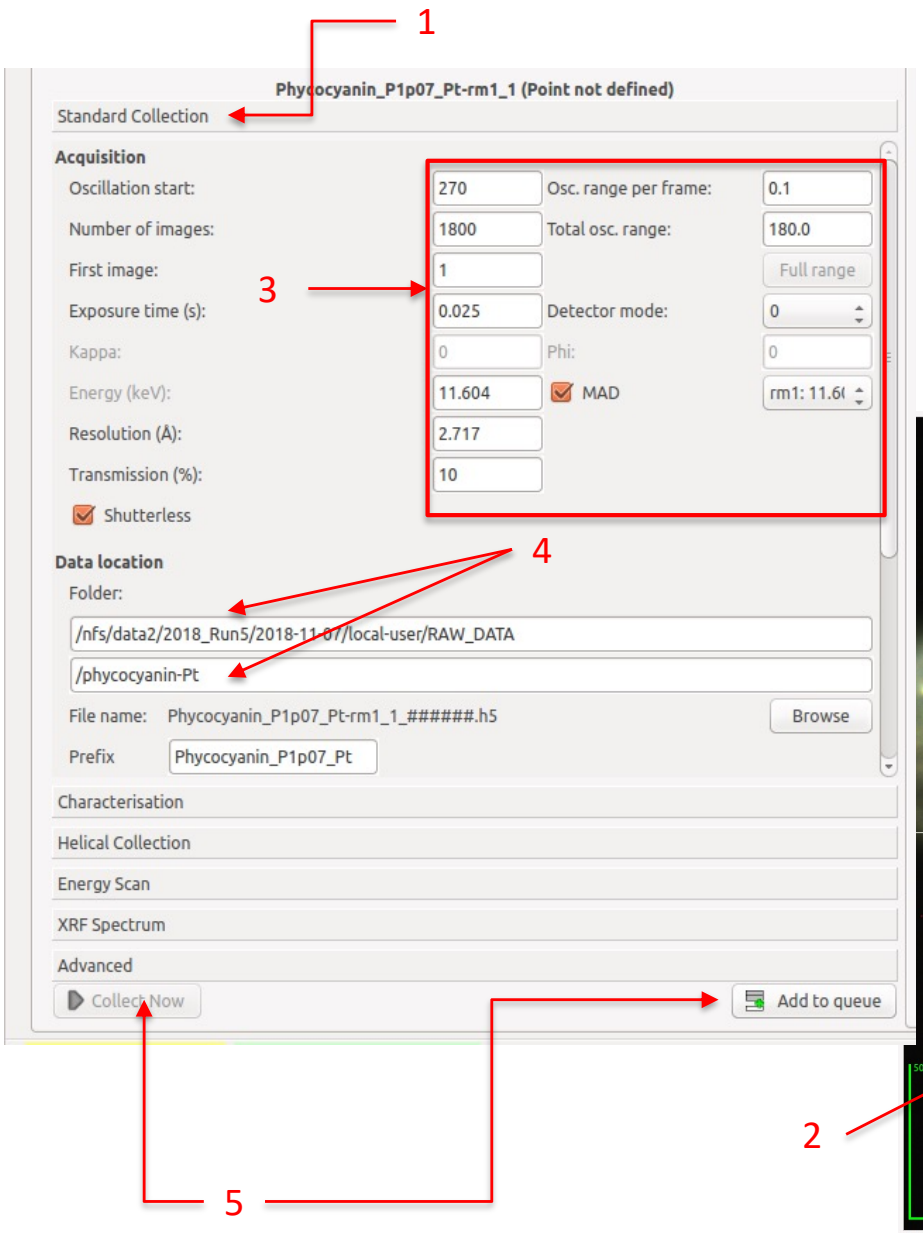
4) Set parameters: default values [range] :
   a) Number of wedges = 4 [1-4]
   b) Oscillation range per frame = 0.1°
   c) Number of images per wedge = 10 [1 – 14400]
   d) Exposure time per image = **0.044 s** [>0.0043 s]
   e) Energy (keV) = 12.650 keV [6 – 18 keV]
   f) Resolution (Å) = depends upon energy & distance
      - Distance = [120 – 1000 mm]
   g) Transmission = **100%** [default, 0.1 - 100%]

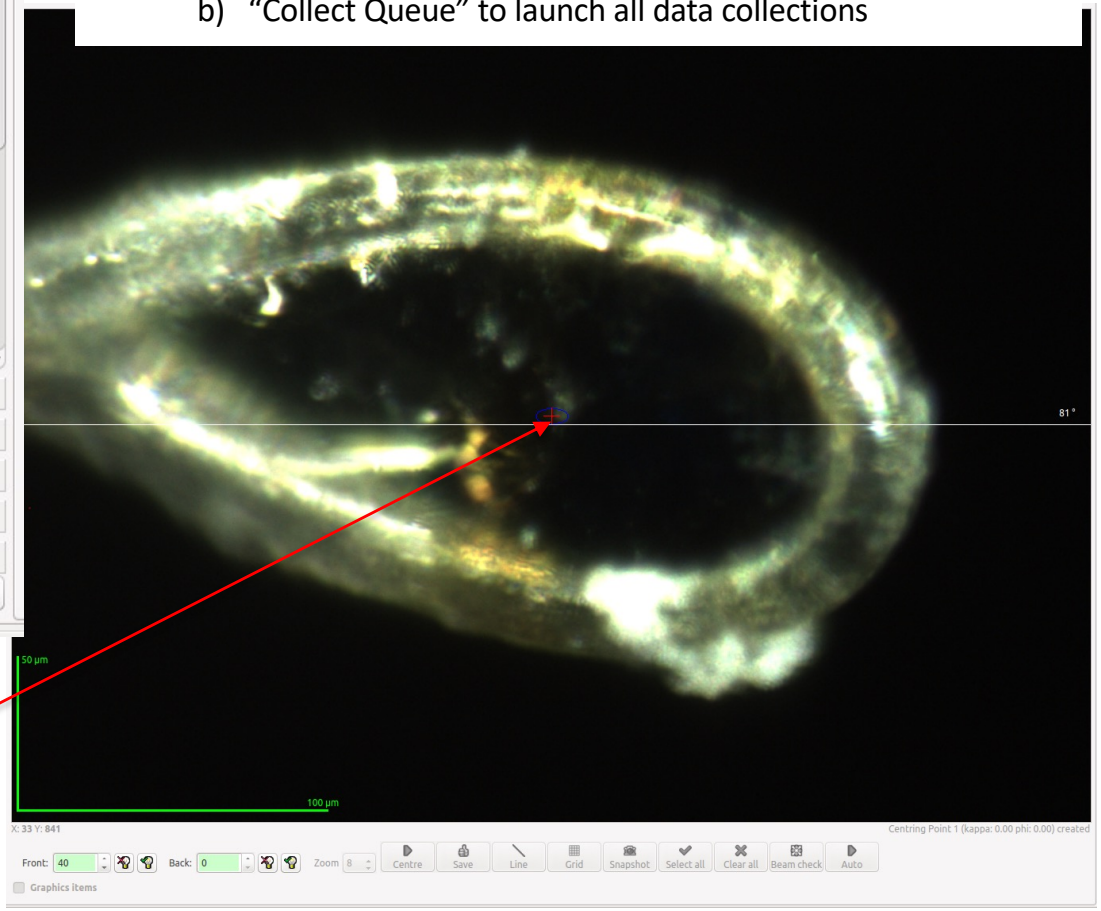
5) Click “Collect Now”, or
   a) “Add to Queue”, if you wish to add another data collection
   b) “Collect Queue” to launch all data collections



Standard Data Collections

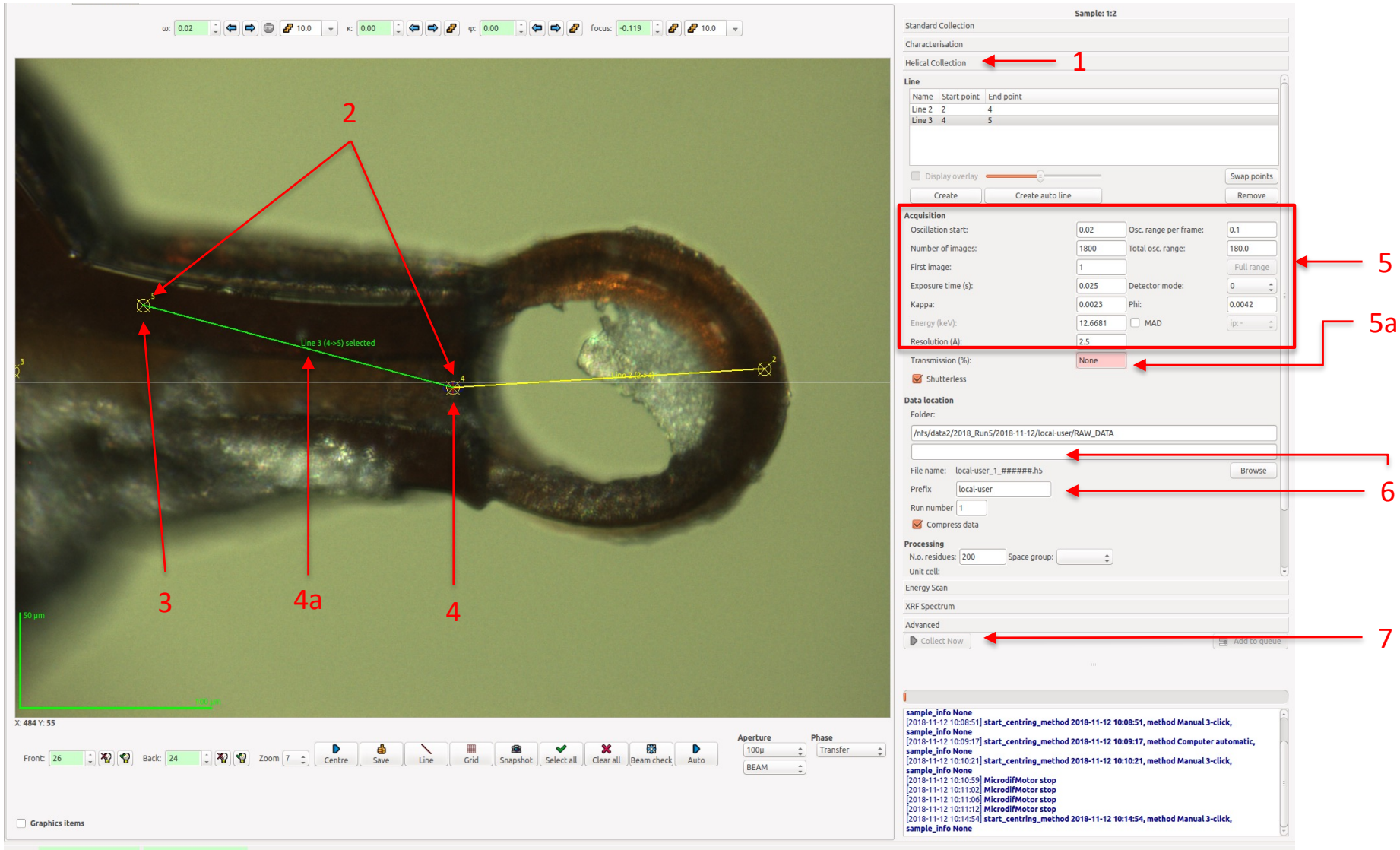
1) Click STANDARD COLLECTION
2) Click on a SAVED centred position
3) Check & set all parameters, typical values [range in brackets] are:
    a) Oscillation range per frame = 0.1° [0.01 - 720°]
    b) Number of images = 3600 [1 – 14400]
    c) Exposure time = **0.044 s** [default, range: >0.0043 s]
    d) Energy (keV) = 12.650 keV [6 – 18 keV]
    e) Resolution (Å) = depends upon energy & distance
        - Distance = [110 – 1000 mm]
    f) Transmission = **100%** [default, range: 0.1 - 100%]
4) Enter directory & prefix
    - NO blanks or special characters!
5) Click “Collect Now”, or
    a) “Add to Queue”, if you wish to add another data collection
    b) “Collect Queue” to launch all data collections





Helical Data Collections

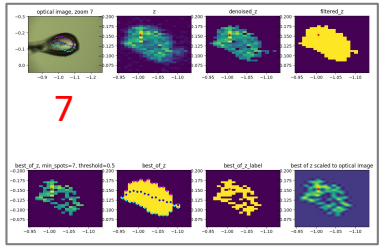
1) Click HELICAL COLLECTION
2) Centre and SAVE TWO positions
3) Click on one SAVED position
4) CNTL-click on a second SAVED position
    a) This will create a GREEN line
5) Check & set all parameters as for Standard Collections
    a) Transmission = **100%** [default]
6) Enter directory & prefix
7) Click “Collect Now”



Grid & Mesh Scans

1) Click ADVANCED
2) Click “Grid”
3) Click-drag the mouse from the Top-Left down to Bottom-Right to select an area to scan

4) Check & set all parameters as for Standard Collections
   - Verify the starting angle
   - Exposure time > 0.010 s [recommended]
5) Enter directory & prefix
6) Click “Collect Now” & wait for the scan to finish
7) The calculation may take a few minutes:
   - A results window will appear
8) Results can be recalculated:
   - raster_scan_analysis.py -h

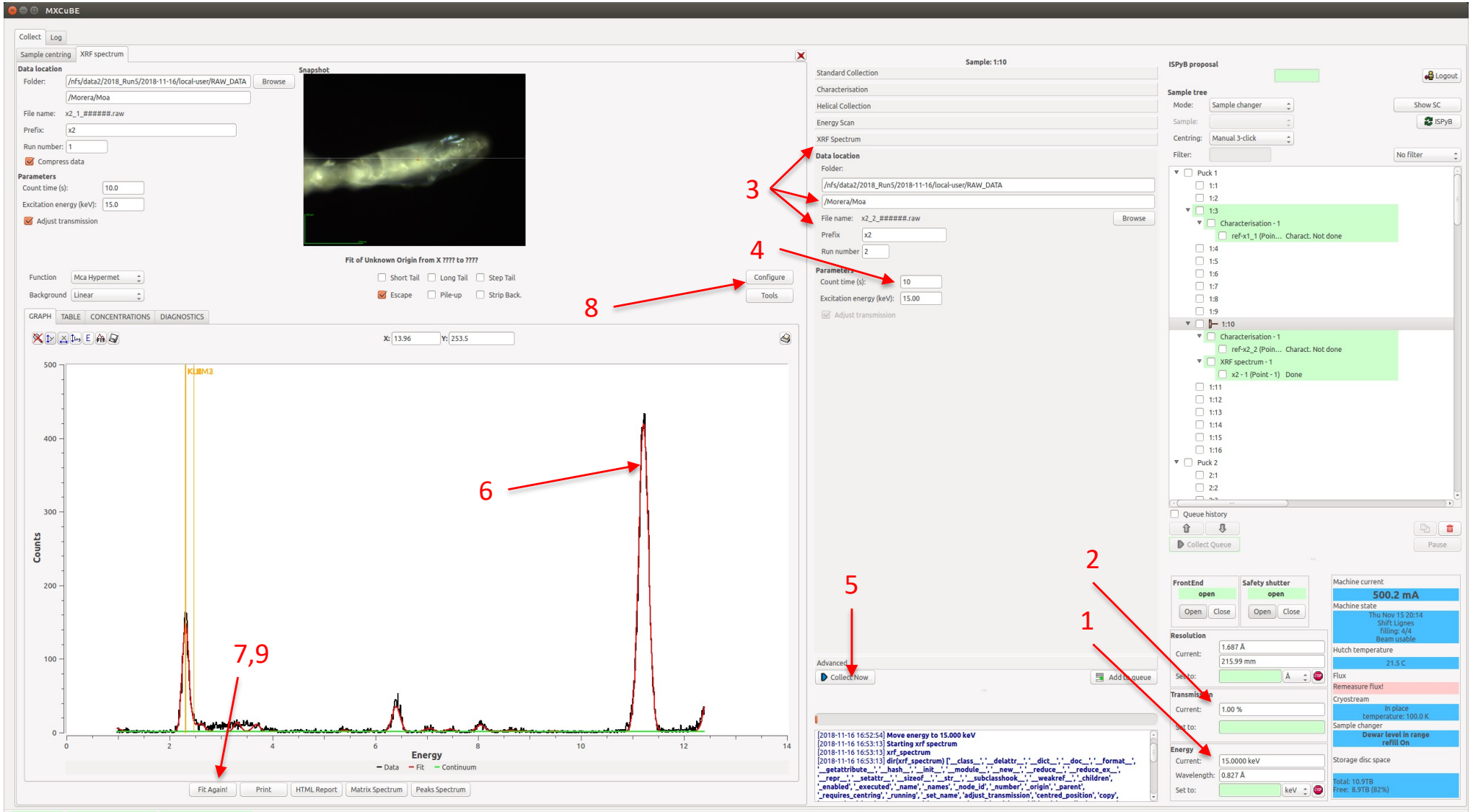


Processing Data

- Open an X-terminal window on proxima2a-10 or SRV4 terminal
  - Note: the cluster process1 is not available on the machines proxima2a-6 and proxima2a-7
- Log on to the calculation cluster by typing:
  - `ssh –X process1`
- Go to current directory where data were collected by typing:
  - `goimg`
- Launch XDS on the current or most recent collected data with:
  - `goxdsme`
- Launch XDS on older data from the process directory with:
  - `xdsme [options] ../my_crystal_1_master.h5`
    - `-h [help shows all options]`
    - `-a [anomalous]`
    - `-s P21 [spacegroup name or number]`
    - `-c “43 55 289 90 90 90” [unit cell in quotes]`
    - `--brute [for difficult indexing cases]`
    - `--weak [for cases with weak diffraction]`
    - `-r 1.2 [sets the high resolution limit]`
    - `-3 [relaunch from IDXREF step]`
    - `-5 [relaunch from CORRECT step]`

X-ray Fluorescence Spectra

1) Set Energy to 15 keV
2) Set Transmission to 1%
3) Click on “XRF” tab to specify folder & prefix
4) Set count time to 10 s
5) Click “Collect Now”
6) The display of X-ray Fluorescence spectra
7) Click “Fit Again” to automatically fit of common elements
8) Click “Configure” and “Peaks” tab to change the selected elements
9) Click “Fit Again” to refit the spectrum
10) Click “Peaks Spectrum”….???

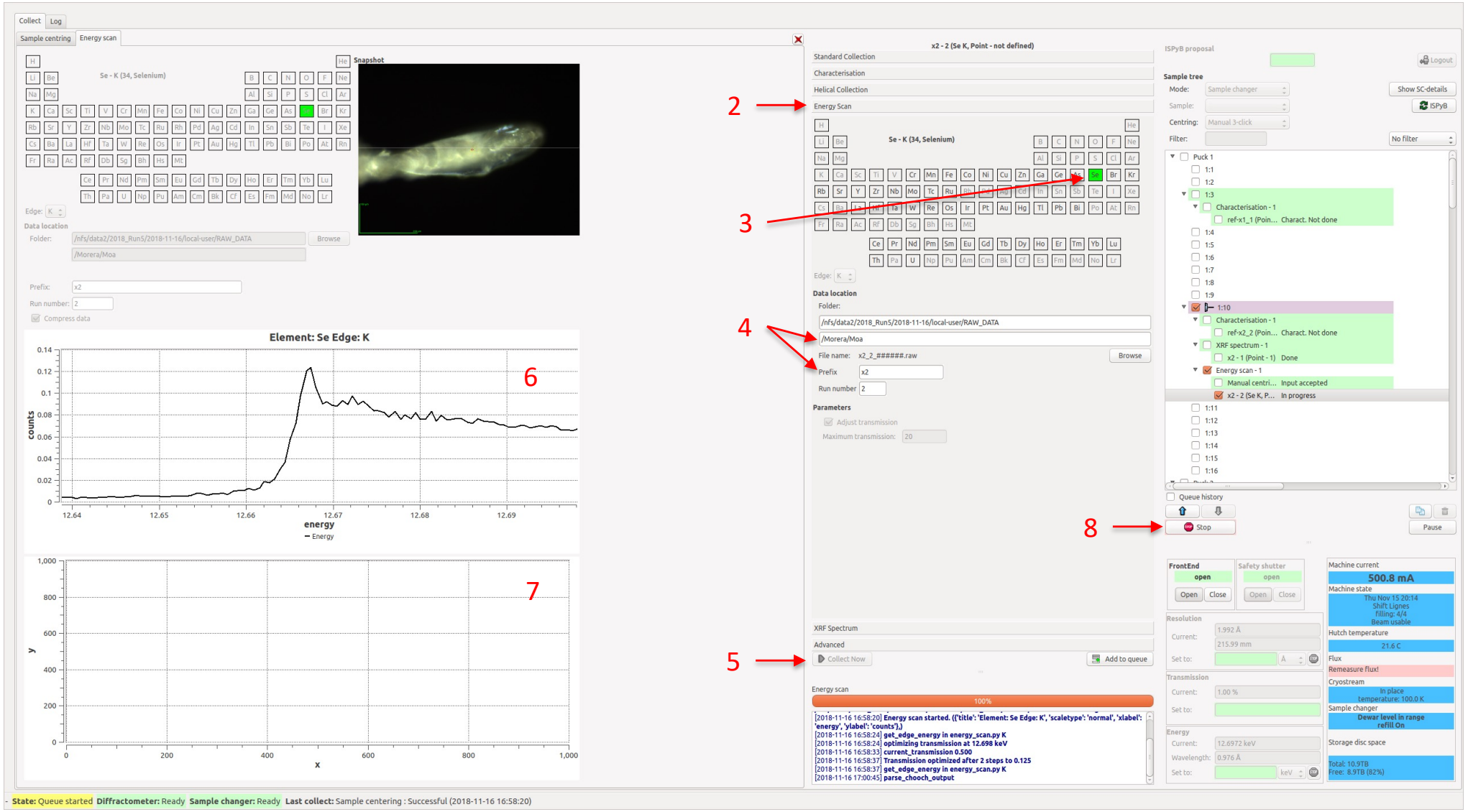


Energy Scans

1) Set Transmission to 1%
2) Click on Energy Scan
3) Select an element
4) Specify the folder & prefix
5) Click “Collect Now”

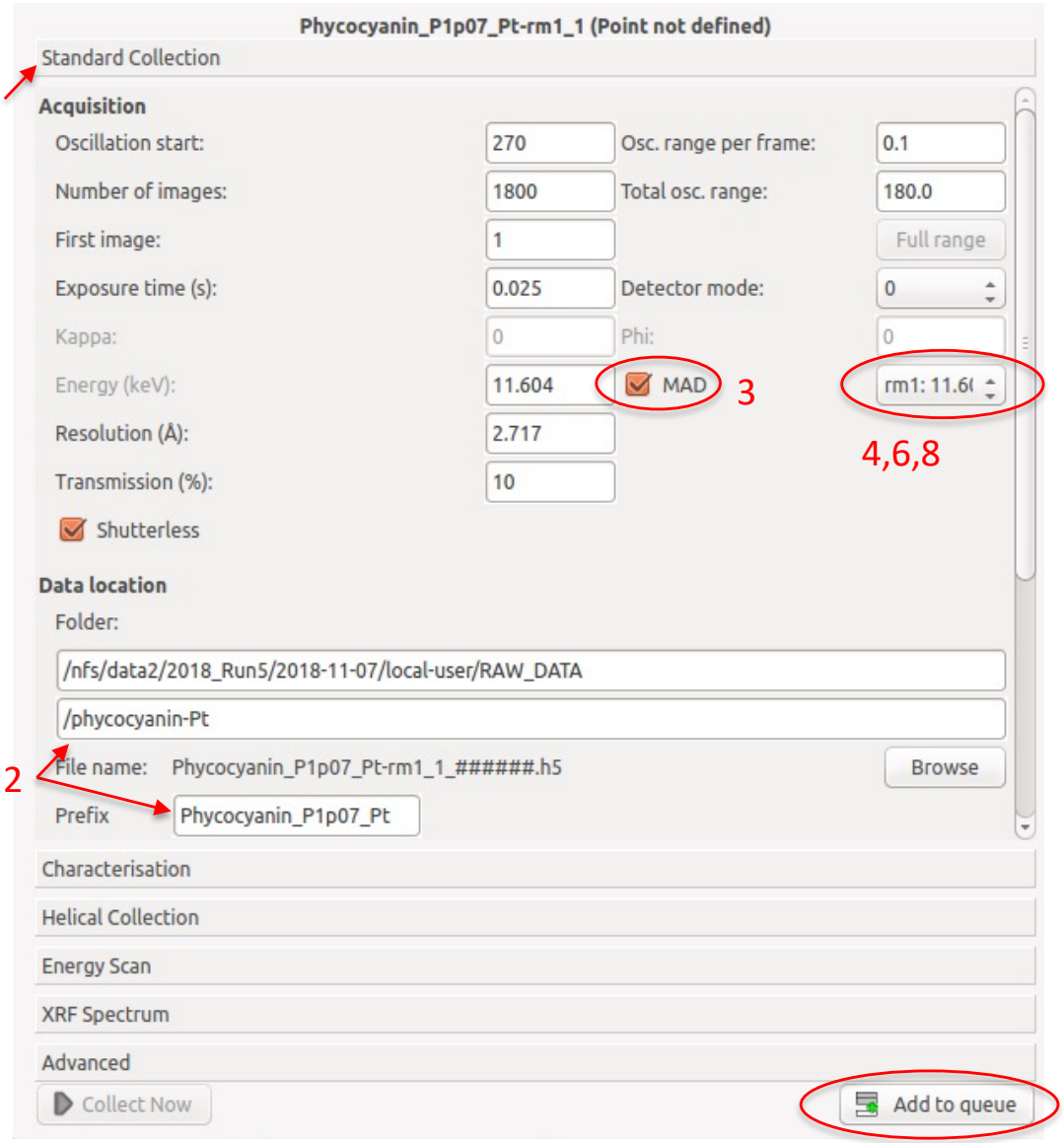
6) The Energy scan will plot the XANES spectra in real-time
7) CHOOCH will be displayed automatically
   - The pk, ip and rm energies will be updated in the Standard Collection tab in the MAD menu
8) If the Energy scan or CHOOCH fail, click STOP
9) If the Energy scan was completed, then in a terminal window type (the option –e specifies the element):

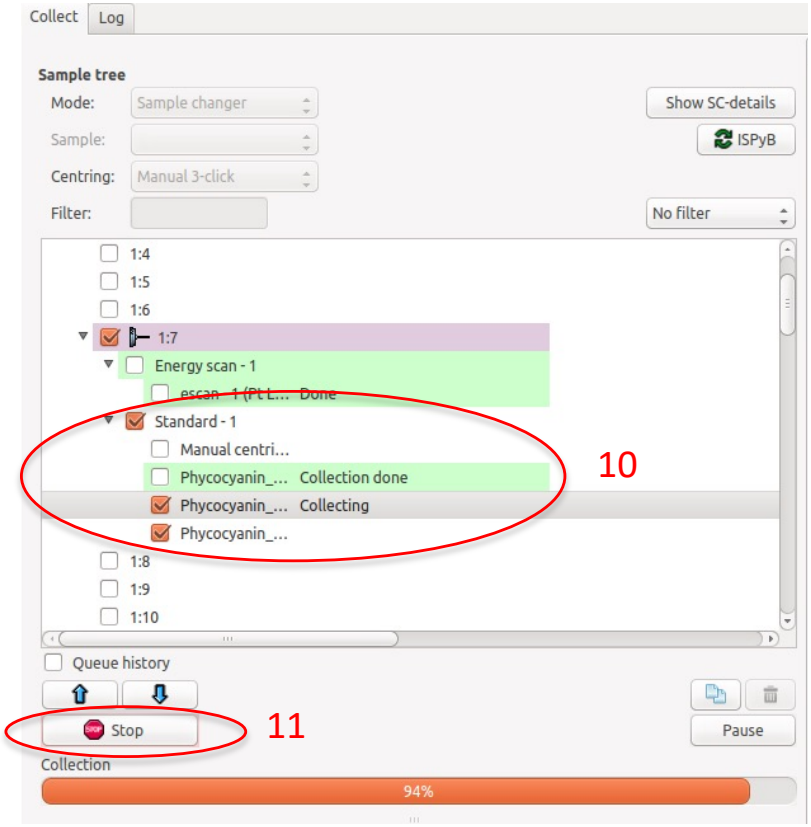
```
chooch –e Se MyEScan.raw
```



MAD Data Collections

1) Click on Standard Collection
2) Enter directory and prefix
3) Click the “MAD” radio button
4) Select an Energy (e.g. rm1, 11.609 keV)
5) Click “Add to Queue”
6) Select second Energy (e.g. pk)
7) Click “Add to Queue”
8) Select a third Energy (e.g. ip)
9) Click “Add to Queue”
10) Each data collection will appear in the Queue
11) Click the “Collect Now/Stop” button



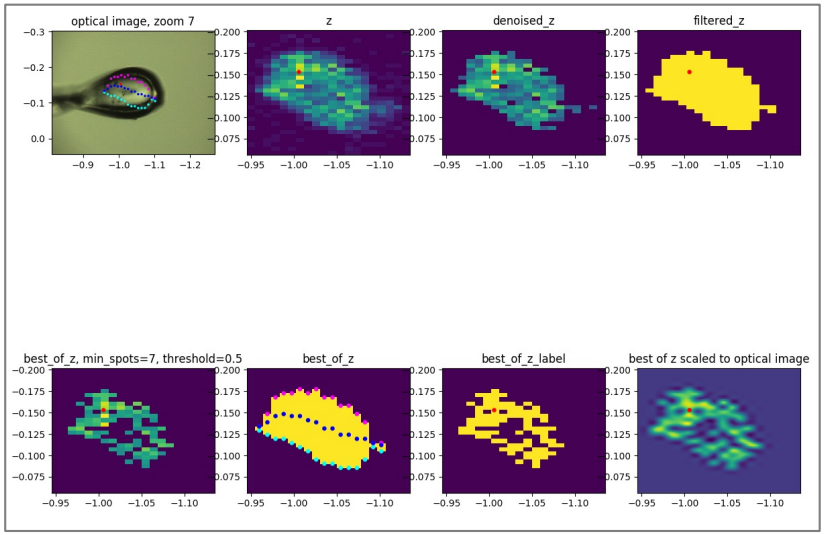


Manual Raster/Grid Scans

For those unafraid of keyboards, raster/grid scans can be done via command line on p10:

```
raster_scan.py
-d /data2/2018_Run5/2018-11-25/local-user/RAW_DATA/crystal1/raster1 [directory]
-n prefix_1   [prefix NAME]
-x 0.1        [Horizontal scan width in mm]
-y 0.1        [Vertical scan height in mm]
-c 50         [number of COLUMNS, 500 * x = 2 µm steps]
-r 50         [number of ROWS, 500 * y = 2 µm steps]
-a 123.25     [start ANGLE in degrees]
-A            [ANALYSE]
```

A Figure_1 window will open and display :



For more information, type:

```
raster_scan.py -h
```

or

```
raster_scan_analyse.py -h
```

Manual Area Scans

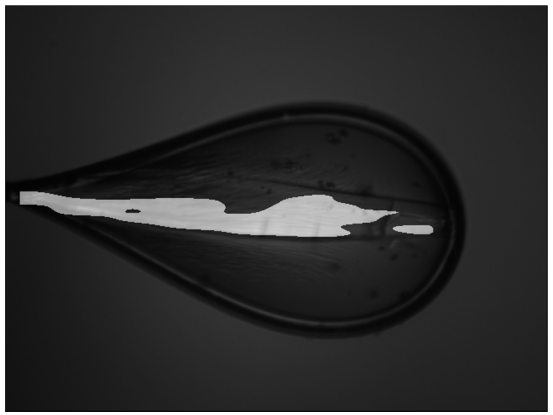
The older version of raster/grid scans can be done via command line on p10:

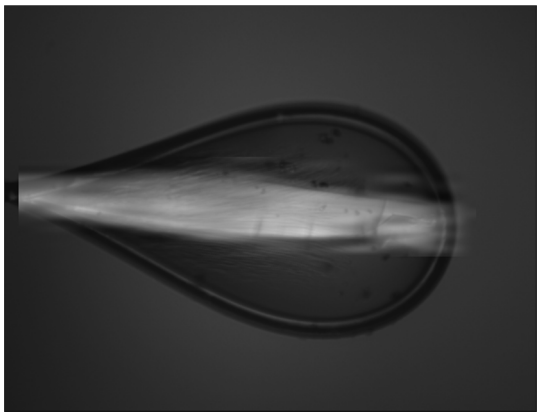
```
area_scan.py
-d /data2/2018_Run5/2018-11-25/local-user/RAW_DATA/crystal1/raster1 [directory]
-n prefix_1  [prefix NAME, default = « grid_ »]
-x 0.1       [Horizontal scan width in mm]
-y 0.1       [Vertical scan height in mm]
-c 50        [number of COLUMNS, 500 * x = 2 µm steps]
-r 50        [number of ROWS, 500 * y = 2 µm steps]
-p 331.25    [start ANGLE in degrees]
```

A window will open and display :

For more information, type:

```
area_scan.py -h
```





X-ray Centering (excenter)

X-ray centering can be accomplished via command line on p10:

```
excenter.py
-d /data2/2018_Run5/2018-11-25/local-user/RAW_DATA/crystal1/raster1 [directory]
-n prefix_1  [prefix NAME, default = « excenter_ »]
-l 0.1       [scan LENGTH in mm]
-a '(60,150,240,330)'     [scan ANGLES in degrees in 'tuple' format]
```

The goniometer should automatically move the calculated center position. Then click **SAVE** in MXCuBE.

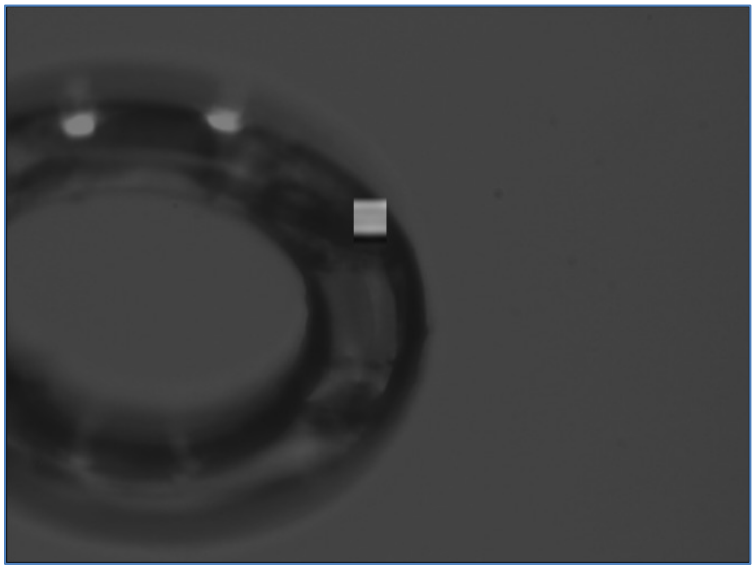
A window for each angle will open and display (note that the windows may display behind other windows) :

To only interpret images already collected:

```
excenter.py -i
```

For more information, type:

```
excenter.py -h
```



Sample Changer – « Error messages »

MXCuBE will display error messages returned by the CATS robot in the Sample Changer tab:

- CATS message window : incoherent diffracto detection. This means that the goniometer and the robot disagree whether a sample is mounted or not…
  - Check the CATS robot arm position
    - SOAK? – Then **ABORT**, **CLEAR**
    - OTHER? – Then contact the BL staff ASAP!
      - Check if the sample in in the CryoTongs
      - **ABORT, CLEAR, HOME, DRY**
  - Is a sample mounted on the MD2?
    - YES? – Then the previous sample was manually mounted, remove it manually and try again.
    - NO? – Then the CATS failed to pick up the SPINE pin (similar to the Missing Sample condition)
      - Try to **MOUNT** or **LOAD** again
      - If it fails again,
        - Check that the sample is really present via webcam “CAM8”
        - Click **DRY** to dry the cryotongs (3 min), and then **MOUNT** again
      - If it fails a third time,
        - Move the puck to another position in the CATS Dewar

- CATS message window: WAIT for TrfGtd condition:
  - The robot is waiting for the “transfer granted condition” to be given by the MD2
    - Check if the MD2 really is in Transfer Phase
      - Select **TRANSFER** in the Phase menu
    - **ABORT**
    - Try to **MOUNT** again

Sample Changer – Missing Samples, Collisions, Arm Power, etc…

If the robot fails to mount a sample pin:

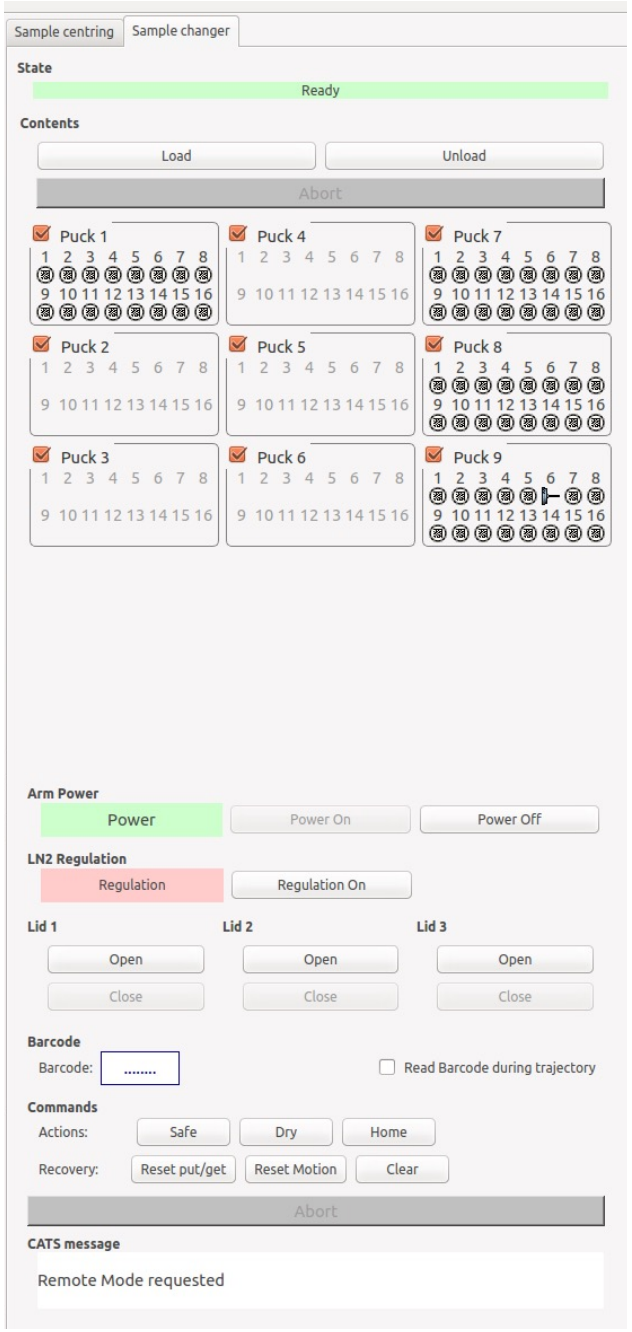
- This is known as a MISSING SAMPLE or INCOHERENT DIFFRACTO condition
- Try to **MOUNT** or **LOAD** again
- If it fails again,
  - Check that the sample is really present via webcam “CAM8”
  - Click **DRY** to dry the cryotongs (3 min), and then **MOUNT** again
- If it fails a third time,
  - Move the puck to another position

There are two types ROBOT COLLISIONS (usually in the Dewar):

- **SOFT** collisions
  - Symptom: the robot arm simply stops
  - These collisions can be recovered easily
    - Click the **SAFE** button (untested in MXCuBE Qt4)
    - Wait several minutes as the robot arm dries and recalibrates
    - Check that the sample is still present in the Dewar
    - Try to **MOUNT** again
- **HARD** collisions require manual intervention:
  - Symptom: the robot arm stops, but the cryotong is dislocated
    - The SAFE command will not run
    - When entering the hutch cryotongs will make a hissing noise
  - Ask for help from the BL staff or call 9797 (Hall Coordinator)
    - They will move the robot arm away from the collision point and to the HOME position

If the robot fails to move after you have been in the hutch (loading pucks):

- This might be because the Arm Power is OFF condition
  - Check that the ROBOT KEY is turned to the DOWN position
  - Click the POWER ON button
  - Try to **MOUNT** again
- If it fails again
  - Contact the BL staff or Hall Coordinator (9797)



Before You Leave PX2-A

1) UNMOUNT the sample with the robot via MXCuBE
   - It is impossible to open a lid with a sample on the goniometer!
2) OPEN the LIDS via MXCuBE
3) CLOSE the Safety Shutter via MXCuBE
4) ENTER the hutch
   - Press the blue PSS button & turn the CATS key to the top position (manual)
5) Remove your pucks
   a) Screw on a puck cover on to a puck-loading-tool
   b) Push the puck cover down onto the puck base
   c) Tilt the puck loading tool slightly towards the center of the lid
   d) Lift the tool up, the base should be attached
      - If the base does not come up, try « girating » the tool to dislodge the base
   e) Unscrew the puck from the tool
6) CLOSE the hutch door
7) TURN the CATS key to the bottom position (remote)
8) **CLOSE the LIDS and DRY-SOAK via MXCuBE**
   - **This keeps ice from forming in the Dewar!**