Endstation Operation and Data Collection Procedures

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∗∗All commands to be typed in the terminal are in italics.∗∗
∗∗Enter a command without arguments to see usage instructions.∗∗

1 Starting Endstation Software

1.1 Starting SPEC Software to collect data.

1. Open a terminal and login to the data acquisition computer by double-clicking **runRIXS_ES** on the desktop.
   - Enter your **group password**. The beamline staff should have provided login credentials for your group.
2. Start spec software on the RIXS ES by typing *runRIXS*.
   - Report any errors during starting to the beamline staff.
   - The procedure for recovering in case of an accidental terminal closure or computer issue is identical to above.

1.2 Starting SPEC dataGUI to visualize live data.

1. Start **dataGUI** by double-clicking **runRIXS_GUI** icon.
   - It will take a few seconds to load.

1.3 Starting cameraGUI to for sample alignment.

1. Start the camera software by double-clicking the **runRIXS_Cameras** icon.

1.4 Starting Jupyter Notebook for Data Analysis.

1. Start the analysis software by double-clicking the **runREIXS_Analysis** icon.
   - This will launch a terminal to start juptyer notebook and then launch a web browser.
   - The analysis notebook can only be run on one computer at a time.
   - Documentation can be found here: `https://pypi.org/project/reixs/`

1.5 Starting EDM screens for additional information.

1. Start the EPICS EDM screen by double-clicking the **REIXS_Users** icon.
   - This will launch window to view some beamline variables within EPICS
   - Launch additional relevant windows or strip-tool using the appropriate buttons.

2 Setup REIXS Beamline

- These steps only need to completed if you are starting for the first time, or if you are changing elemental edges.

2.1 Setup EPU

1. Check the current settings of the EPU using *statEPU* or observe the parameters on the **dataGUI**.
2. Select the EPU polarization using *setEPU polar*. Examples below:
   - Typically one uses horizontal polarization for almost all experiments. Vertical polarization is used to for spectrometer calibration or for specific experiments.
     - *setEPU polar lh* - linear horizontal (default)
     - *setEPU polar lvn* - linear vertical negative (use for calibration)
     - *setEPU polar lvp* - linear vertical positive
     - *setEPU polar li* - linear inclined
     - *setEPU polar cl* - circular left
     - *setEPU polar cr* - circular right
3. Select the EPU harmonic using *setEPU harmo*. Examples below:
   - Use the correct harmonic to ensure best higher order rejection and optimal photon flux.
     - *setEPU harmo 1* - set EPU harmonic to "1"
       - 95 - 850 eV
     - *setEPU harmo 3* - set EPU harmonic to "3"
       - 850 - 1500 eV

2.2 Setup Mono

1. Check the current settings of the Mono using *statMONO* or observe the parameters on the **dataGUI**.
2. Use *moveM* to select the mirror and *moveG* to select the grating for the desired energy range.
   - Ensure that you select the **mirror coating** before selecting the grating.
   - Generally, you can use the following:
     - Carbon mirror and Au LEG when using 100 eV to 250 eV energy range.
     - Nickel mirror and Ni LEG when using 200 eV to 750 eV energy range.
     - Silicon mirror and Au HEG when using 350 eV to 1500 eV energy range.
   - Examples below:
     - **Carbon** mirror and **Au LEG** grating
       - If changing from **Nickel/NiLEG**:
         (a) *moveE 250*
         (b) *moveM Carbon*
         (c) *moveG AuLEG*
       - If changing from **Silcon/AuHEG**:
         (a) *moveE 600*
         (b) *moveM Nickel*
         (c) *moveG NiLEG*
         (d) *moveE 250*
         (e) *moveM Carbon*
         (f) *moveG AuLEG*
     - **Nickel** mirror and **Ni LEG** grating
       - If changing from **Carbon/AuLEG**:
         (a) *moveE 250*
         (b) *moveM Nickel*
         (c) *moveG NiLEG*
       - If changing from **Silcon/AuHEG**:
         (a) *moveE 600*
         (b) *moveM Nickel*

(c) *moveG NiLEG*

– **Silicon** mirror and **Au HEG** grating

∗ If changing from **Carbon/AuLEG**:

(a) *moveE 250*

(b) *moveM Nickel*

(c) *moveG NiLEG*

(d) *moveE 600*

(e) *moveM Silicon*

(f) *moveG AuHEG*

∗ If changing from **Nickel/NiLEG**:

(a) *moveE 600*

(b) *moveM Silicon*

(c) *moveG AuHEG*

3. Move to the desired energy using *moveE*

- Typically the excitation energy is 30-40 eV above the edge. Consult electron binding energy values or consult beamline staff.

3 Setup Spectrometer

- Check the current settings of the Spectrometer using *statXES* or observe the parameters on the **dataGUI**.

1. Select the appropriate grating, use *setXES*.
   – Grating selection is based on a balance between efficiency and resolution.
   – Consult charts on wall for specific performance. In general:
   ∗ XLEG: 90 - 200 eV
   · *setXES XLEG*
   ∗ LEG: 200 - 400 eV
   · *setXES LEG*
   ∗ MEG: 400 - 700 eV
   · *setXES MEG*
   ∗ HEG: 700 - 1000 eV
   · *setXES HEG*
2. Move the spectrometer using *moveXES*
   – The location of the emission line is generally 10 - 15 lower than the absorption edge. Consult tables for binding energies or emission energies.
   ∗ Alternatively ask beamline staff for assistance.
   – The spectrometer requires image correction for optimal resolution and therefore, pre-work is required.
   – Use a location out of the list below (< 5 eV and < 1 deg) to ensure correct image processing. For example:
   ∗ *moveXES 520 -1* to move to 520 eV with -1 deg offset on the LEG.
   – After movement is complete ensure the correct shift file is loaded, otherwise software will report **NULL** and no image correction will be used.
3. Optimize the position of the spectrometer with 5 eV in later step once your sample is aligned.

SHIFT FILES AVAILABLE

- XLEG 80eV 4deg
- XLEG 93eV 4deg
- XLEG 114eV 4deg
- XLEG 120eV 4deg
- XLEG 126eV 4deg
- LEG 117eV 2deg
- LEG 276eV 0deg
- LEG 380eV 0deg
- LEG 390eV 0deg
- LEG 400eV 0deg
- LEG 455eV 0deg
- LEG 485eV -1deg
- LEG 515eV -1deg
- LEG 520eV -1deg
- LEG 575eV -1deg
- LEG 645eV -1deg
- LEG 675eV -1deg
- MEG 276eV 4deg
- MEG 380eV 1deg
- MEG 390eV 0deg
- MEG 400eV 0deg

- MEG 455eV 0deg
- MEG 485eV 0deg
- MEG 515eV 0deg
- MEG 520eV 0deg
- MEG 575eV 0deg
- MEG 645eV 0deg
- MEG 675eV 0deg
- MEG 710eV 0deg
- MEG 785eV -1deg
- HEG 645eV 2deg
- HEG 675eV 2deg
- HEG 710eV 1deg
- HEG 785eV 0deg
- HEG 860eV 0deg
- HEG 940eV 0deg
- HEG 1020eV 0deg

4 Setup Datafile

- Before attempting any data collection, it is best to setup the file for saving data. Some tips are:
  - Do not use a new filename for each scan.
  - Do not include spaces or math symbols in the filename.
  - Group scans together either with sample and/or element measured. This will depend on the number of scans, try to keep fewer than 100 scans per file.

1. Set datafile name using *newfile*
   - For example: *newfile test_file*
   - No extension is needed, all file extensions will be changed automatically.
   - If you wish to resume adding to existing files, just specify the name and it will change files.
   - **ENSURE YOU MAKE NOTE OF THE SCAN NUMBER AND FILENAME FOR YOUR LOG BOOK**

5 Aligning Samples

- While it is tempting to align samples only prior to the measurement, it is best to align all samples, save their positions and then proceed. This will significantly improve throughput.
- The sample alignment is achieved using the camera cross-hairs. The principle is that if the cross-hairs are co-located on the same position, the beam is there and the sample is in focus.

1. Turn on the beam using *setBEAM on*.
2. Ensure the chamber light is on using *setLAMP*.
   - Can be toggled on or off using *setLAMP on* and *setLAMP off*.
3. Roughly change the sample depth to ensure the cross-hairs are properly aligned in that they appear at the same sample position.
   - Use *umvr ssd* to add or subtract sample depth. For example, *umvr ssd 0.5* to add 0.5 mm of sample thickness.
4. Roughly position the sample in the beam using a combination of *umvr ssv* and *umvr ssh*.
   - *umvr ssv* will move the sample vertical position; positive vertical moves the sample upward.
   - *umvr ssh* will move the sample horizontal position; positive horizontal moves the sample to the right.
   - It is possible to combine movement for faster alignment such as *umvr ssv 1 ssh 2*
   - All units are in mm.
5. Use a line up scan *lup* to precisely align the sample.
   - For example *lup ssh -1 1 10 1* will scan relative -1mm to 1mm with 0.2mm steps counting for 1s each point.
   - ALWAYS scan positive and ensure the step size does not fall for below **50um for ssh** and **25um for ssv**.
   - Make use of all detectors, tey, sdd and mcp, to assist with determining good sample positions.
6. Save your sample position using *setSS*
   - For example: *setSS Sample_1*
   - Same rules for filenames, no spaces or math symbols.
   - If you want to re-save an existing sample, you will be asked to confirm the overwrite.
7. Move samples using *moveSS*

- For example: *moveSS Sample_1*
- Sample moves can be added to the macro files for automated experiments.

8. Save an image of the selected location using *captureSS*
   - This will save an image with the beamline cameras and cross-hairs indexed by filename, Scan#, sample name and date/time.
   - Images located in ../Screenshots folder.

6 Collecting XAS Spectra

- When collecting XAS spectra, it is important to ensure that all detectors are in an acceptable range.
- Adjusting detectors is easiest done above the edge, non-resonant. However, it may be necessary to collect a quick XAS scan and then move to a resonance to adjust the detectors.

1. Move above the absorption edge using *moveE*, typically 30 - 40 eV higher.
   - For K-edges, typically above the edge is 75-80 % of the maximum intensity.
   - For L-edge, you may need to a rough scan and move to the L3 resonance.
2. Count for 1s using *ct* to determine the event rate on detectors.
3. Adjust the count rate on the SDD_A (sdda) using the overall beamline flux, *setFLUX*
   - Ensure the count rate is $<$ 100,000 cps, typically set to 80,000 cps above resonance.
   - Reduce or increase the flux using *setFLUX* where the argument is % nominal, 100% is largest.
   - For example: *setFLUX 100* to set to maximum
   - For example: *setFLUX 50* to reduce flux to 50% of maximum.
4. One beamline flux is adjusted, adjust amplifiers using *setAMP* to optimize dynamic range.
   - Beamline flux meter is Mesh (i0), adjust using *setAMP i0 down* or *setAMP i0 up*.
   - Sample current meter is Sample (tey), adjust using *setAMP tey down* or *setAMP tey up*.
   - Changes the rate by a factor or 2 or 2.5.
   - Ensure rate does not exceed 1,000,000 cps.
   - Typically, set to 400,000 - 600,000 cps is ideal.
   - For the sample current (tey), 400,000 - 600,000 cps is okay above the edge for K-edge. L-edge may need to be lower.
   - Sensitivity can be set explicitly using *setAMP tey 10nA/V* for example. This is best for macros.
5. Check the count rate once again using *ct*
   - These settings can be added to a macro file to speed up the experiment.
6. Scan the energy using *Escan*.
   - Typically ones does a quicker rough scan to check beamline calibration and ascertain where there are resonances.
   - It is also advisable to always do two XAS scans to check radiation damage, either two normal scans added together to produce the final spectrum, or two quick scans followed by a longer final scan. The latter will aid in detector optimization.
   - For example: *Escan 390 395 25 405 200 415 100 425 50 5* will scan from 390 eV to 425 eV with step regions 0.2 eV, 0.05 eV, 0.1 eV and 0.2 eV with a count time per point of 5 seconds.
7. Do XAS calibration prior to experiment.

6.1 Calibrating XAS Spectra

- Typically, one uses a reference material to calibrate the XAS energy.

1. Select an appropriate reference material, either simple oxide or nitride.
2. Measure an XAS spectrum with the required precision at the resonances used for calibration.
   - 50 meV step size when 100 - 700 eV
   - 100 meV step size when 700 - 1200 eV
   - 200 meV step size above 1200 eV
3. Complete a reference XAS scan before an after the experiment per edge.
4. If running multi-day experiment, complete at least once per day.

7 Collecting XES Spectra

- While XES spectra seem to be a separate measurement, they are in fact the same as XAS, in that you acquire an XES spectrum at every point.
- Typically for XES, you simple count longer at specific energy.

1. Ensure that you have not left the flux reduced from previous XAS scans, *setFLUX 100*.
   - Typically all XES measurements are completed using full beamline flux.
2. Move the beamline energy to the required energy, *moveE*.
   - Typically, one does at the very least one non-resonant XES, about 30-40 eV above the edge.
   - Additional excitation energies can be selected based on peaks in the corresponding XAS spectra.
3. Ensure sample is in focus using cameras or pinhole.
   - Typically, the camera cross-hairs are sufficient to ensure the sample is in focus, provided the cross-hairs are located at the sample sample position.
   - If you are in doubt or have some challenge viewing your sample, you can use the pinhole to determine the focus location.
     (a) Set pinhole in place using *setXES pinhole*.
     (b) Count for 5s using *ct 5*.
     (c) Toggle image on the lower left plot on the **dataGUI**.
     (d) Use *umvr ssx 0.1* or *umvr ssx -0.1* successively to place the image centred vertically at channel 127.
     (e) Save the sample location using *setSS*.
     (f) Place the mask back in place using *setXES mask*.
     (g) Un-toggle image on the lower left plot on the **dataGUI**.
4. Determine the appropriate count time for the spectrum using *ct*.
   - Complete a 10s count *ct 10*.
   - Determine the total time required to achieve the require statistics.
   - Ideally, the best spectrum would have 10,000 peak counts per channel, but 1000 counts would be minimum.
   - For spectra where the point spacing is small < 50 meV, the adjacent channels can be binned to increase the count rate.
5. Collect XES spectra using *Tscan*
   - Collect for a total time with 30s exposures: *Tscan 360* counts for a total of 360s.
   - Collect for a total time with custom exposure time: *Tscan 360 120* counts for a total of 360s with 120s exposures.
   - Collect for a total time with custom exposure time and setting the excitation energy: *Tscan 360 120 420* counts for a total of 360s with 120s exposures at 420 eV.
   - If the energy is not specified, it will be acquired at the current beamline energy.
6. Do XES calibration prior to experiment.

7.1 Calibrating XES Spectra

- The calibration of the XES occurs relative to the XAS, with the XAS used as the reference from other beamtimes.
- The calibration consists of 5 elastic peaks and two reference spectra.

1. Collect resonant elastic scattering peaks on a bare material, stainless steel plate is okay, but Indium foil or Gold foil is better.
   (a) Place beam on bare scattering target using *umvr ssh* and *umvr ssv*
   (b) Set EPU polarization to linear vert- using *setEPU polar lvn*
   (c) Select 5 energy locations distributed throughout the XES energy window. Typically one at the centre and two on either edge.
   (d) Collect XES such that the peak counts is 1000 for each excitation.
   (e) Set EPU polarization back to linear horizontal using *setEPU polar lh*
   (f) Move to the reference compound and collect a non-resonant XES spectrum.
   (g) Move excitation to the first peak and collect a resonant XES spectrum

- Complete a reference XES scan before and after the experiment per edge.
- If running multi-day experiment, complete at least once per day.

8 Collecting XEOL Spectra

- Collecting XEOL presents a challenge in that the optical output is not correlated to x-ray absorption.
- Special care needs to be taken to ensure you take quality XEOL spectra.

1. Set the desired energy using *moveE*.
   - Typically XEOL is strongest at lower energy and before the XAS edge.
2. Determine the correct frame rate. Set the frame rate using *setXEOL rate*, where rate is in seconds.
   - Set the rate such that the detector is not clipping at 65,000 counts, but sufficient that the spectra are good quality, $>$ 30,000 counts.
   - For example: *setXEOL rate 10* sets the frame rate to 10s.
3. Collect XEOL spectra using *Tscan*
   - Ensure the exposure time matches the frame rate. For example *Tscan 300 10*.
     * You can see the exposure time before the *Tscan* using *setXEOL rate*.
   - The exposure time can be a multiple of the frame rate also.
4. Take a back ground spectrum with the same time rate, typically just complete the same scan with the beam off.
   - *setFLUX off*
   - *Tscan 300 10*
   - *setFLUX on*
     * The background spectra need to be in the same file as the data you want to use it for, then specify background scan in notebook plotting.