I14 user videos

GDA setting and scanning





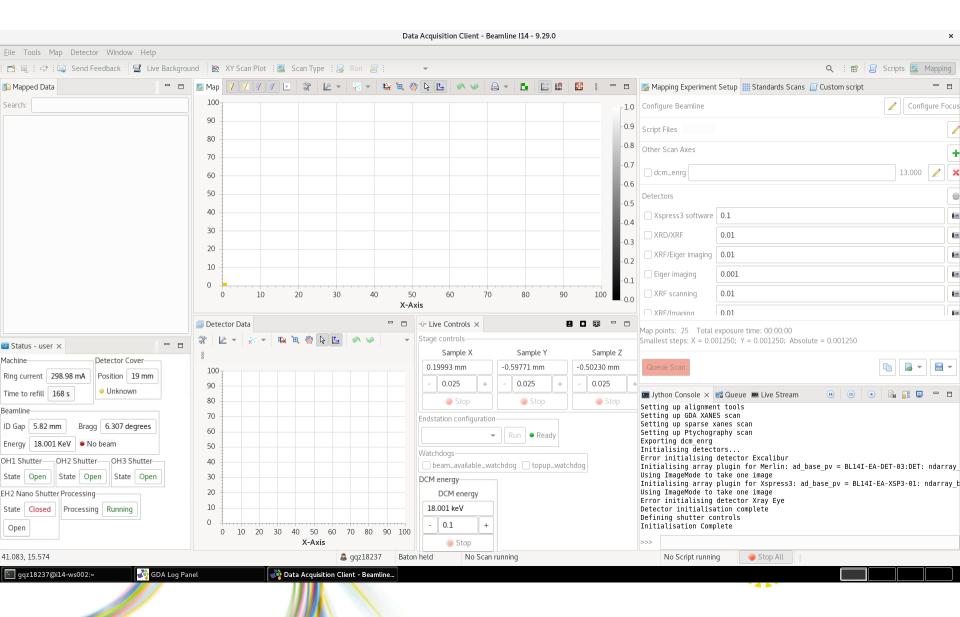
GDA is continuously evolving, so in order to get the latest release & updates, type: 'gdaclient --reset' in a terminal at the beginning of your experiment (as shown below), to launch the more updated version

If the GDA freezes during the beamtime, you can close it (and the Log Panel), and just type: 'gdaclient' for re-launching it or the full 'gdaclient --reset' for a new reset

FedID@i14-ws002:~	×
File Edit View Search Terminal Help	
FedID@i14-ws002 ~]\$ gdaclientreset	

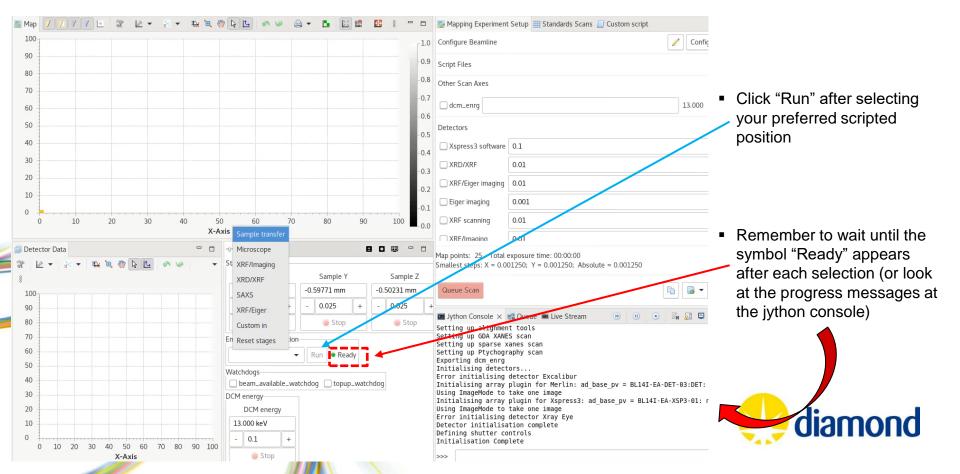
A more comprehensive restart may be needed if the GDA refuses to work, which can be triggered by typing 'gda servers', but please inform your local contact before doing so





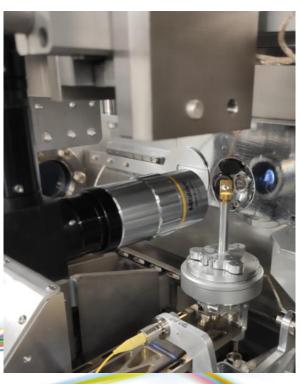
The 'Endstation configuration' menu at the centre of the screen would prepare the instruments accordingly:

- Sample transfer: Detectors out of the way, safe position for the users to go inside the hutch
- Reset stages: Automatically reset the stages for an accurate positioning (use after every sample change)
- Microscope: Move the microscope at the back of your sample, looking for the focus position
- **XRF/Imaging:** Place the Merlin detector at the back of the sample for phase contrast imaging, detector cover out of the way, preparation to allow beam on
- XRD/XRF or XRF/Eiger: Similar to above but placing the Excalibur detector for XRD or the Eiger in position



The 'Endstation configuration' menu at the centre of the screen would prepare the instruments accordingly:

- **Microscope:** Move the microscope at the back of your sample, waiting until the completion message appears at the jython console (or until you can see the camera stops through the webcams inside the EH2)



- At the 'Live Stream' tab, the "sample microscope camera" can be opened by doubleclicking or selecting connect.
- That would open a microscope tab at the top-centre (red-ellipse), in which the exposure time (0.01 below) may be adjusted to increase/decrease the brightness of the view and start/stop the camera using the symbols.
- Once you are looking at the live view, the X,Y, Z stages should be moved using the differential value below (+ or -) until the region of interest is located at the cross displaying on the camera view.

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Setting up the GDA session: Mapping

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at I14:

- **Detectors:** The small gear would allow to "clean" the area, by selecting only the detectors in use (XRF/Imaging would trigger the XRF detector at backscatter position and the Merlin photon counting detector (at transmission geometry)

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Setting up the GDA session: Mapping

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at I14:

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~300-500 nm values are adequate for coarse scanning, while 50-100 nm will be for high-	SampleX Centre 0.5 mm - SampleX Step 100	nm 👻
resolution maps)	SampleX Range 5 µm - SampleY Step 100	nm 👻
 Select the mapping (X,Y) size 	SampleY Centre 0.5 mm - Alternating	
About 50 micron is good for low-resolution then adjust to lower values for high-res	SampleY Range 5	•
 Set the "Sample name" for clarity 	Sample Name	Edit metadata
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provide an estimation on how long the scan proposed is going to take	Queue Scan	•
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	Catting up alignment tools	

Setting up the GDA session: XRF windowing

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at 114:

- Processing: The 'Add Processing' button allows selecting some automatic postprocessing of the data.
 - At the 'Specify application and setup config', the 'i14-xrf' option can be selected

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Setting up the GDA session: XRF windowing

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at I14:

- Processing: The 'Add Processing' button allows selecting some automatic postprocessing of the data.

The 'i14-xrf' template would allow some live visualisation of the data

• Change the 'Width' value to 20, and then click at the "XRF" button

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Setting up the GDA session: Mapping

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at I14:

- Processing: The 'Add Processing' button allows selecting some automatic postprocessing of the data.

The 'i14-xrf' template would allow some live visualisation of the data

- The corresponding processing chain will be selected by default, which could be discarded by unclicking the small square at the left Mapping Experiment Set
- If the detector is in place (look info advancing two more slides), you could use 'Queue Scan' to submit a singular scan in the sample X,Y range selected
 (*Note:* If the beam is OFF the 'Queue Scan' button will be red-coloured, meaning that it need to turned ON before submission, which will happen automatically when selecting the detector)

 ✓ An autogenerated processing chain will be displayed When selected, it will generate a scan in the /mgxxxxy/processed/ directory to investigate the data.
 Also the same scan will be loaded back in the "Mapped Data space at the left of the GDA console, for a live assessment



Mapping Experiment Setup Standa	ards Scans [Custom script	13.00	0 🖉 🗙
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SampleX Range 5	µm 🔻	SampleY Step	100	nm 👻
SampleY Centre 0.5	mm 💌	Alternating		
SampleY Range 5	µm 🔻	Orientation	Horizontal 👻	
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App Name				
il4-xrf {"element_list":"Fe-Ka","	'window_width	n":20}		
Nap points: 2,601 Total exposure time: Smallest steps: X = 0.0001000; Y = 0.000		lute = 0.0001000	1	
Queue Scan				•
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Catting up alignment tools				

The 'Mapping Experiment Setup' tab allows controlling how to submit all the analysis requested at I14:

- Processing: The 'Add Processing' button allows selecting some automatic postprocessing of the data.
 - The 'i14-xrf' template would allow some live visualisation of the data

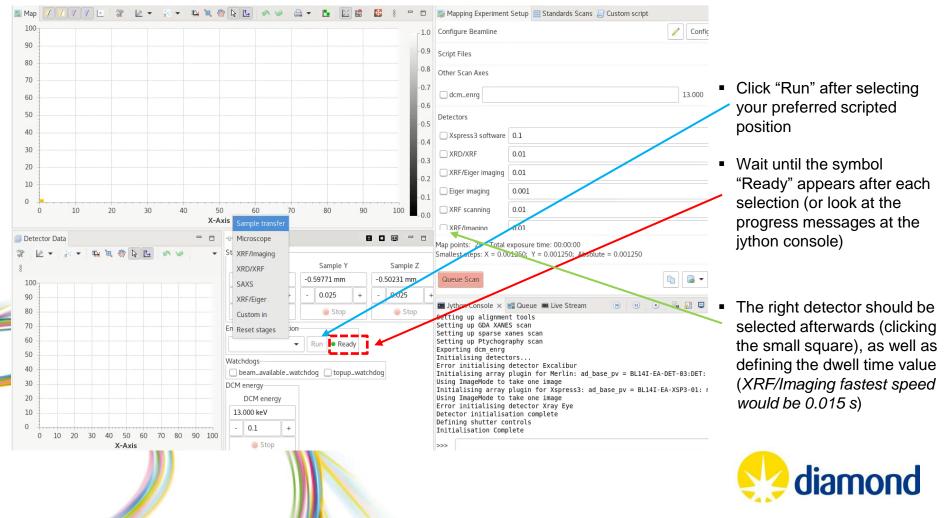
In the third section "specify application and config file", type 'dpc' at the App Name space This will trigger an automatic differential phase contrast (DPC) reconstruction of the transmitted signal acquired from either the Merlin or the Eiger detector, which will be loaded back at the "Mapped Data" area, as 'i14_xxxxxx_dpc_phase.nxs' file at the left of the GDA console

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 Click "Finish" 		rocessing Template and Detector Selection	
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🗐 lazic_include		provide a nice phase contrast image	
		provide à fille priase contrast image	

Scanning at GDA: Placing the detector in position

Before scanning, remember to select the appropriate detectors to be used at the 'Endstation configuration' menu:

- **XRF/Imaging:** Place the Merlin detector at the back of the sample for phase contrast imaging, detector cover out of the way, preparation to allow beam on
- XRD/XRF or XRF/Eiger: Similar to above but placing the Excalibur detector for XRD or the Eiger in position



Scanning at GDA: Checking the beam status

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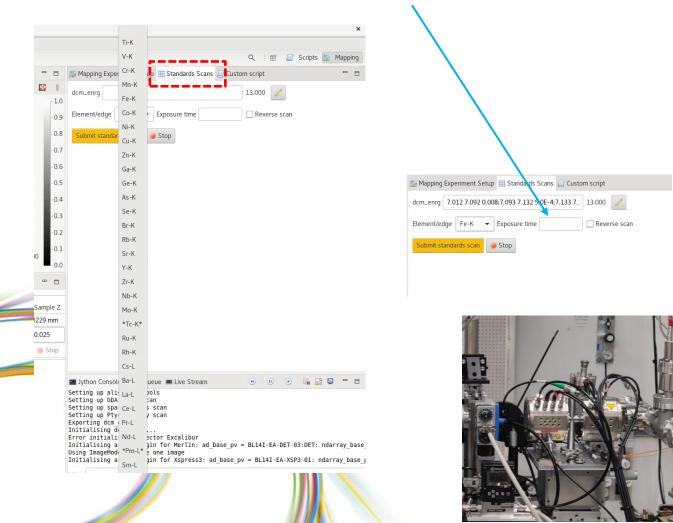
Scanning at GDA: Checking the beam status

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Scanning at GDA: point XANES

Point XANES or standard XANES acquisition: The 'Standards Scan' tab (red rectangle below) allows performing point xanes acquisition on a fixed location of the sample as well as measuring the XANES pellets. In there, the 'Element/edge' to analyse should be selected from the drop-down list (i.e., Fe-K) as sown at the left below, which will populate the energy list accordingly (right image). The exposure time (dwell time) should be typed (for example to 0.1 s).



Things to notice before submitting this analysis:

- The beam may need to be manually opened beforehand
- For point XANES, double click on the position of interest firstly in the mapping area to move the stages (a warning message will appear to confirm the movement)
- Standard XANES: The pellet should be placed between the ion chambers at the EH2, and remember to remove it straight away once finishes!

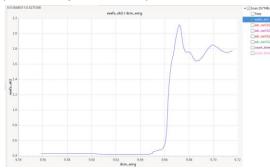


Scanning at GDA: point XANES

Point XANES or standard XANES acquisition: The 'Standards Scan' tab (red rectangle below) allows performing point xanes acquisition on a fixed location of the sample as well as measuring the XANES pellets. In there, the 'Element/edge' to analyse should be selected from the drop-down list (i.e., Fe-K) as sown at the left below, which will populate the energy list accordingly (right image). The exposure time (dwell time) should be typed (for example to 0.1 s).

Standard XANES pellet

The running scan will generate a new tab: 'Scan Plot' at the right of the Queue and the jython console, in which the signal between the ion chambers may be selected: eh2_exafs



Point XANES

After selecting a region of interest on the mapping area (double click will move the stages first!), a point XANES on that nanoregion can be submitted. In this case the data will NOT be live on the 'Scan Plot' tab, which only shows the ion chambers signal. For reconstructing the point xanes, check this <u>video</u> on the <u>l14 website</u>, on how to load jupyter notebooks. Then: 1) select: /dls_sw/i14/ops/notebooks/centralised_notebooks/point-XANES_XRF-standard.ipynb, 2) update the 'inpath' details, and 3) "Cell" – "Run all"



Scanning at GDA: XRF Mapping

At the 'Scan Type' icon (red rectangle below), the main analysis techniques available at I14 can be selected. Remember to select the 'Mapping' option for XRF imaging.

	Data Acquisition Client - Beamline I14 - 9.29.0		x
nd 🛛 🖄 XY Scan Plot 🗄 🔟 Scan Type 🛛 😹 Run 🚇	•		🔍 i 😰 🔲 Scripts 💹 Mapping
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	Choose the scan type	-0.9	Detectors
80		-0.8	Region shape: Centred Rectangle SampleX Centre 0.5 Image: SampleX Step 100 Image: SampleX Step
60 50 40	Mapping scan XANES scan Sparse XANES scan	-0.6 -0.5	SampleX Range 5 µm - SampleY Step 100 nm -
30 20	Tomography scan Ptychography scan	-0.4	SampleY Centre 0.5 Mm Mm Continuous SampleY Range 5 Mm Orientation Horizontal
		-0.2	Sample Name Test_sample Edit metadata Processing Add Processing
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90	- 0.025 + - 0.025 + -	230 mm 0.025	Map points: 2,601 Total exposure time: 00:00:39 Smallest steps: X = 0.0001000; Y = 0.0001000; Absolute = 0.0001000
	Stop Stop	Je Stop	

Scanning at GDA: XANES spectromicroscopy

Scan type → XANES

Increasing photon energy

The 'Mapping Experiment Setup' tab will change its layout, adjusting to this acquisition methodology.

In there, the 'Element/edge' to analyse should be selected from the drop down list (i.e., Zn-K) as shown at the image, which will populate the energy list accordingly at the top.

The "Tracking" allows to select a background element (i.e., Ca) and a electronic transition (i.e., Ka) whose signal should be constant. Hence, please select an element/transition with an energy lower than the edge/element for XANES. This <u>published methodology</u> (Quinn *et al.*

J. Synchrotron Rad. (2021). 28, 1528-1534) automatically adjusts the sampling area at the beggining of each scan to ensure that the micro-structure remains in the field of view.

The 'Total exposure time' will be an estimation based on the submission of the ~150 individual scans, but some time on overheads, tracking and energy changes should be accounted (usually around ~1 hour more)

Again, the 'Submit XANES scan' button will be red-coloured if the beam is OFF. Please, ensure that this is activated before submission.

μ(E)X

Vormalised

5650

5700

Energy (eV)

5750

5800

	ment Setup Standa				13.000		' E
Detectors							100
✓ XRF/Imaging	0.015						
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			di	am	00	d	

Scanning at GDA: underscanning XANES

Scan type → sparse XANES

This is based on a published <u>undersampling methodology</u> (Townsend *et al.* Optics Express (2022). 30, 43237-43254) for reducing the dose and scanning times. This is achieved by skipping rows within scans and reconstructing the X-ray spectromicroscopy data using low-rank matrix completion.

The 'Mapping Experiment Setup' tab will change its layout, adjusting to this acquisition methodology.

Only the XRF detector can be triggered in this analysis, since missingrows would make the photon counting detectors to fail. This is automatically selected by default, but remember to re-select XRF/Imaging or XRD/XRF when transitioning back to the "Mapping" scan type.

The 'Element/edge' to analyse should be selected from the drop down list (i.e., Zn-K) as shown at the image, which will populate the energy list accordingly at the top.

An 'Energy Offset' may be inputted if the energy calibration has been done in advance. Please, input only non-decimal numbers (in eV units) either positive or negative according to the calibration.

The 'Percentage' of undersampling may be adjusted if needed, but a 20% missing data is adequate, and it has been scientifically proven. The 'Total exposure time' is an estimation based on the submission of the ~150 individual scans with FULL ROWS. Hence, the scan will be considerably faster (total time about 1/3 of the estimated time) Again, the 'Submit Sparse XANES scan' button will be red-coloured if the beam is OFF. Please, ensure that this is activated before submission.

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_	Percentage (%) 20 - +							
	Sample Name Test_sample Edit metadata							
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	Submit Sparse XANES scan 💽 Stop							
		- 8						
	Setting up alignment tools Setting up GDA XANES scan Setting up Sparse xanes scan Setting up Ptychography scan							



Scanning at GDA: Ptychography

Scan type → ptychography (currently under development)

This methodology is still "under development", so please speak with your local contact before trying it.

Once triggered, the GDA will automatically move the stages 800 μ m out of focus (positive Z, making the beam bigger at the point of exposure), adjust the stage offset, submit the ptychography scan plus the associated reconstruction strategies, and place the stages back to the focus position upon scan completion.

The 'Mapping Experiment Setup' tab will reflect the ideal mapping resolution (sampleX and Y Step), which is 100 nm (although this may be decreased to ~50 nm to ensure enough data overlapping).

There will be some <u>PtyPy</u> reconstruction algorithms triggered by default (listed at the right of the 'ptypyi14' app), which should be evaluated upon completion at the '/mgxxxxy/processed/ptychography_ptypy/' directory (Please, allow at least 2-3 hours for completion depending on the dataset size).

Again, the 'Submit ptychography scan' button will be red-coloured if the beam is OFF. Please, ensure that this is activated before submission.

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Scanning at GDA: XRD

Scan type → Mapping This

To be completed





Scanning at GDA: Tomography

Scan type → tomography This

To be completed



