



Webinar Series

Small Crystals, Big Insights: How Electron Diffraction is Transforming Materials, Life Science, and Chemistry Research

Episode 3 – Simple Electron Diffraction Workflow from
Sample Prep to Structural Solutions

Presenter: Jessica Burch, PhD

Wednesday, May 7, 9 am CST

- *You will be muted during the workshop*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working*





Webinar Series

Small Crystals, Big Insights: How Electron Diffraction is Transforming Materials, Life Science, and Chemistry Research

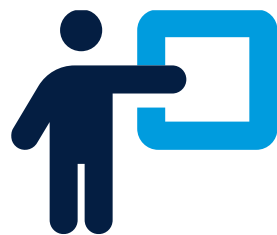
Episode 3 – Simple Electron Diffraction Workflow from
Sample Prep to Structural Solutions

Presenter: Jessica Burch, PhD

Wednesday, May 7, 9 am CST

We are starting now





Presenter:
Jessica Burch
Application Scientist



Host:
Joseph Ferrara
CSO, Rigaku Americas

You can ask questions during the presentation. Please use the Q&A to ask questions.



Recording will be
available tomorrow.



Simple Electron Diffraction Workflow from Sample Prep to Structural Solutions



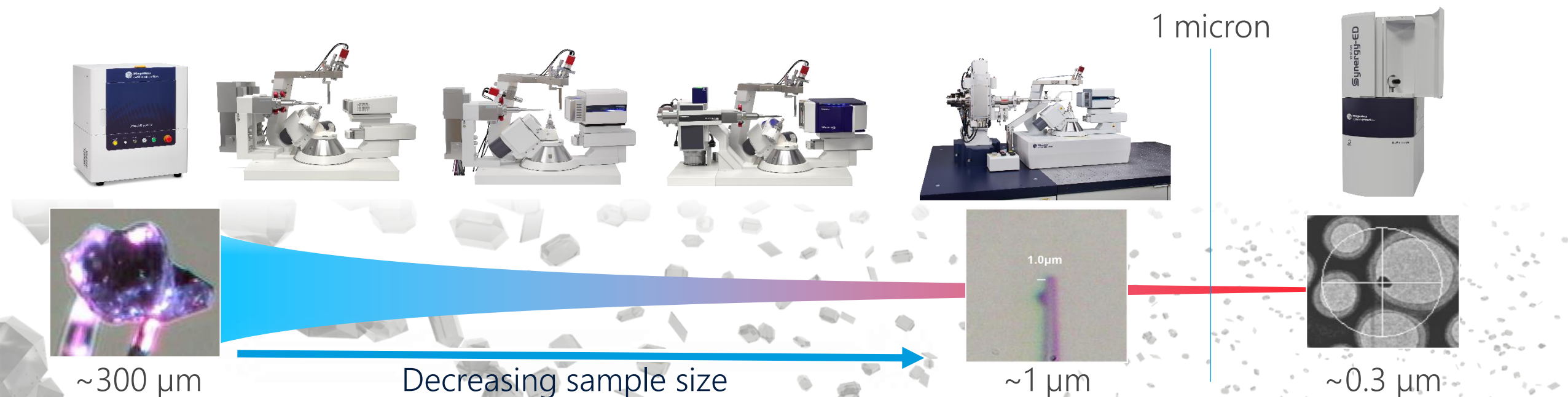
Agenda

1. Overview of 3D ED / microED
2. Sample preparation strategies
3. 3D ED data collection and processing with the Synergy-ED
4. Use of automation
5. Summary

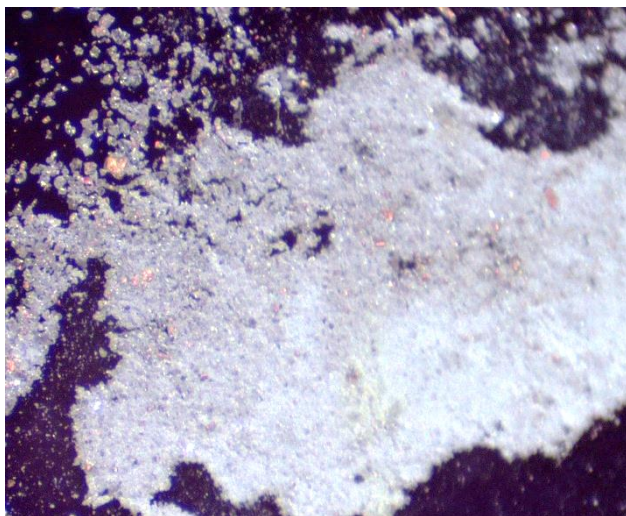
1. Overview of 3D ED / microED

Breaking the 1 Micron Barrier

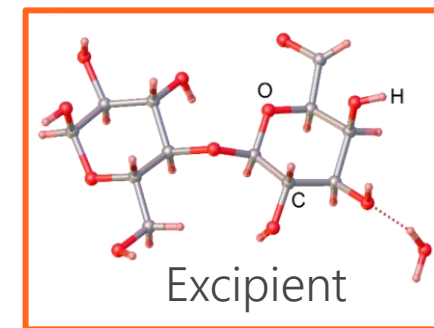
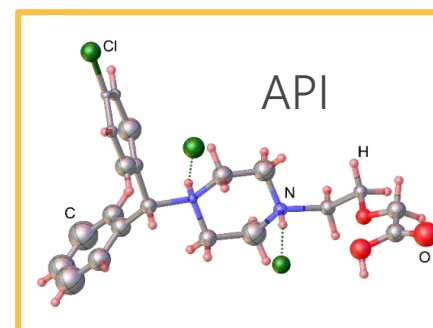
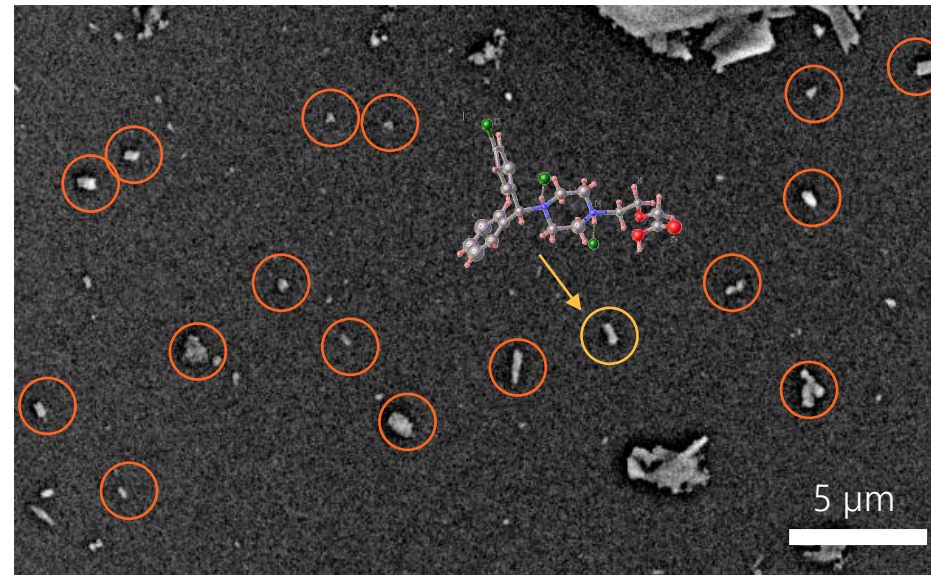
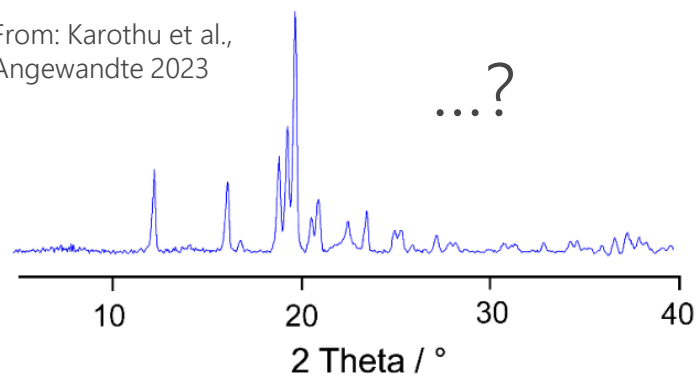
- X-rays reach down to micron size crystals
- Electrons reach *up* to micron sized samples.
- X-ray and electron diffraction are **complementary** techniques, together allowing study of samples from several hundred microns all the way down to the nanoscale.



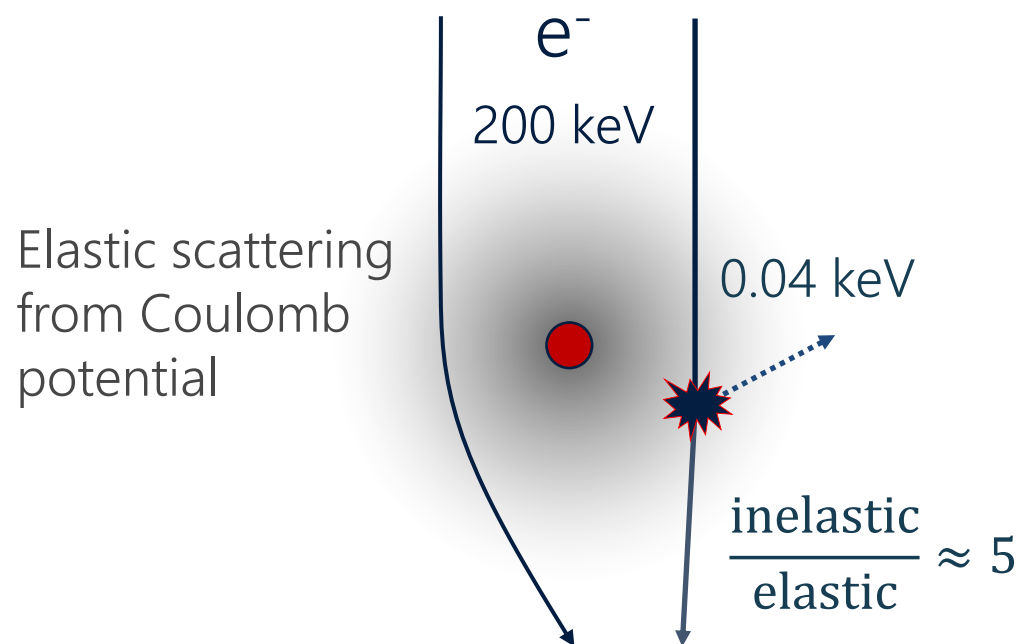
Powders Grain-by-Grain



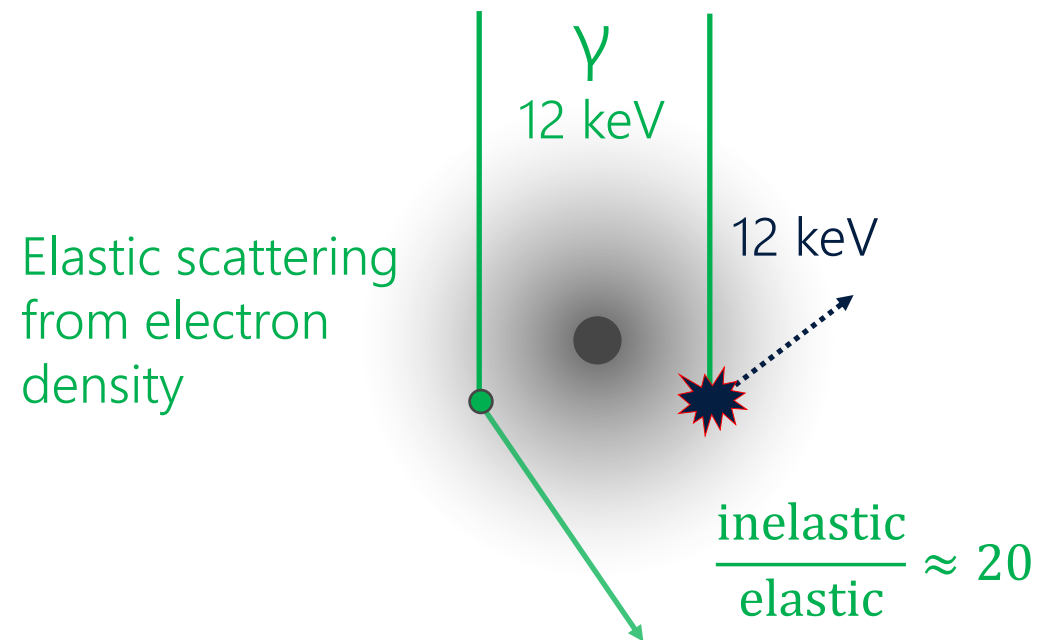
From: Karothu et al.,
Angewandte 2023



Electrons vs. X-rays



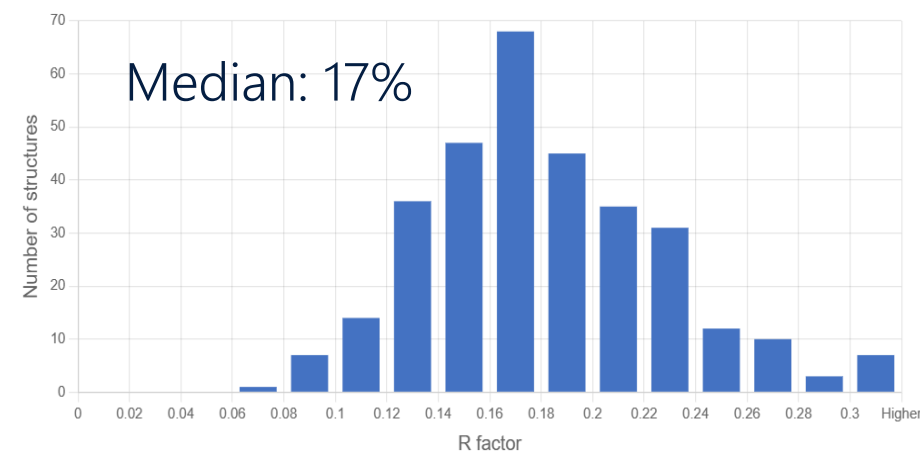
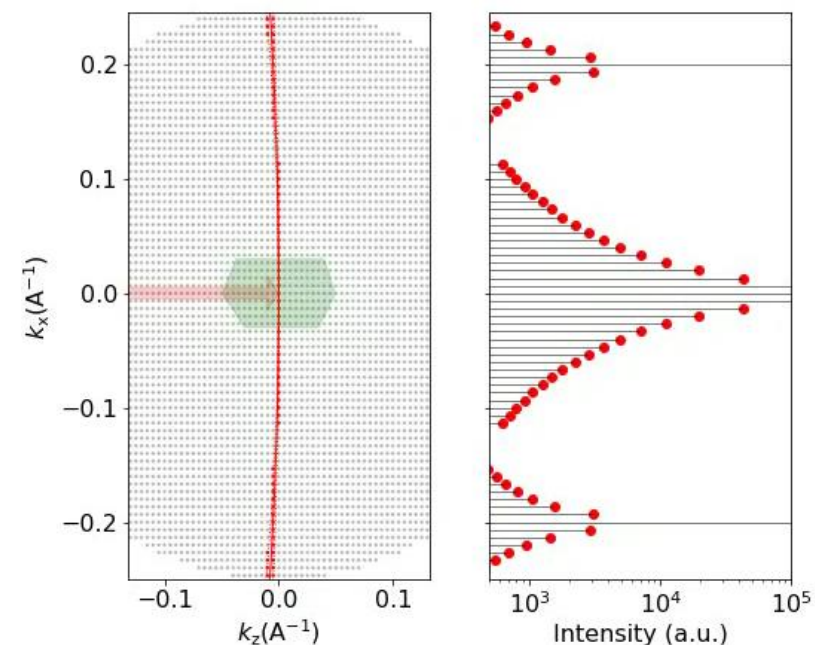
Electrons interact 10^6 times stronger
 → sub- μm crystals are possible *and required*



10^3 x lower energy deposit per elastic event
 → less radiation damage per information

Electron Diffraction - Data

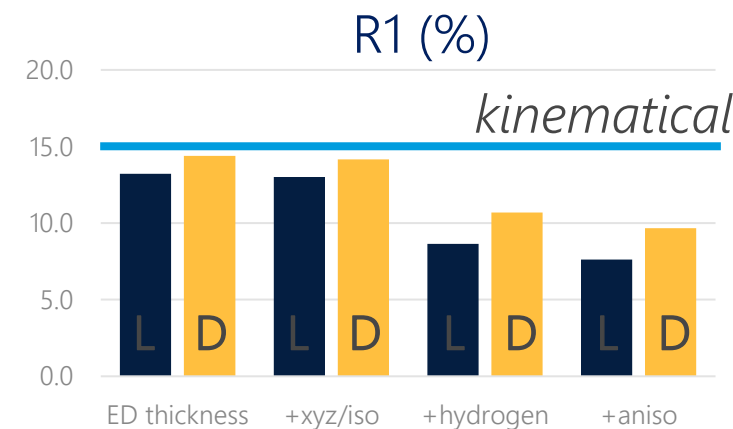
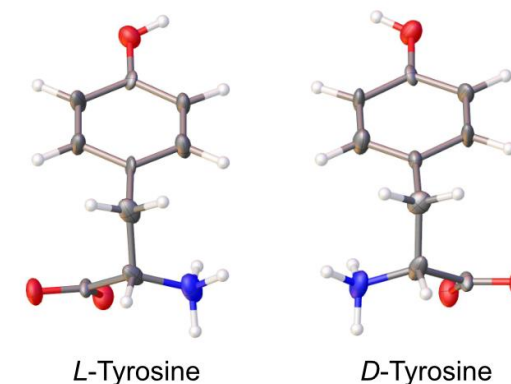
- Very strong interaction: dynamical diffraction due to multiple scattering
 - $I_{hkl} \neq |F_{hkl}|^2$
- New data collection approach *unlike* "classical" ED
 - Dynamical diffraction gets suppressed!
 - Refinement in **kinematical** approximation: *systematically* high R-values, but good structures!



Spence, J. C. H. (2006). High-resolution electron microscopy for materials science.

Electron Diffraction - 3D ED / microED

- Replace X-rays in single-crystal diffraction by electrons! Invented from 2007 by several groups independently: 3D ED, microED, RED, cRED...
- Full dynamical refinement: heavy computation, but improved R-values and maps, absolute structure!
- Conducted in adapted TEMs by specialized research groups



Palatinus, L.; Petříček, V.; Corrêa, C.A., *Acta Cryst. A* **2015**, 71, 235–244.

Brázda, P.; Palatinus, L.; Babor, M., *Science* **2019**, 364, 667–669.

Klar, P.B.; Krysiak, Y.; Xu, H.; Steciuk, G.; Cho, J.; Zou, X.; Palatinus, L., *Nat. Chem.* **2023**, 15, 848.

Truong, K.-N. *et al. Symmetry* **2023**, 15(8), 1555.

Crystallography Meets Electron Microscopy

Transmission Electron Microscope



- Wide variety of measurement types
- Frequent manual alignments required
- Often qualitative/image analysis

Single Crystal X-ray Diffractometer



- Specific measurement modalities
- Fully automated and integrated
- Quantitative and (reduced) data-driven

→ Differing expectations for instrumentation and analysis

XtaLAB Synergy-ED

A dedicated electron diffractometer,
made for non-specialists

Highly optimized and integrated hardware and
software design for microED / 3D ED

Seamless workflows, from instrument control to
structure solution

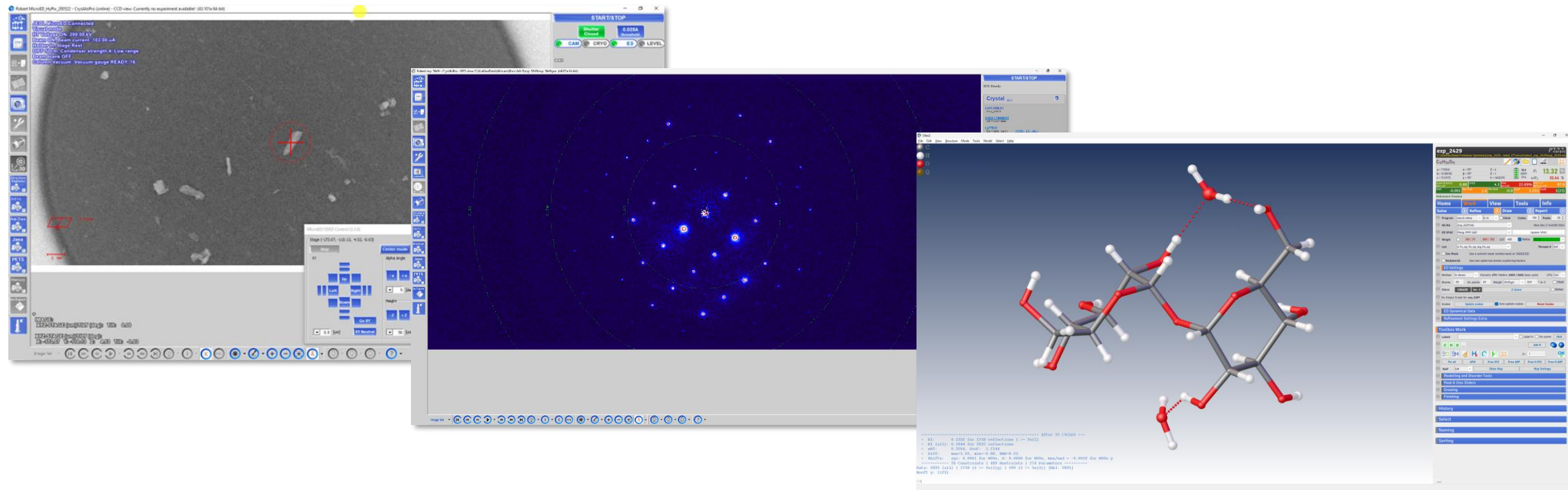


Rigaku



XtaLAB Synergy-ED: An Integrated Experience

Screen, collect, automate, process, solve, manage,... in *one* seamless interface

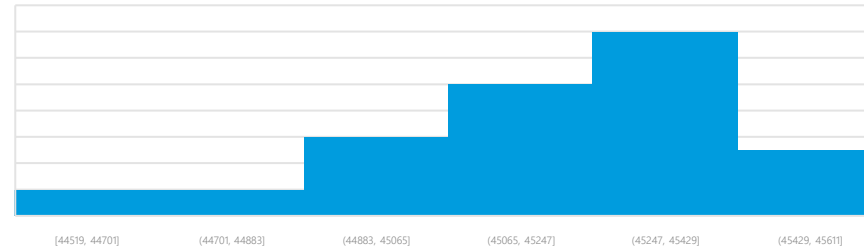


Screen for crystals

Collect diffraction data

Solve and refine structure

New Synergy-ED publications (by 6 months since Nov 2021)

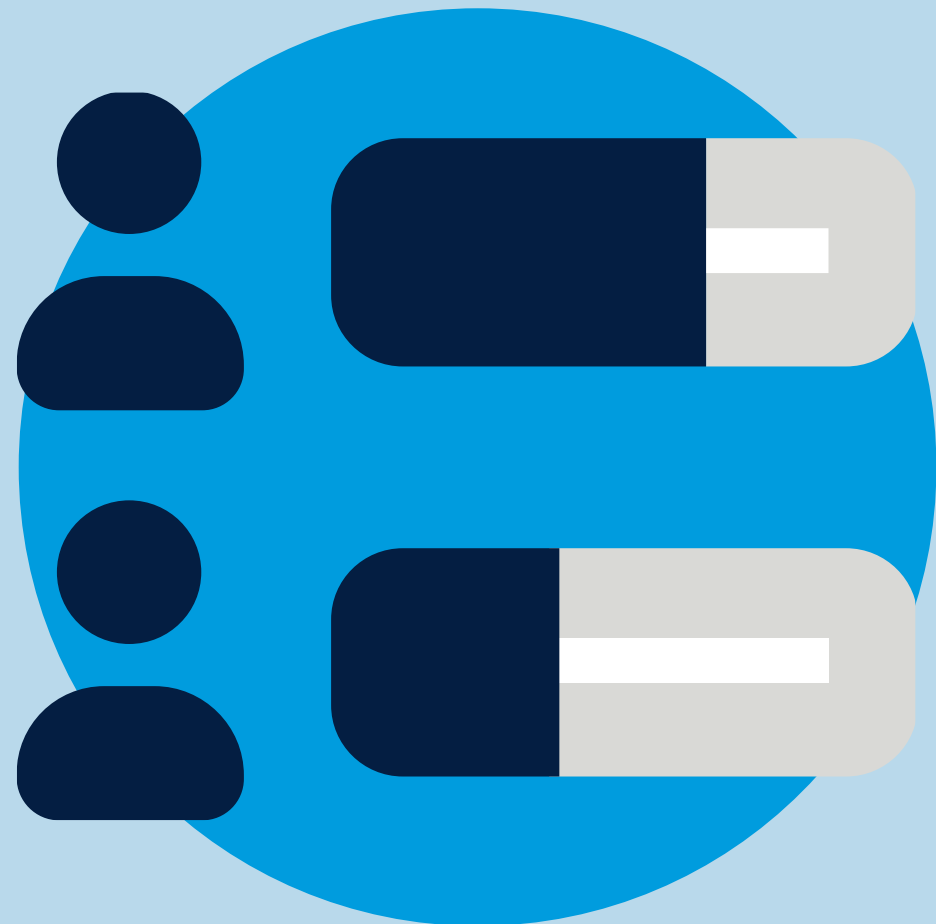


<https://rigaku.com/resources/publication-library>
>50 Synergy-ED peer-reviewed publications as of December 2024

...many more in preparation

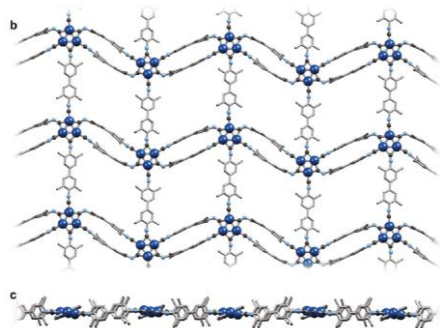
Polling Question

#1

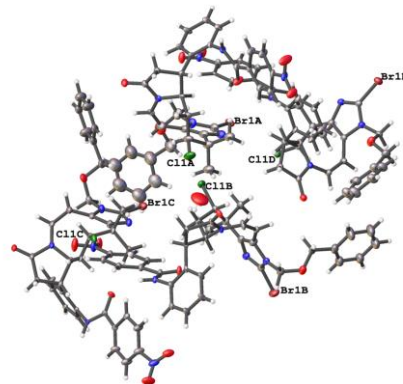


2. Sample preparation strategies

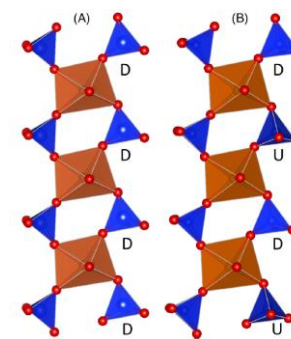
Sample Preparation: Types of Samples



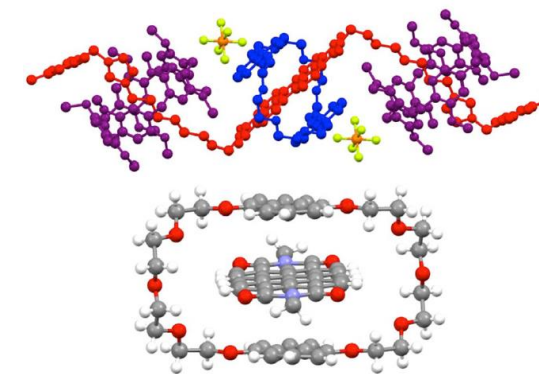
MOFs/COFs/ZIFs



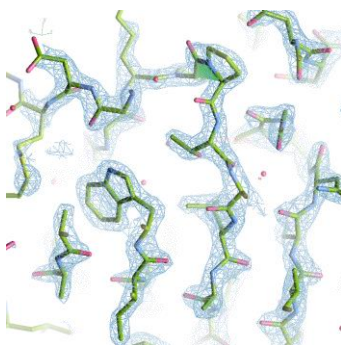
Natural products



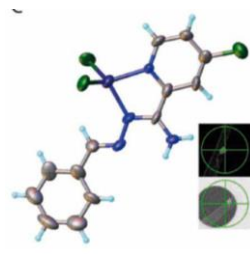
Ceramics



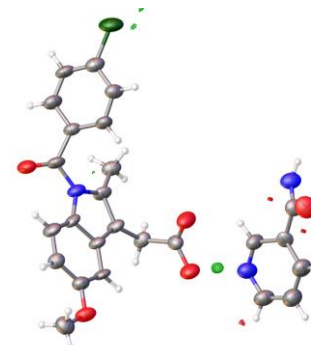
Macromolecular complexes



Proteins



Organometallics

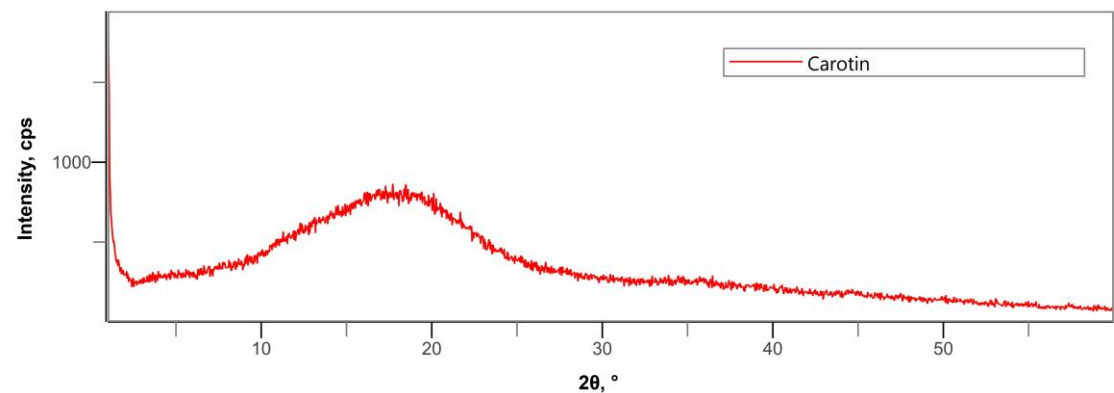
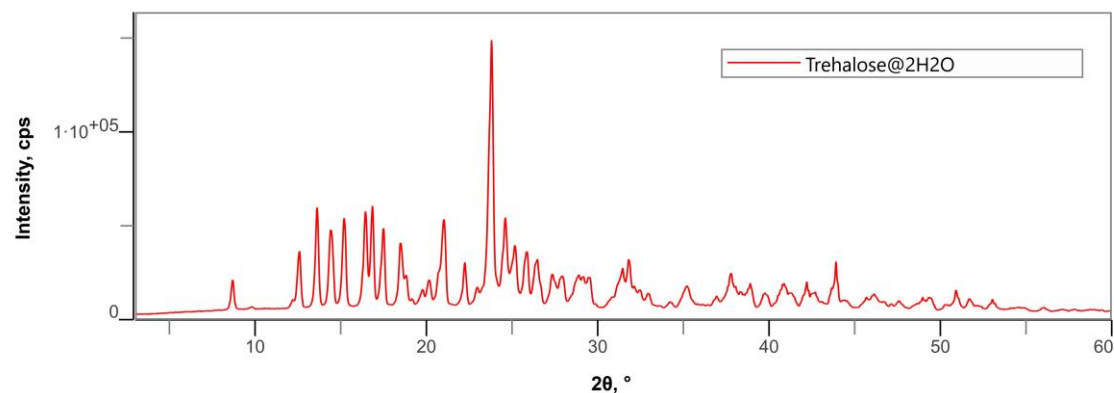


Pharmaceuticals

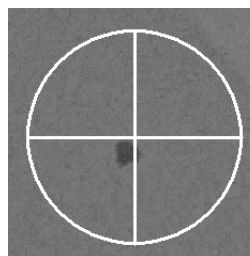
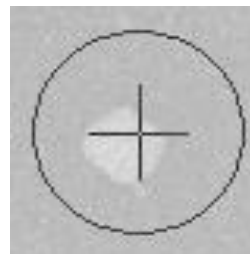
- Mixtures/impure
- Air-sensitive
- Sub-milligram quantities
- Require solvation

Sample Requirements: Crystallinity

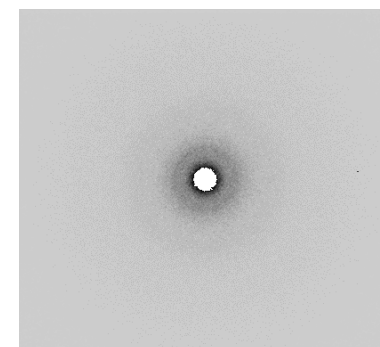
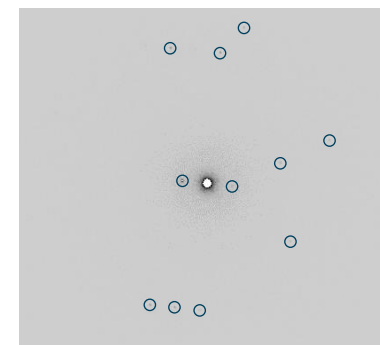
Powder pattern



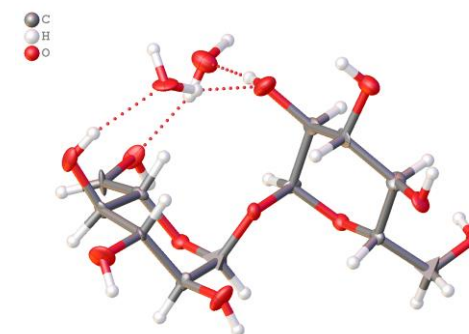
Particle



Electron diffraction



Structure solution

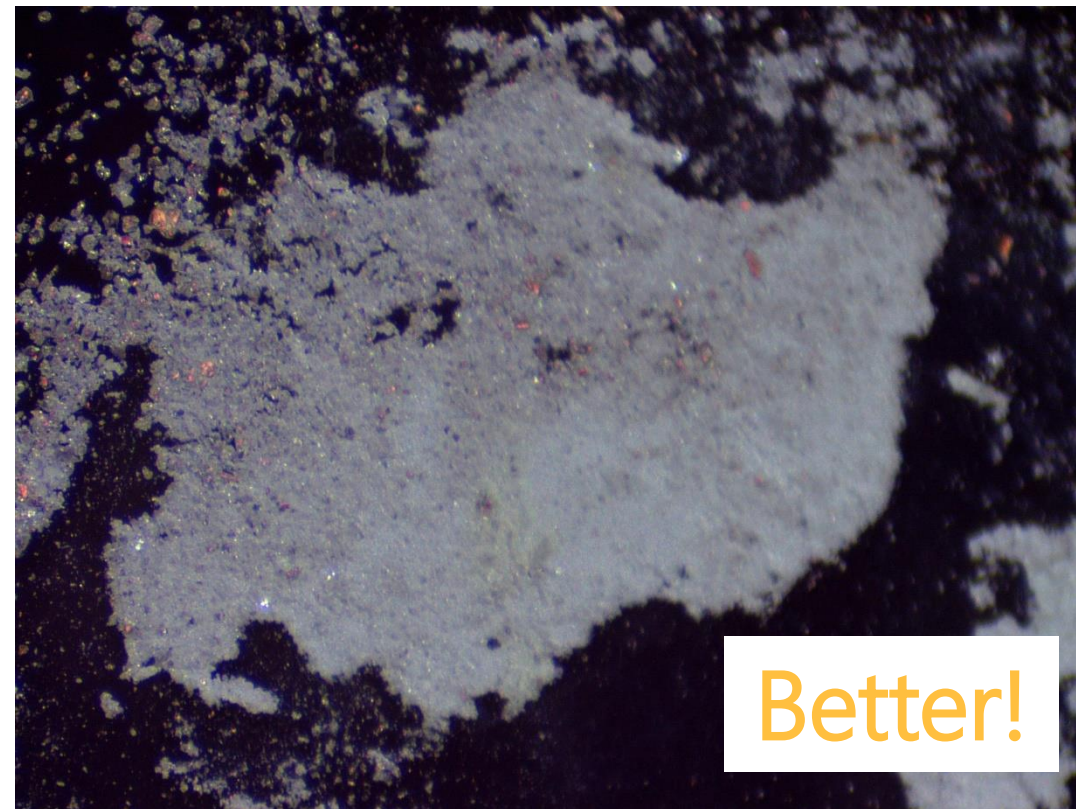
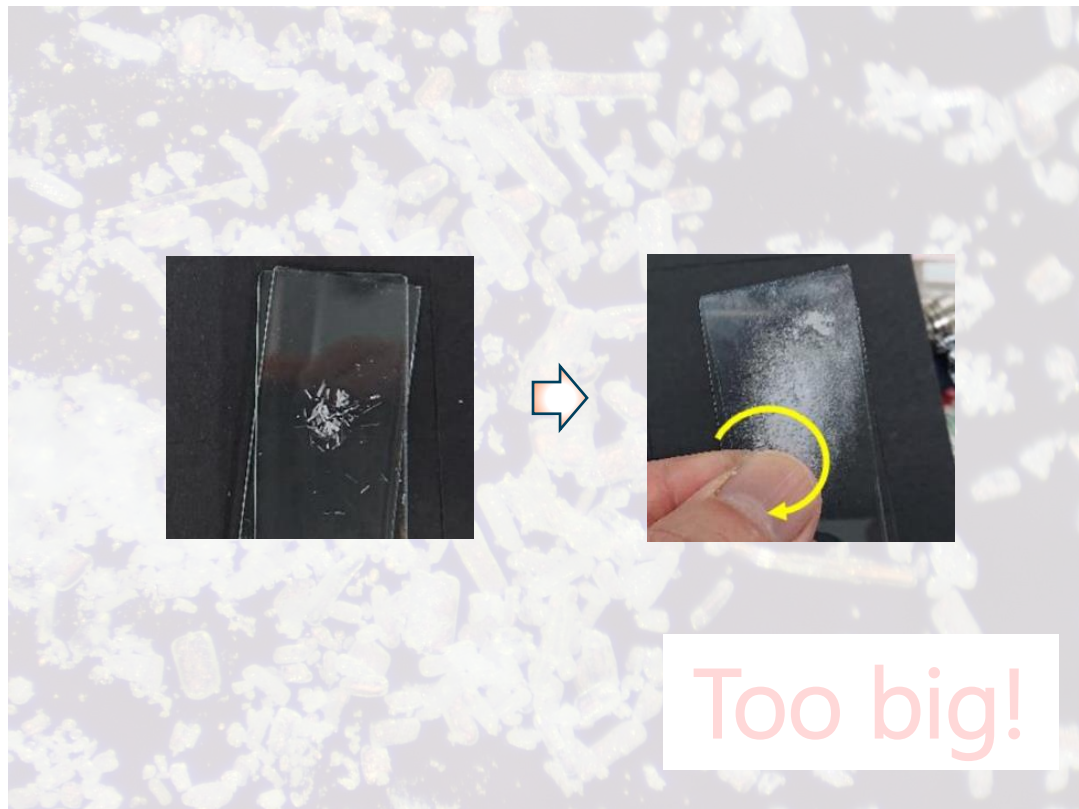


?

Crystallinity is a prerequisite for electron diffraction

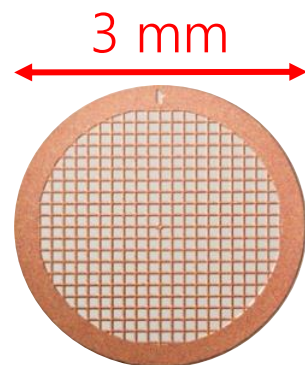
Sample Requirements: Particle Size

Particle size $< 1\ \mu\text{m}$?

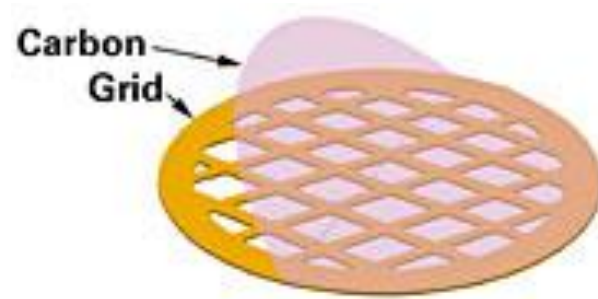


If crystals are visible by an optical microscope, they are too big for 3D ED. Crush or try SCXRD instead!

Sample Grids



200 mesh Cu TEM grid



Can purchase grids with varying:

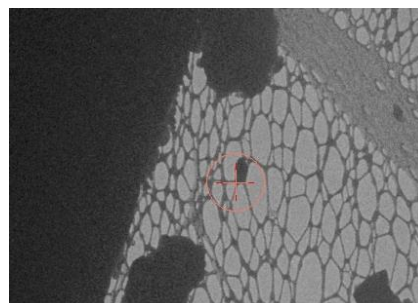
- Mesh size
- Metal grating
- Carbon / polymer supports

Continuous Carbon



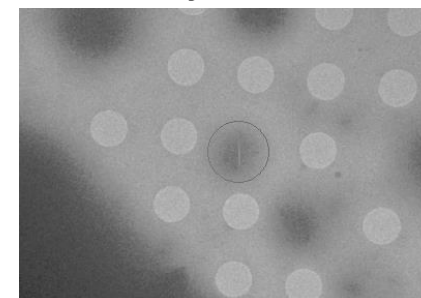
A good starting point

Lacey Carbon



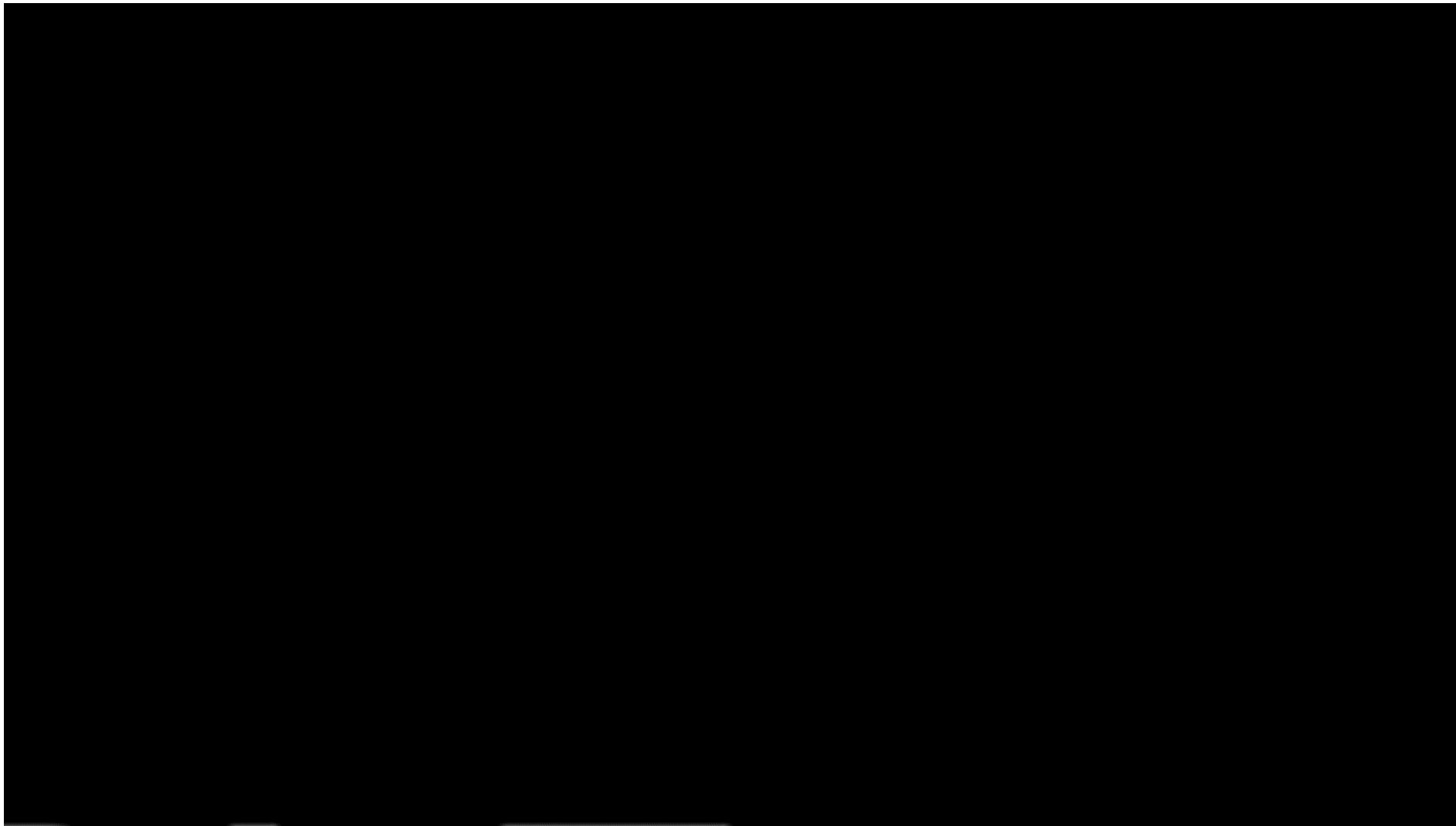
Can help with preferred orientation

Hole Array (Quantifoil)



Useful when freezing samples within solvent

Grid Preparation

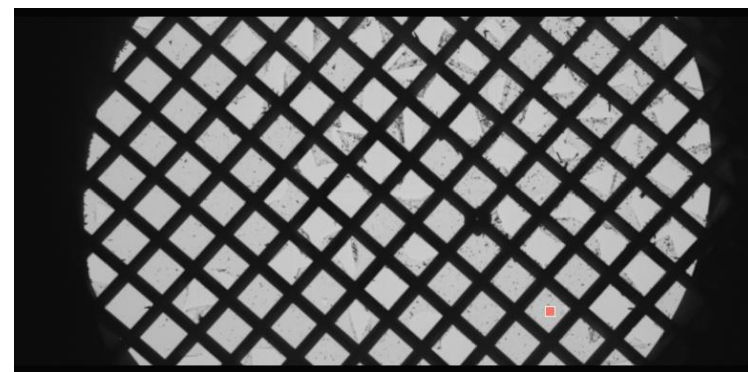
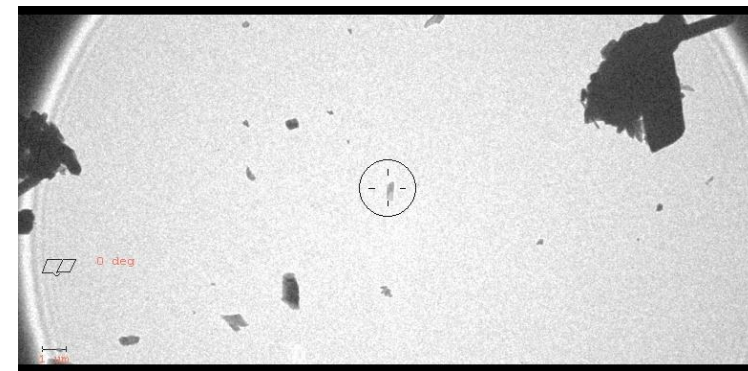
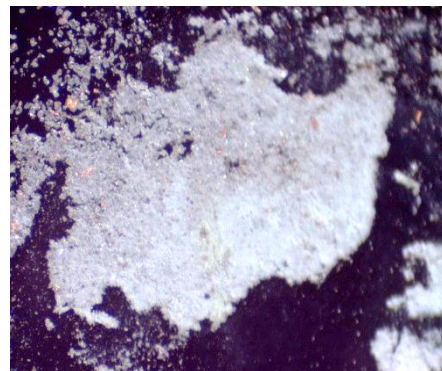


Grid Preparation

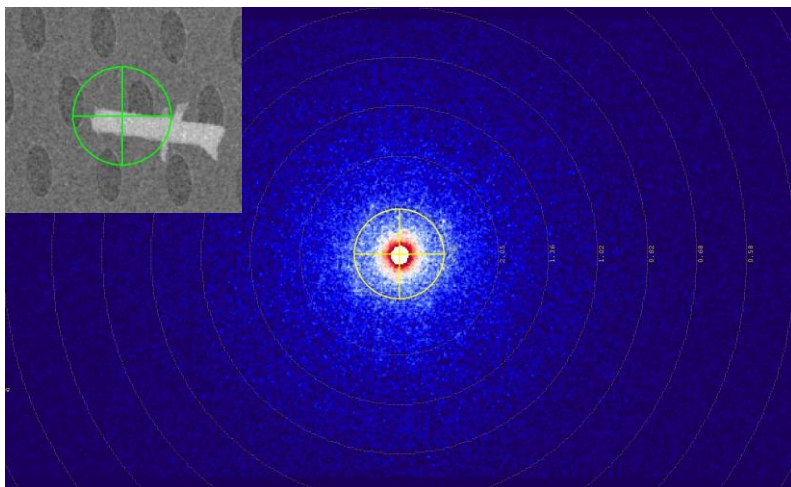
What makes a good sample grid?

- Crystallite size $< 1\ \mu\text{m}$
- No preferred orientation of crystallites
- Crystallite spacing of few μm

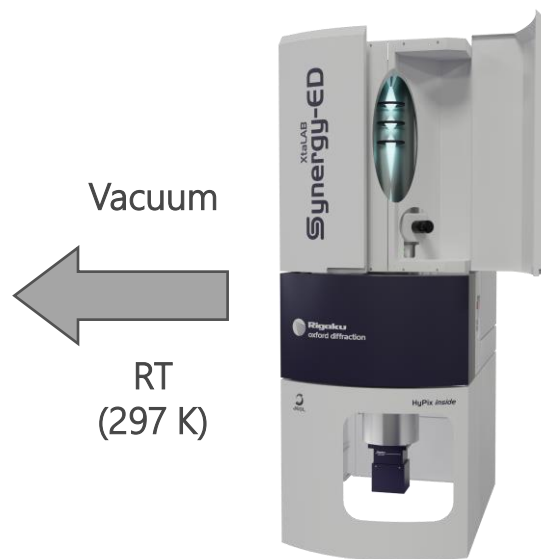
Grid is much larger than required sample volume: it's fine, if only parts are well loaded. The optimal regions can be identified during data collection.



Cryogenic Preparation

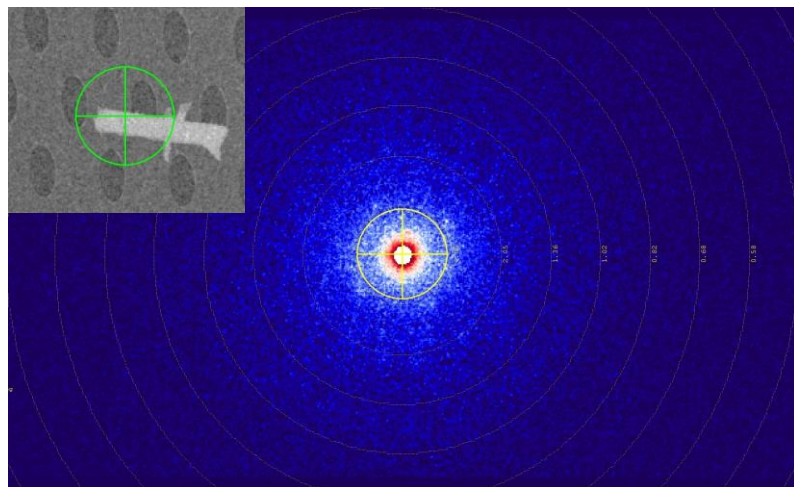


No useful diffraction data

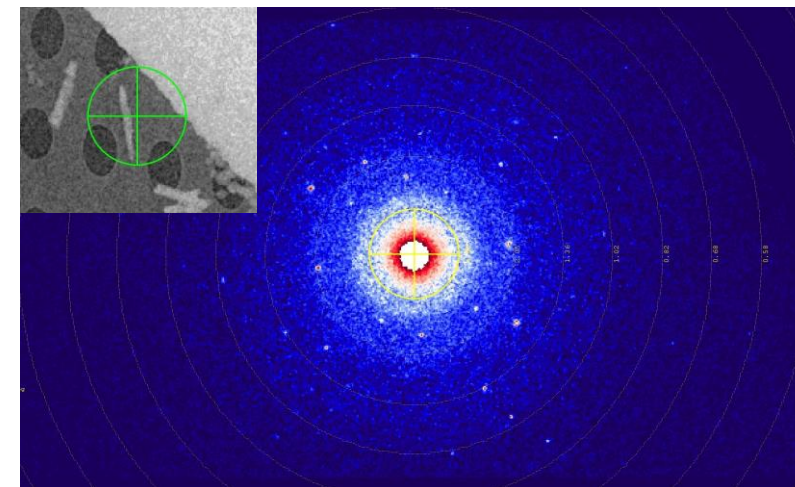
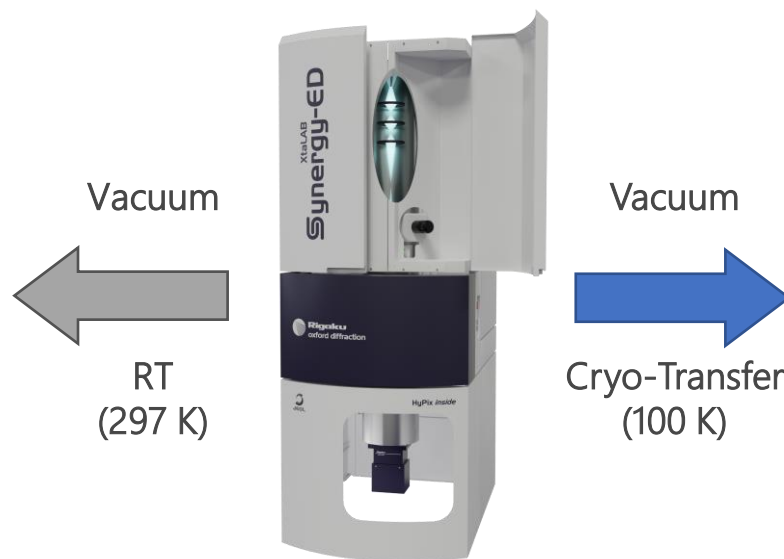


See strong diffraction >30 degrees in 2θ by powder, but nothing by ED?

Cryogenic Preparation



No useful diffraction data

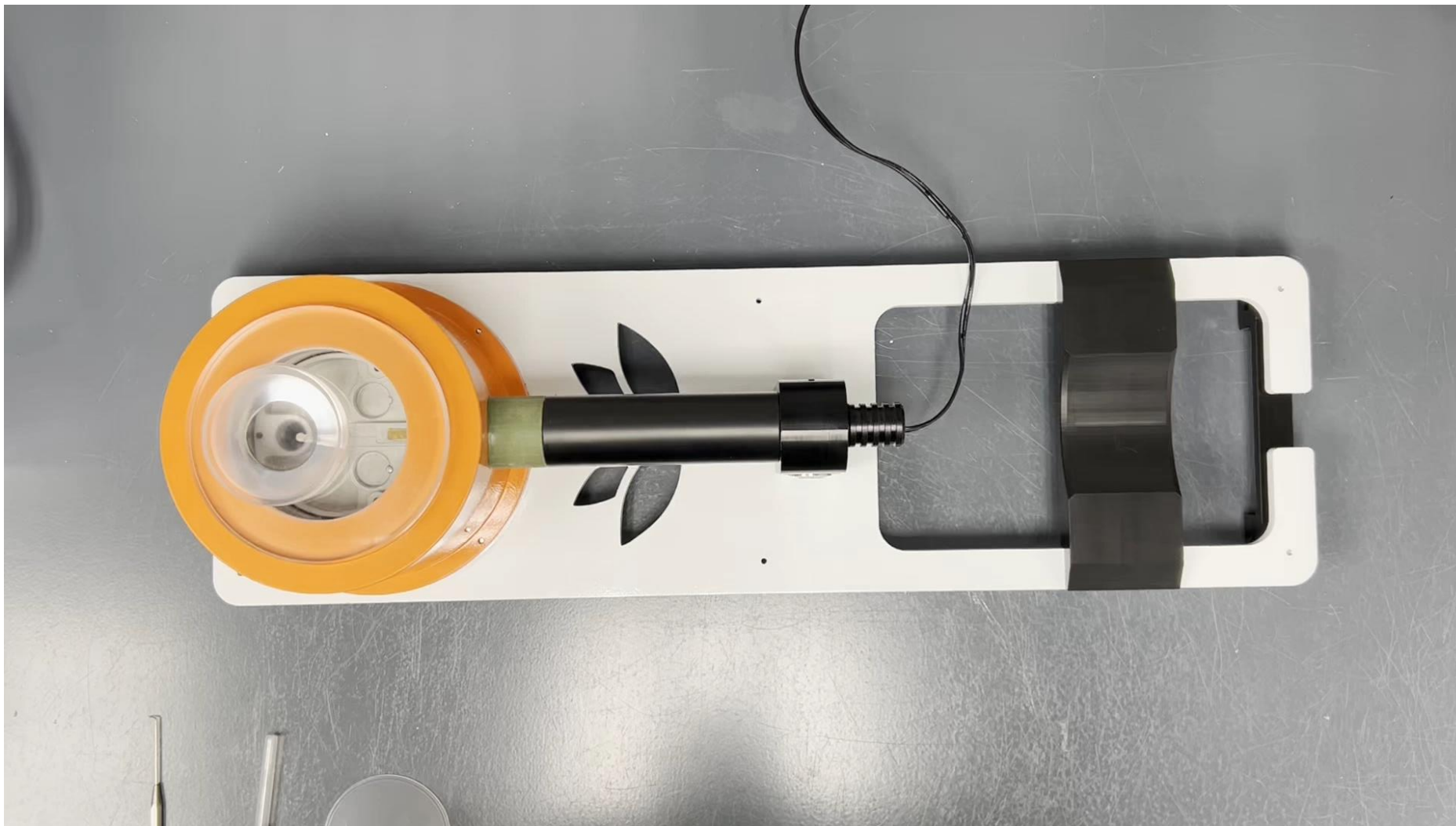


0.60 Å data, immediate structure

See strong diffraction >30 degrees in 2θ by powder, but nothing by ED?

It could be due to the strong vacuum!
A cryo transfer can address vacuum instability

Cryogenic Sample Loading

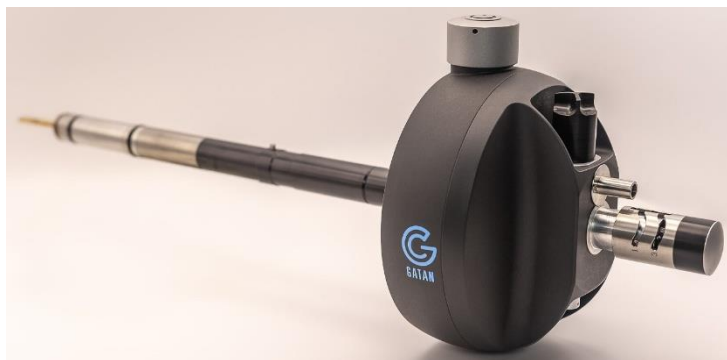


Sample Loading

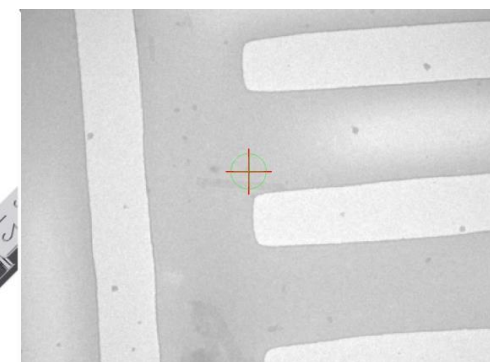


Sample Preparation: The Possibilities

Multi-specimen holders



Liquid/gas cells

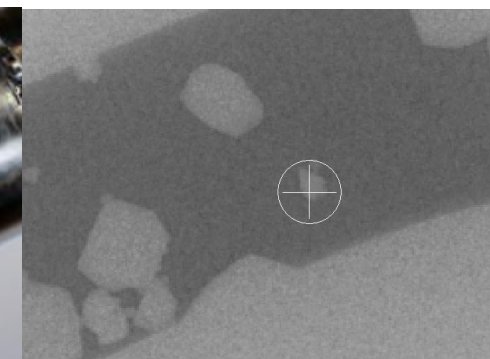


API crystal in solvent

Air-free holders



Heating holders



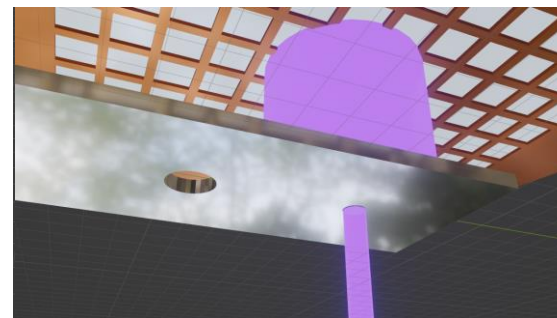
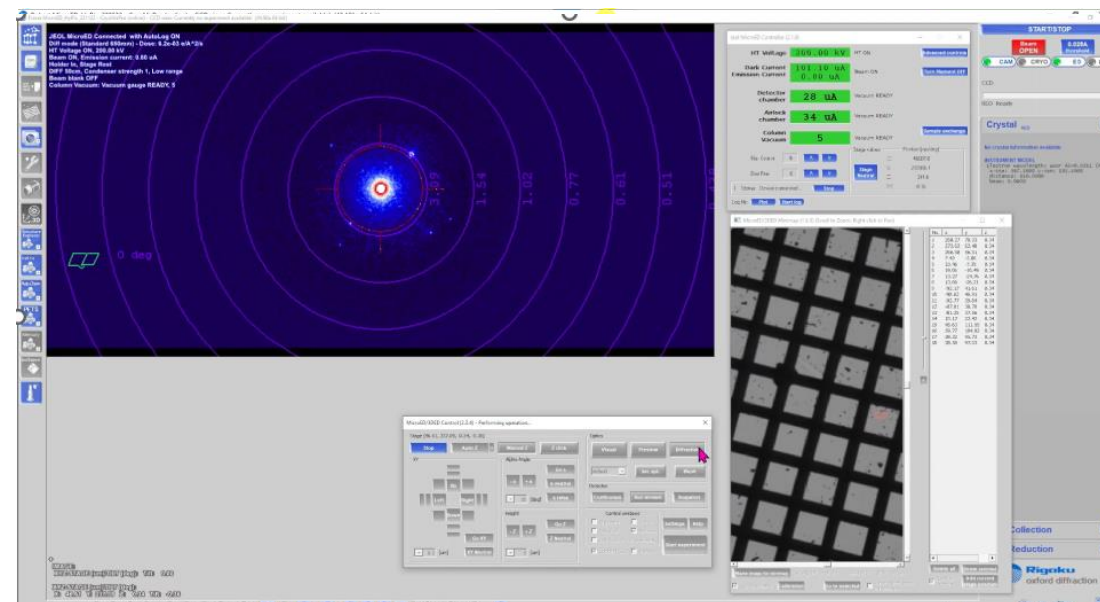
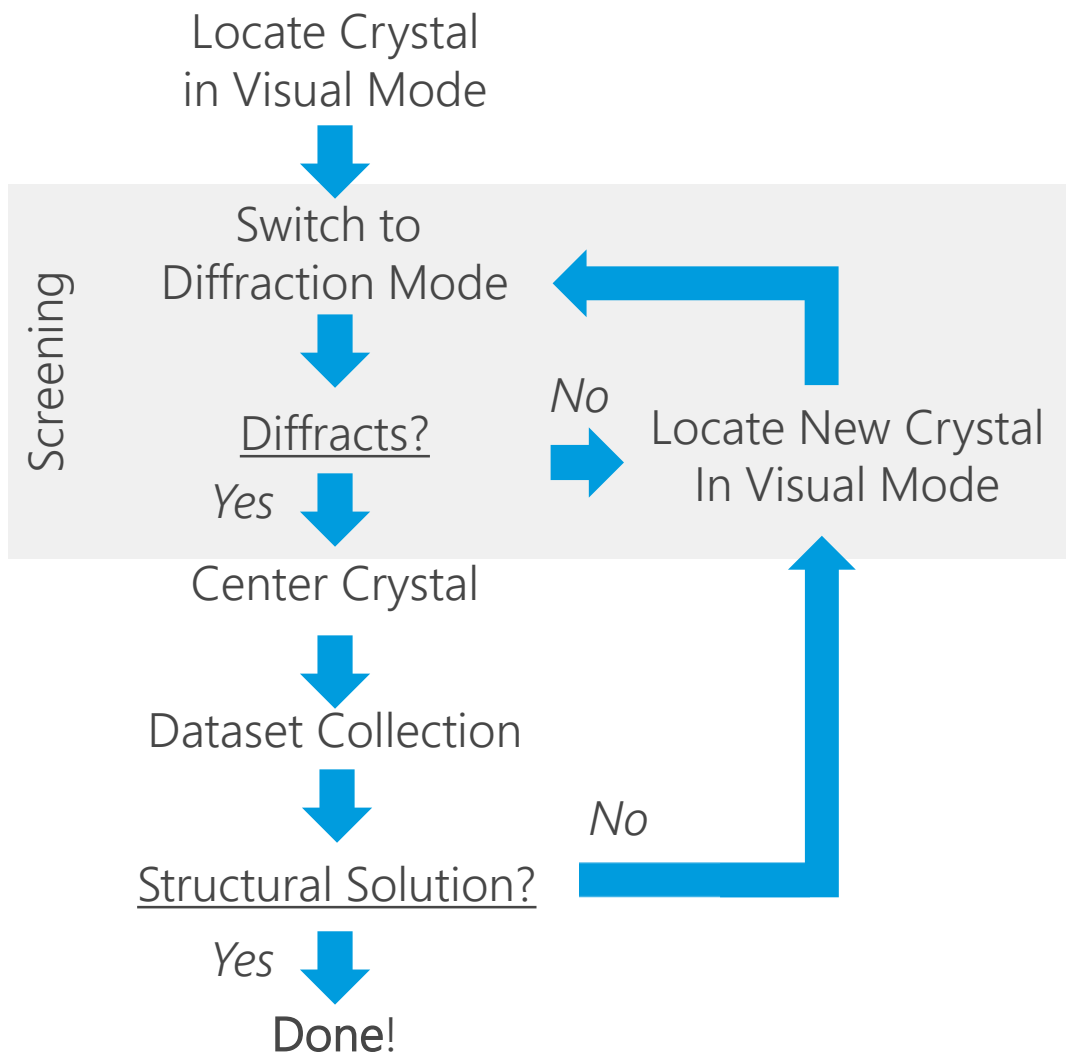
MOF heated to 200 °C

Questions?



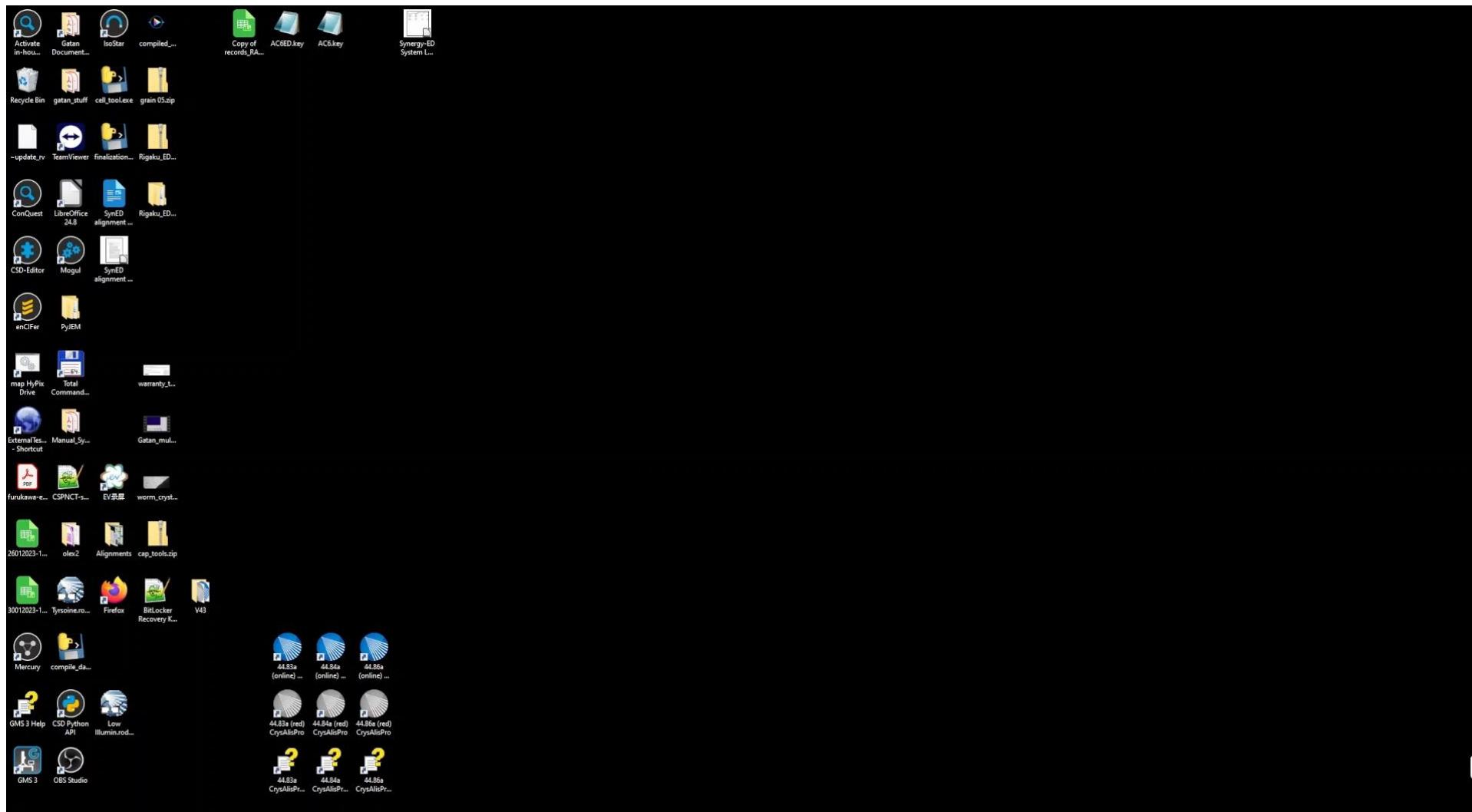
3. 3D ED data collection and processing with the Synergy-ED

Manual Data Collection Workflow

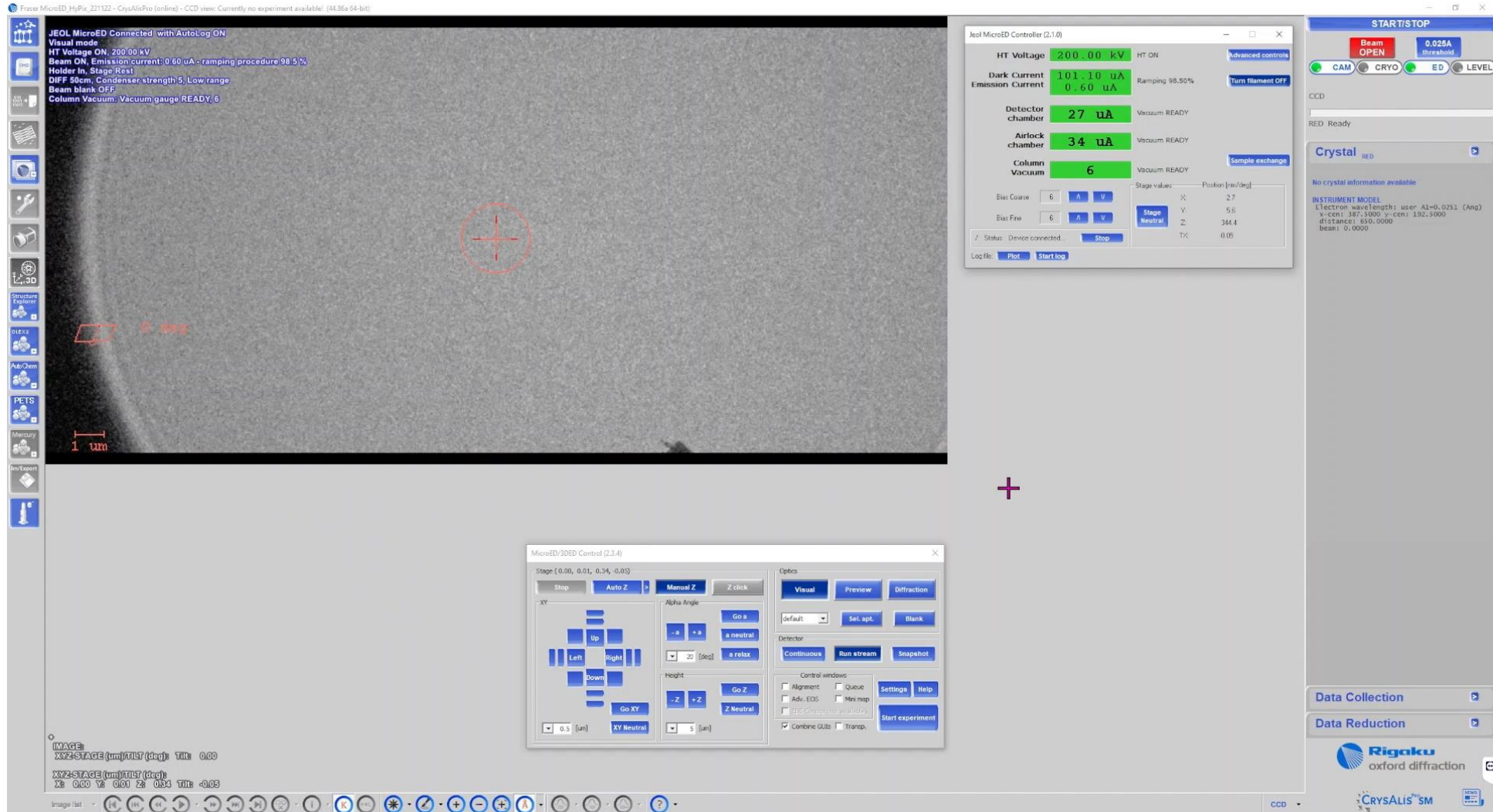


Selection Aperture

Getting Started



Minimap Collection



The screenshot displays the JEOL MicroED software interface, featuring a large central minimap collection window and several control panels.

JEOL MicroED Connected with AutoLog ON

Visual mode
 HT Voltage ON: 200.00 kV
 Beam ON, Emission current: 0.60 uA - ramping procedure 98.5 %
 Holder In, Stage Rest
 DIFF 50cm, Condenser strength 5, Low range
 Beam blank OFF
 Column Vacuum: Vacuum gauge READY: 6

JEOL MicroED Controller (2.1.0)

HT Voltage: 200.00 kV HT ON
 Dark Current: 101.10 uA
 Emission Current: 0.60 uA
 Detector chamber: 27 uA Vacuum READY
 Airlock chamber: 34 uA Vacuum READY
 Column Vacuum: 6 Vacuum READY
 Stage values: Position [mm/deg]
 X: 2.7
 Y: 5.6
 Z: 344.4
 TX: -0.05
 Status: Device connected
 Log file: Plot Start log

START/STOP

Beam OPEN 0.025A threshold
 CAM CRYO ED LEVEL
 CCD
 RED Ready
 Crystal RED
 No crystal information available
 INSTRUMENT MODEL
 Electron wavelength: user A3=0.0251 (Ang)
 x-cent: 387.5000 y-cent: 192.5000
 distance: 650.0000
 beam: 0.0000

MicroED/3DED Control (2.3.4)

Stage (0.00, 0.01, 0.34, -0.03)
 Stop Auto Z Manual Z Z click
 XY
 Up
 Left Right
 Down
 Go XY
 XY Neutral
 Alpha Angle
 -a +a
 a neutral
 a relax
 Height
 -Z +Z
 Go Z
 Z Neutral
 Optics
 Visual Preview Diffraction
 default Sel. apt. Blank
 Detector
 Continuous Run stream Snapshot
 Control windows
 Alignment Queue
 Adv. EOS Mini map
 EDS Control (if available)
 Combine QED Transp.
 Settings Help
 Start experiment

IMAGE
 XYZ-STAGE (um)/TILT (deg): Tilt: 0.00
 XYZ-STAGE (um)/TILT (deg)
 X: 0.00 Y: 0.01 Z: 0.34 Tilt: -0.03

Image list

Particle Screening

JEOL MicroED Connected with AutoLog ON
Visual mode
HT Voltage ON: 200.00 kV
Beam ON: Emission current: 0.80 uA
Holder in: Stage Moving
DIFF: 50cm, Condenser strength: 5, Low range
Beam blank OFF
Column Vacuum: Vacuum gauge READY: 3

0 deg

1.2um

MicroED/3DED Control (2.3.4)

Stage (44.25, 217.20, 0.34, -0.36)

Stop Auto Z Manual Z Z click

XY

Up

Left Right

Down

Go XY

XY Neutral

Alpha Angle

-a a

a neutral

20 [deg]

a relax

Height

-Z +Z

Go Z

Z Neutral

Optics

Visual Preview Diffraction

default Set. opt. Blank

Detector

Continuous Run stream Snapshot

Control windows

Alignment Queue

Adv. EDS Min map

RTS (Real Time Streaming)

Combine GUIs Transp.

Settings Help

Start experiment

IMAGE

XYZ-STAGE (um)/TILT (deg) Tilt: 0.00

XYZ-STAGE (um)/TILT (deg)

X: 44.25 Y: 217.20 Z: 0.34 Tilt: -0.36

Image list

JEOL MicroED Controller (2.1.0)

HT Voltage 200.00 kV HT ON

Dark Current 101.10 uA

Emission Current 0.80 uA

Beam ON

Detector chamber 26 uA Vacuum READY

Airlock chamber 33 uA Vacuum READY

Column Vacuum 5 Vacuum READY

Sample exchange

Stage values

Position [mm/deg]

X: 44.245

Y: 217.201

Z: 0.34

Tilt: -0.36

Status: Device connected

Stop

Log file: Plot Start log

START/STOP

Beam OPEN 0.025A threshold

CAM CRYO ED LEVEL

CCD

RED Ready

Crystal RED

No crystal information available

INSTRUMENT MODEL

Electron wavelength: user A3=0.0251 (Ang)

x-cen: 387.3000 y-cen: 192.3000

distance: 650.0000

beam: 0.0000

MicroED/3DED Minimap (1.6.1) (Scroll to Zoom, Right click to Pan)

No.	x	y	z
1	288.27	70.33	0.34
2	773.62	62.48	0.34
3	266.98	56.91	0.34
4	7.42	-3.86	0.34
5	13.46	-7.35	0.34
6	19.06	-16.49	0.34
7	13.27	-24.76	0.34
8	13.09	-26.21	0.34
9	92.17	41.61	0.34
10	-80.62	46.51	0.34
11	92.77	39.84	0.34
12	-87.01	38.70	0.34
13	81.28	37.56	0.34
14	10.17	22.42	0.34
15	46.63	111.35	0.34
16	39.77	104.92	0.34
17	38.32	95.73	0.34
18	38.58	97.03	0.34

Make image for minimap X: -120 Y: -186 um Selected X: 38 Y: 97 um

Rotate minimap Calibration Go to selected Show visited spots on minimap

Delete all Delete selected

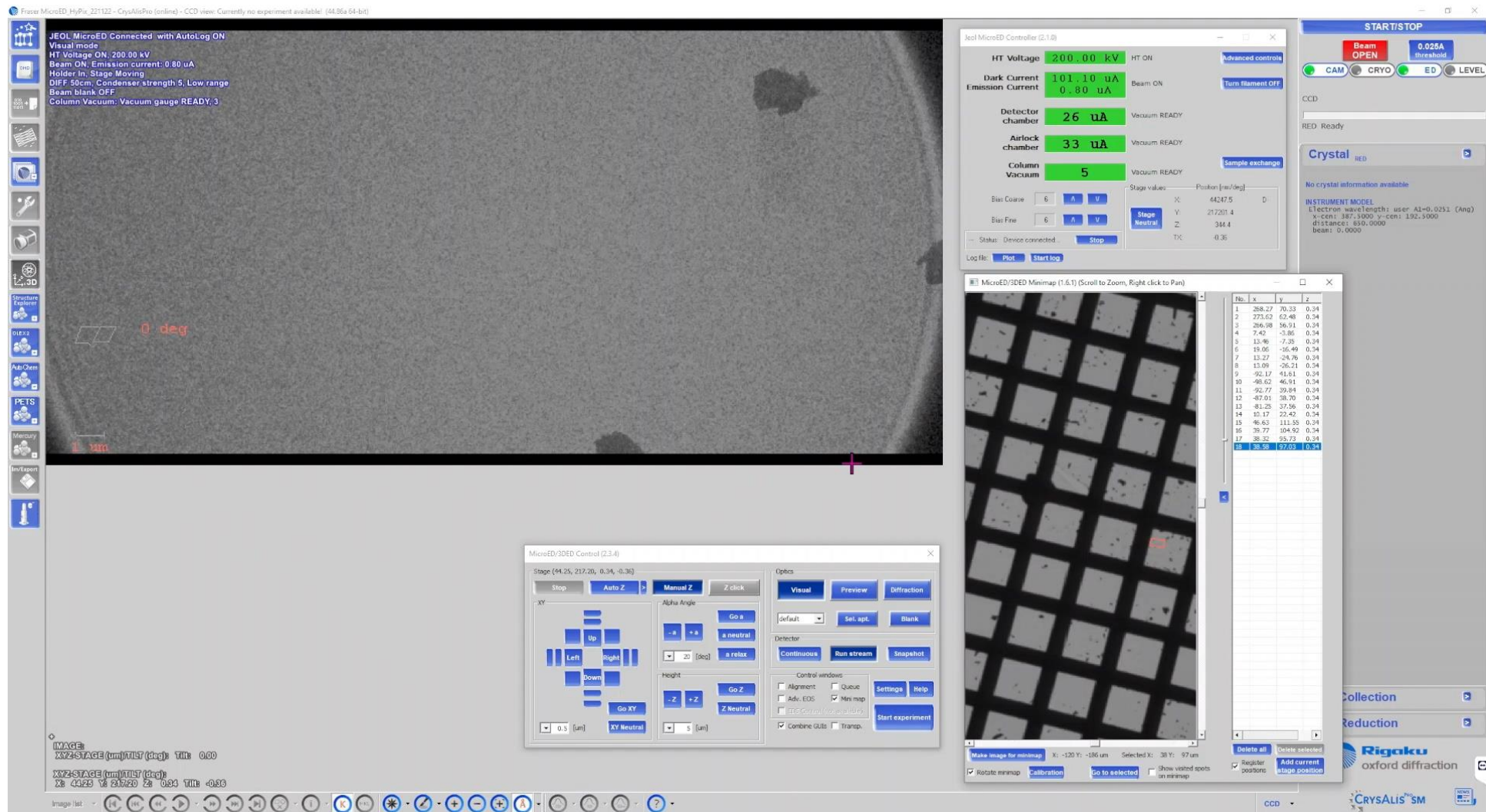
Register positions Add current stage position

CCD

Rigaku oxford diffraction

CRYALISSM

Particle Screening: Thickness



The screenshot displays the JEOL MicroED software interface, showing a large central image of a sample with a grid overlay. The interface includes several control panels and data windows.

JEOL MicroED Connected with AutoLog ON
 Visual mode
 HT Voltage ON: 200.00 kV
 Beam ON: Emission current: 0.80 uA
 Holder in: Stage Moving
 DIFF: 50cm, Condenser strength: 5, Low range
 Beam blank OFF
 Column Vacuum: Vacuum gauge READY: 3

JEOL MicroED Controller (2.1.0)
 HT Voltage: 200.00 kV HT ON
 Dark Current: 101.10 uA
 Emission Current: 0.80 uA
 Detector chamber: 26 uA Vacuum READY
 Airlock chamber: 33 uA Vacuum READY
 Column Vacuum: 5 Vacuum READY
 Stage values: X: 44247.5, Y: 217201.4, Z: 344.4, TX: 0.36
 Status: Device connected
 Log file: Plot Start log

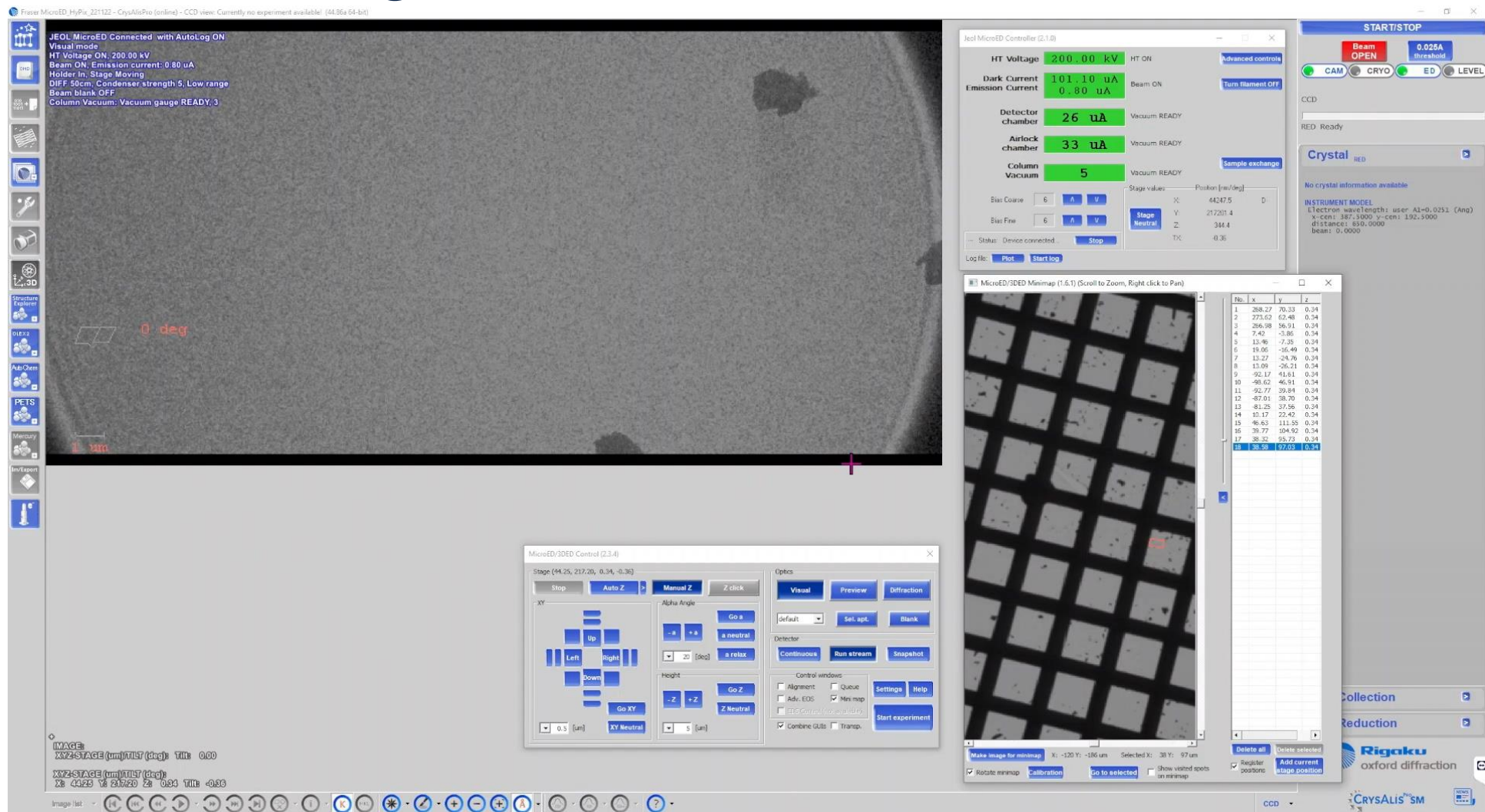
START/STOP
 Beam OPEN 0.025A threshold
 CAM CRYO ED LEVEL
 CCD
 RED Ready
 Crystal RED
 No crystal information available
 INSTRUMENT MODEL
 Electron wavelength: user A3=0.0251 (Ang)
 X-cent: 387.3000 Y-cent: 192.3000
 distance: 650.0000
 beam: 0.0000

MicroED/3DED Minimap (1.6.1)
 (Scroll to Zoom, Right click to Pan)
 No. x y z
 1 288.27 70.33 0.34
 2 773.62 62.48 0.34
 3 266.98 56.91 0.34
 4 7.42 -5.86 0.34
 5 13.46 -7.25 0.34
 6 19.06 -16.49 0.34
 7 13.22 -24.76 0.34
 8 13.09 -26.21 0.34
 9 92.17 41.61 0.34
 10 -80.62 46.51 0.34
 11 92.77 39.84 0.34
 12 -87.01 38.70 0.34
 13 81.28 37.56 0.34
 14 10.17 22.42 0.34
 15 46.63 111.35 0.34
 16 39.77 104.92 0.34
 17 38.32 95.73 0.34
 18 38.58 97.03 0.34

MicroED/3DED Control (2.3.4)
 Stage (44.25, 217.20, 0.34, -0.36)
 Stop Auto Z Manual Z Z click
 XY
 Up
 Left Right
 Down
 Go XY
 XY Neutral
 0.5 (um)
 Alpha Angle
 -a a
 a neutral
 20 (deg)
 a relax
 Height
 -Z +Z
 Go Z
 Z Neutral
 5 (um)
 Optics
 Visual Preview Diffraction
 default Set. opt. Blank
 Detector
 Continuous Run stream Snapshot
 Control windows
 Alignment Queue
 Adv. EDS Min map
 RTIS Control (no auto)
 Combine Gifs Transp.
 Settings Help
 Start experiment

IMAGE
 XYZ-STAGE (um)/TILT (deg) Tilt: 0.00
 XYZ-STAGE (um)/TILT (deg)
 X: 4425 Y: 21720 Z: 0.34 Tilt: -0.00

Particle Screening: Thickness



JEOL MicroED Connected with AutoLog ON
Visual mode
HT Voltage ON, 200.00 kV
Beam ON, Emission current: 0.80 uA
Holder In, Stage Moving
DIFF 50cm, Condenser strength 5, Low range
Beam blank OFF
Column Vacuum: Vacuum gauge READY, 3

0 deg

100

MicroED/3DED Control (2.3.4)

Stage (44.25, 217.20, 0.34, -0.36)

Stop Auto Z Manual Z Z click

XY

Up

Left Right

Down

Go XY

XY Neutral

Alpha Angle

-a +a

a neutral

a relax

Height

-Z +Z

Z Neutral

Optics

Visual Preview Diffraction

default Set. apt. Blank

Detector

Continuous Run stream Snapshot

Control windows

Alignment Queue Mini map

Adv. EOS EDS TIG Control (not ready)

Combine GUIs Transp.

Settings Help

Start experiment

MicroED/3DED Minimap (1.6.1) (Scroll to Zoom, Right click to Pan)

No.	x	y	z
1	288.27	70.33	0.34
2	273.62	62.48	0.34
3	266.98	56.91	0.34
4	7.42	-3.86	0.34
5	13.46	-7.35	0.34
6	19.06	-16.49	0.34
7	13.27	-24.76	0.34
8	13.09	-26.21	0.34
9	92.17	41.61	0.34
10	-89.62	46.91	0.34
11	-92.77	39.84	0.34
12	-87.01	38.70	0.34
13	-81.25	37.56	0.34
14	10.17	22.42	0.34
15	-86.63	111.35	0.34
16	78.77	104.62	0.34
17	38.32	95.73	0.34
18	38.58	97.03	0.34

Make image for minimap X: -120 Y: -186 um Selected X: 38 Y: 97 um

Rotate minimap Calibration Go to selected Show unselected spots on minimap

Delete all Delete selected Register positions Add current stage position

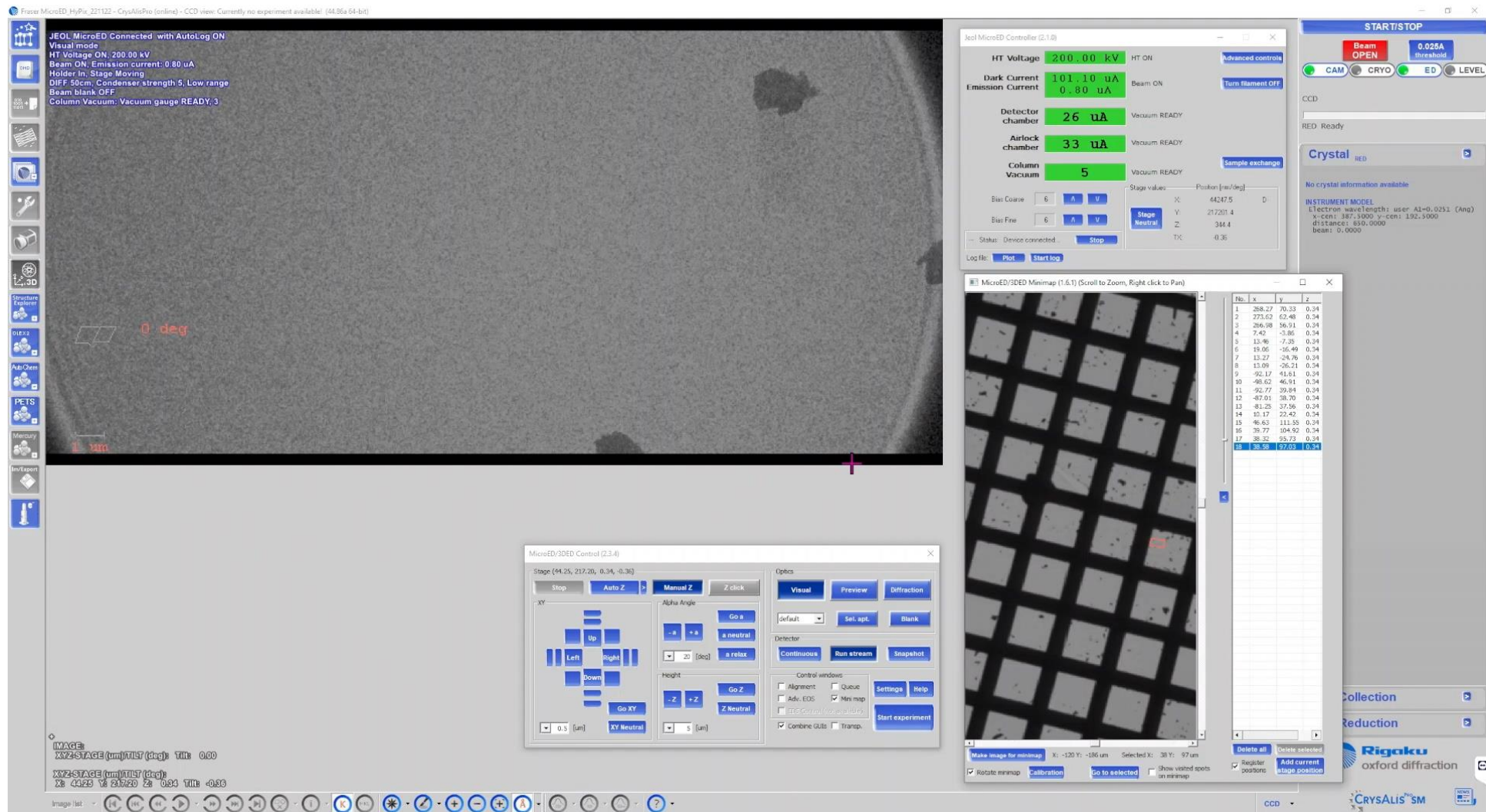
Collection

Reduction

Rigaku oxford diffraction

CRYALISSM

Particle Screening: Thickness



The screenshot displays the JEOL MicroED software interface, showing a large central image of a sample with a grid overlay. The interface includes several control panels and data windows.

JEOL MicroED Connected with AutoLog ON
 Visual mode
 HT Voltage ON: 200.00 kV
 Beam ON: Emission current: 0.80 uA
 Holder In/Stage Moving
 DIFF: 50cm, Condenser strength: 5, Low range
 Beam blank OFF
 Column Vacuum: Vacuum gauge READY: 3

JEOL MicroED Controller (2.1.0)
 HT Voltage: 200.00 kV HT ON
 Dark Current: 101.10 uA
 Emission Current: 0.80 uA
 Detector chamber: 26 uA Vacuum READY
 Airlock chamber: 33 uA Vacuum READY
 Column Vacuum: 5 Vacuum READY
 Stage values: X: 44247.5, Y: 217201.4, Z: 344.4, TX: 0.36
 Status: Device connected
 Log file: Plot Start log

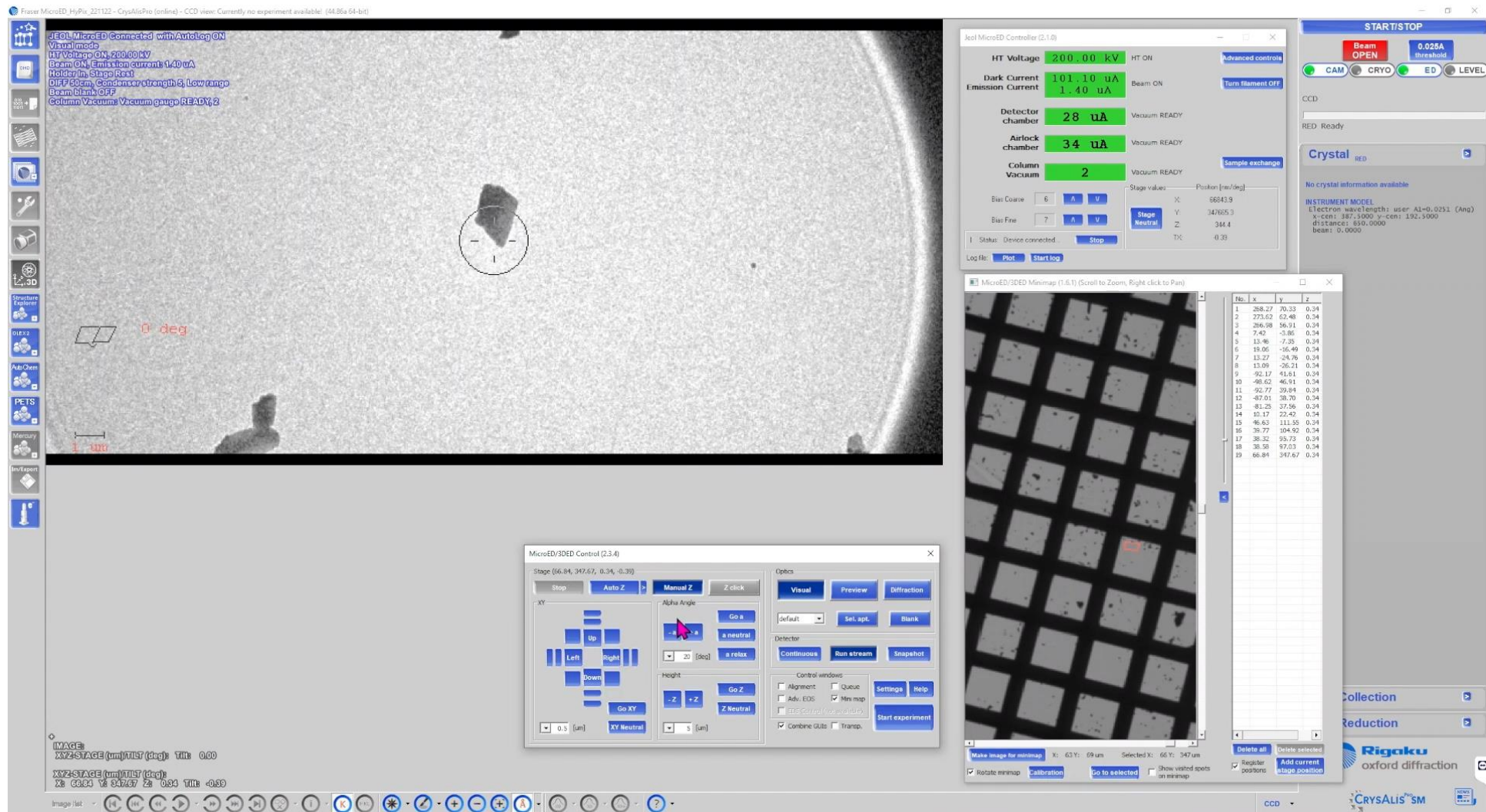
START/STOP
 Beam OPEN 0.025A threshold
 CAM CRYO ED LEVEL
 CCD
 RED Ready
 Crystal RED
 No crystal information available
 INSTRUMENT MODEL
 Electron wavelength: user A3=0.0251 (Ang)
 x-cen: 387.3000 y-cen: 192.3000
 distance: 650.0000
 beam: 0.0000

MicroED/3DED Minimap (1.6.1) (Scroll to Zoom, Right click to Pan)
 No. x y z
 1 288.27 70.33 0.34
 2 773.62 62.48 0.34
 3 266.98 56.91 0.34
 4 7.42 -5.86 0.34
 5 13.46 -7.25 0.34
 6 19.06 -16.49 0.34
 7 13.27 -24.76 0.34
 8 13.09 -26.21 0.34
 9 92.17 41.61 0.34
 10 -80.62 46.51 0.34
 11 92.77 39.84 0.34
 12 -87.01 38.70 0.34
 13 81.28 37.56 0.34
 14 10.17 22.42 0.34
 15 46.63 111.35 0.34
 16 39.77 104.92 0.34
 17 38.32 95.73 0.34
 18 38.58 97.03 0.34

MicroED/3DED Control (2.3.4)
 Stage (44.25, 217.20, 0.34, -0.36)
 Stop Auto Z Manual Z Z click
 XY
 Up
 Left Right
 Down
 Go XY
 XY Neutral
 Alpha Angle
 -a a
 a neutral
 20 [deg]
 a relax
 Height
 -Z +Z
 Go Z
 Z Neutral
 Optics
 Visual Preview Diffraction
 default Set. opt. Blank
 Detector
 Continuous Run stream Snapshot
 Control windows
 Alignment Queue
 Adv. EDS Min map
 RTSP (Real Time Spot Finding)
 Combine Gifs Transp.
 Settings Help
 Start experiment

IMAGE
 XYZ-STAGE (um)/TILT (deg) Tilt: 0.00
 XYZ-STAGE (um)/TILT (deg)
 X: 4424 Y: 21720 Z: 0.84 Tilt: -0.80

Data Collection Through Structural Solution



JEOL MicroED Connected with AutoLog ON
Visual mode
HT Voltage ON: 200.00 kV
Beam ON: Emission current: 1.40 uA
Holder in Stage: Rcs1
DIFF 60cm, Condenser strength: 5, Low range
Beam blank OFF
Column Vacuum: Vacuum gauge READY: 2

HT Voltage: 200.00 kV HT ON
Dark Current: 101.10 uA Beam ON
Emission Current: 1.40 uA Turn filament OFF
Detector chamber: 28 uA Vacuum READY
Airlock chamber: 34 uA Vacuum READY
Column Vacuum: 2 Vacuum READY
Sample exchange
Stage values: Position [mm/deg]
X: 66843.9
Y: 34765.3
Z: 344.4
TX: 0.33
Status: Device connected
Log file: Plot Start log

START/STOP
Beam OPEN 0.025A threshold
CAM CRYO ED LEVEL
CCD
RED Ready
Crystal RED
No crystal information available
INSTRUMENT MODEL
Electron wavelength: user A3=0.0251 (Ang)
x-cen: 387.3000 y-cen: 192.3000
distance: 650.0000
beam: 0.0000

MicroED/3DED Minimap (1.5.1) (Scroll to Zoom, Right click to Pan)

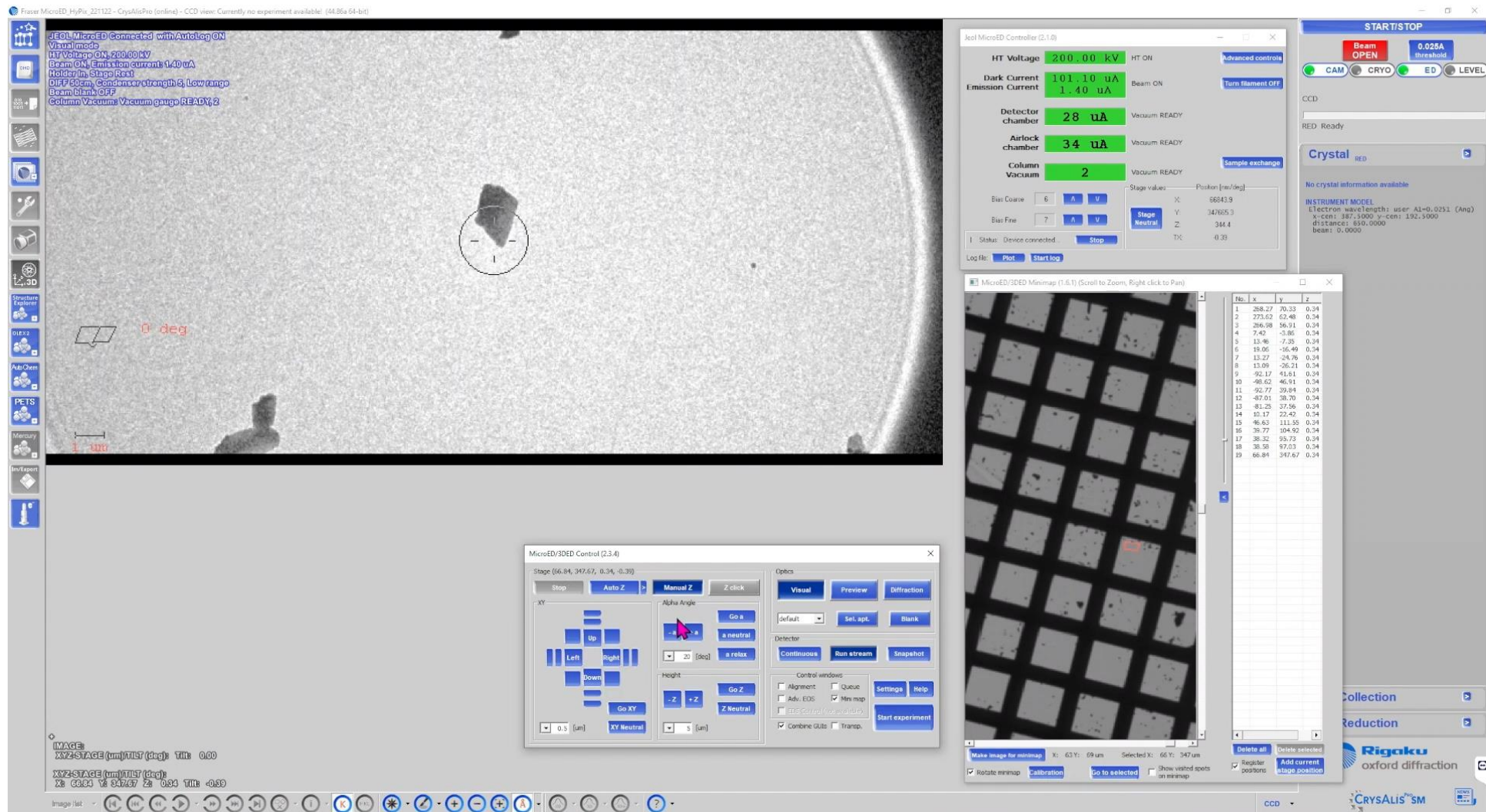
No.	x	y	z
1	288.27	70.33	0.34
2	273.62	62.40	0.34
3	266.98	56.91	0.34
4	7.42	-5.86	0.34
5	13.46	-7.35	0.34
6	19.06	-16.49	0.34
7	13.27	-24.76	0.34
8	13.09	-26.21	0.34
9	92.17	41.61	0.34
10	-88.62	46.51	0.34
11	92.77	39.84	0.34
12	-87.01	38.70	0.34
13	81.28	37.56	0.34
14	10.17	22.42	0.34
15	46.63	111.35	0.34
16	39.77	104.92	0.34
17	38.32	95.73	0.34
18	38.58	97.03	0.34
19	66.84	347.67	0.34

MicroED/3DED Control (2.3.4)
Stage (66.84, 347.67, 0.34, -0.30)
Stop Auto Z Manual Z Z click
Alpha Angle: 20 [deg]
Height: 5 [um]
XY: 0.5 [um]
XY Neutral
Go XY
Go Z
Z Neutral
Optics: Visual Preview Diffraction
Detector: default Set. apt. Blank
Continuous Run stream Snapshot
Control windows: Alignment Queue Adv. EDS Min map STS Control window
Combine Gulls Transp.
Settings Help
Start experiment

IMAGE
XYZ-STAGE (um)/TILT (deg) Tilt: 0.00
XYZ-STAGE (um)/TILT (deg)
X: 66.84 Y: 347.67 Z: 0.34 Tilt: -0.30

Collection
Reduction
Rigaku oxford diffraction
CRYSTALISSM

Data Collection Through Structural Solution



The screenshot displays the JEOL MicroED software interface, which is used for data collection and structural solution. The main window shows a large image of a sample, with a small inset showing a magnified view of a specific region. The interface includes various control panels and status indicators.

JEOL MicroED Connected with AutoLog ON
 Visual mode
 HT Voltage ON: 200.00 kV
 Beam ON: Emission current: 1.40 uA
 Holder in Stage OK
 DIFF 60cm, Condenser strength 5, Low range
 Beam blank OFF
 Column Vacuum: Vacuum gauge READY 2

JEOL MicroED Controller (2.1.0)
 HT Voltage: 200.00 kV HT ON
 Dark Current: 101.10 uA Beam ON
 Emission Current: 1.40 uA Turn filament OFF
 Detector chamber: 28 uA Vacuum READY
 Airlock chamber: 34 uA Vacuum READY
 Column Vacuum: 2 Vacuum READY
 Stage values: Position [mm/deg]
 X: 66843.9
 Y: 34765.3
 Z: 344.4
 TX: 0.33
 Status: Device connected
 Log file: Plot Start log

START/STOP
 Beam OPEN 0.025A threshold
 CAM CRYO ED LEVEL
 CCD
 RED Ready
 Crystal RED
 No crystal information available
 INSTRUMENT MODEL
 Electron wavelength: user A3=0.0251 (Ang)
 x-cen: 387.3000 y-cen: 192.3000
 distance: 650.0000
 beam: 0.0000

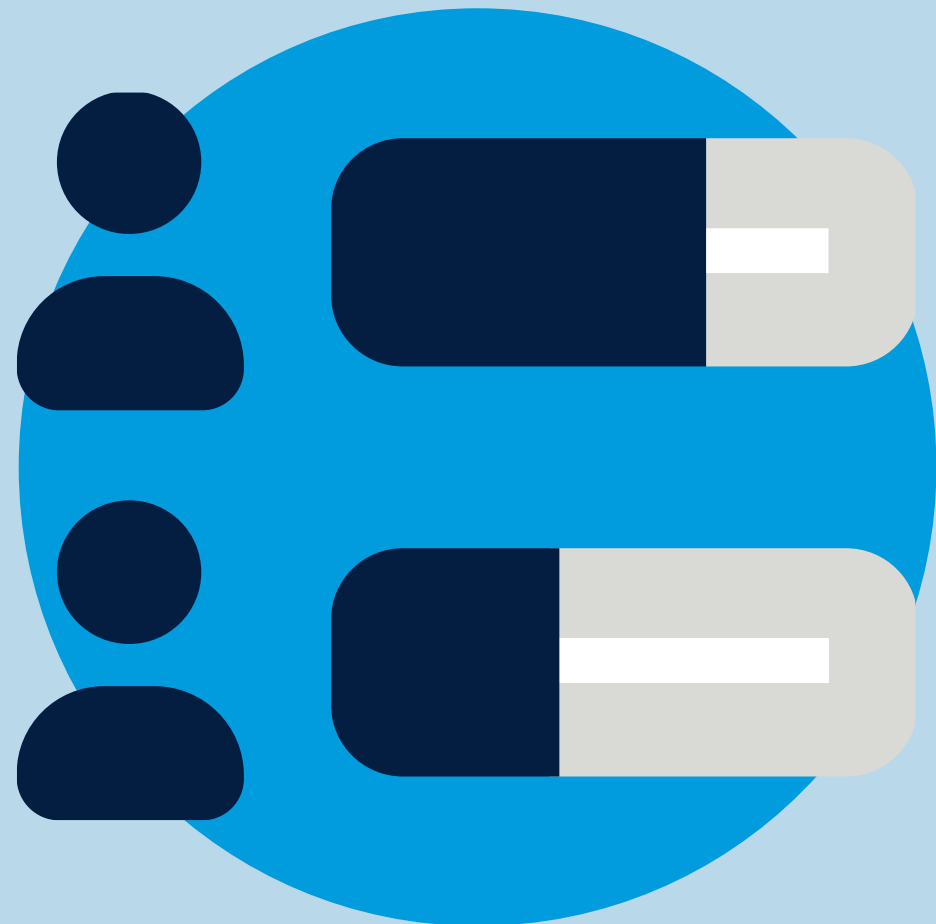
MicroED/3DED Minimap (1.5.1) (Scroll to Zoom, Right click to Pan)
 No. x y z
 1 288.27 70.33 0.34
 2 273.62 62.48 0.34
 3 266.98 56.91 0.34
 4 7.42 -5.86 0.34
 5 13.46 -7.35 0.34
 6 19.06 -16.49 0.34
 7 13.27 -24.76 0.34
 8 13.09 -26.21 0.34
 9 92.17 41.61 0.34
 10 -88.62 46.51 0.34
 11 92.77 39.84 0.34
 12 -87.01 38.70 0.34
 13 81.28 37.56 0.34
 14 10.17 22.42 0.34
 15 46.63 111.35 0.34
 16 39.77 104.92 0.34
 17 38.32 95.73 0.34
 18 38.58 97.03 0.34
 19 66.84 347.67 0.34

MicroED/3DED Control (2.3.4)
 Stage (66.84, 347.67, 0.34, -0.33)
 Stop Auto Z Manual Z Z click
 XY Left Right Up Down Go XY XY Neutral
 Alpha Angle: 20 [deg] Go a neutral a relax
 Height: -Z +Z Go Z Z Neutral
 Optics Visual Preview Diffraction
 Detector Continuous Run stream Snapshot
 Control windows: Alignment Queue Adv. EDS Min map STS Control window
 Settings Help Start experiment

IMAGE
 XYZ-STAGE (um)/TILT (deg) Tilt: 0.00
 XYZ-STAGE (um)/TILT (deg)
 X: 66.84 Y: 347.67 Z: 0.34 Tilt: -0.33

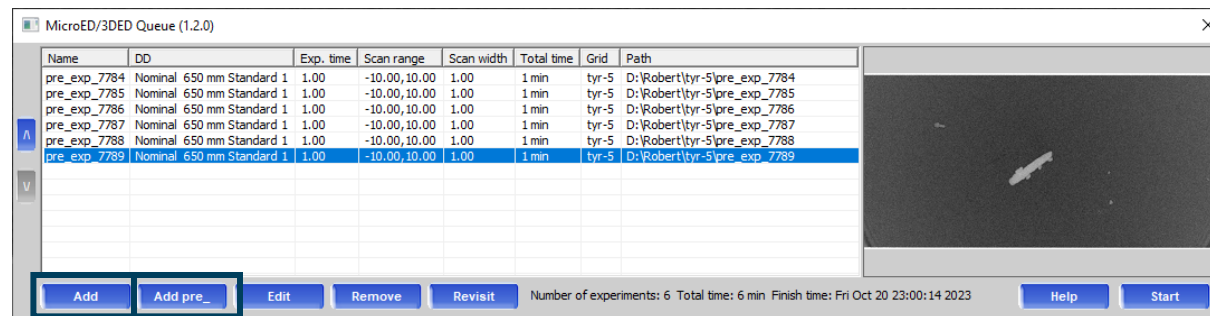
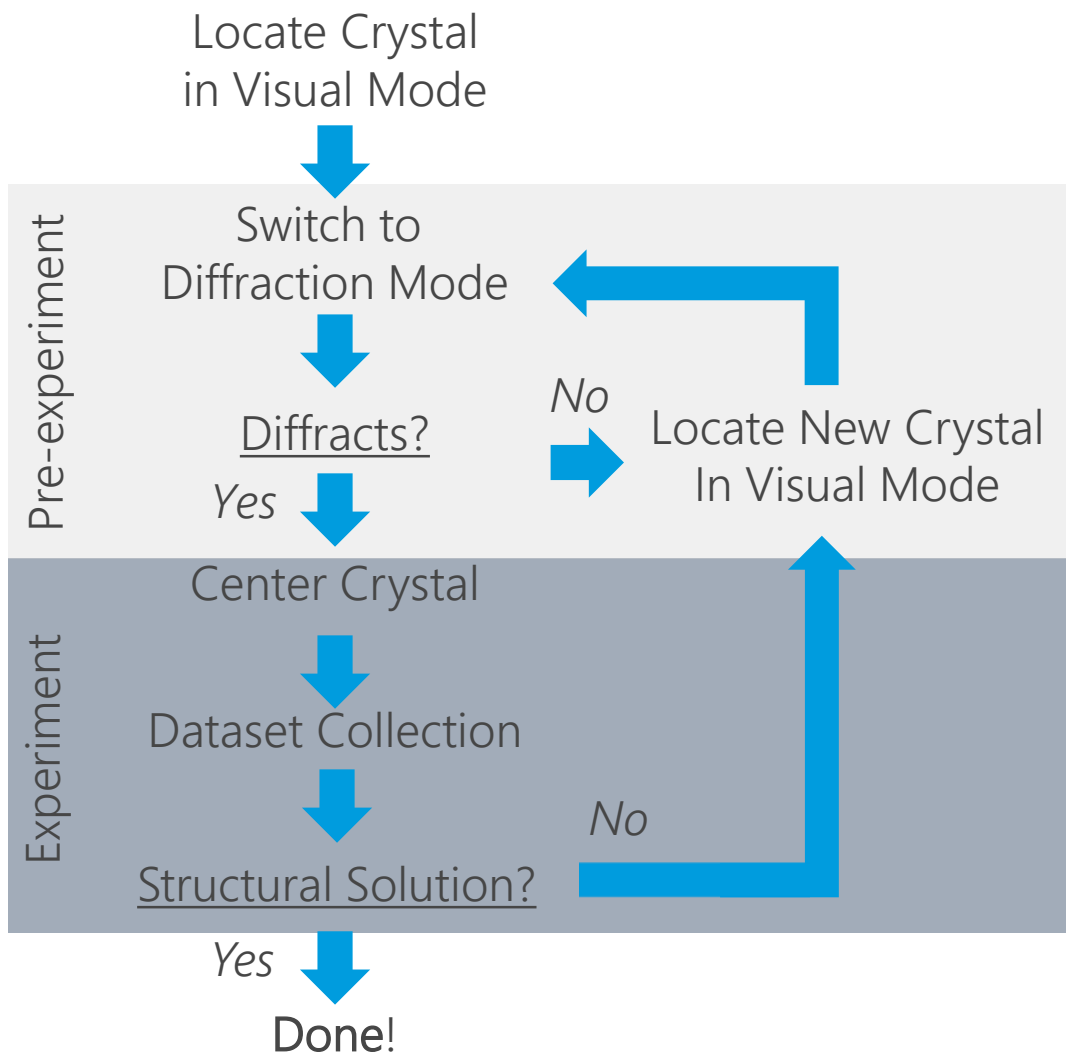
Polling Question

#2

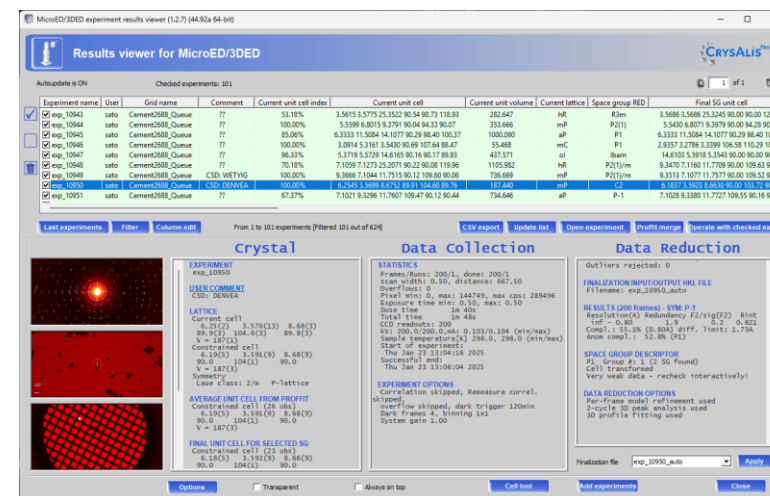


4. Use of automation

Workflow with Automation

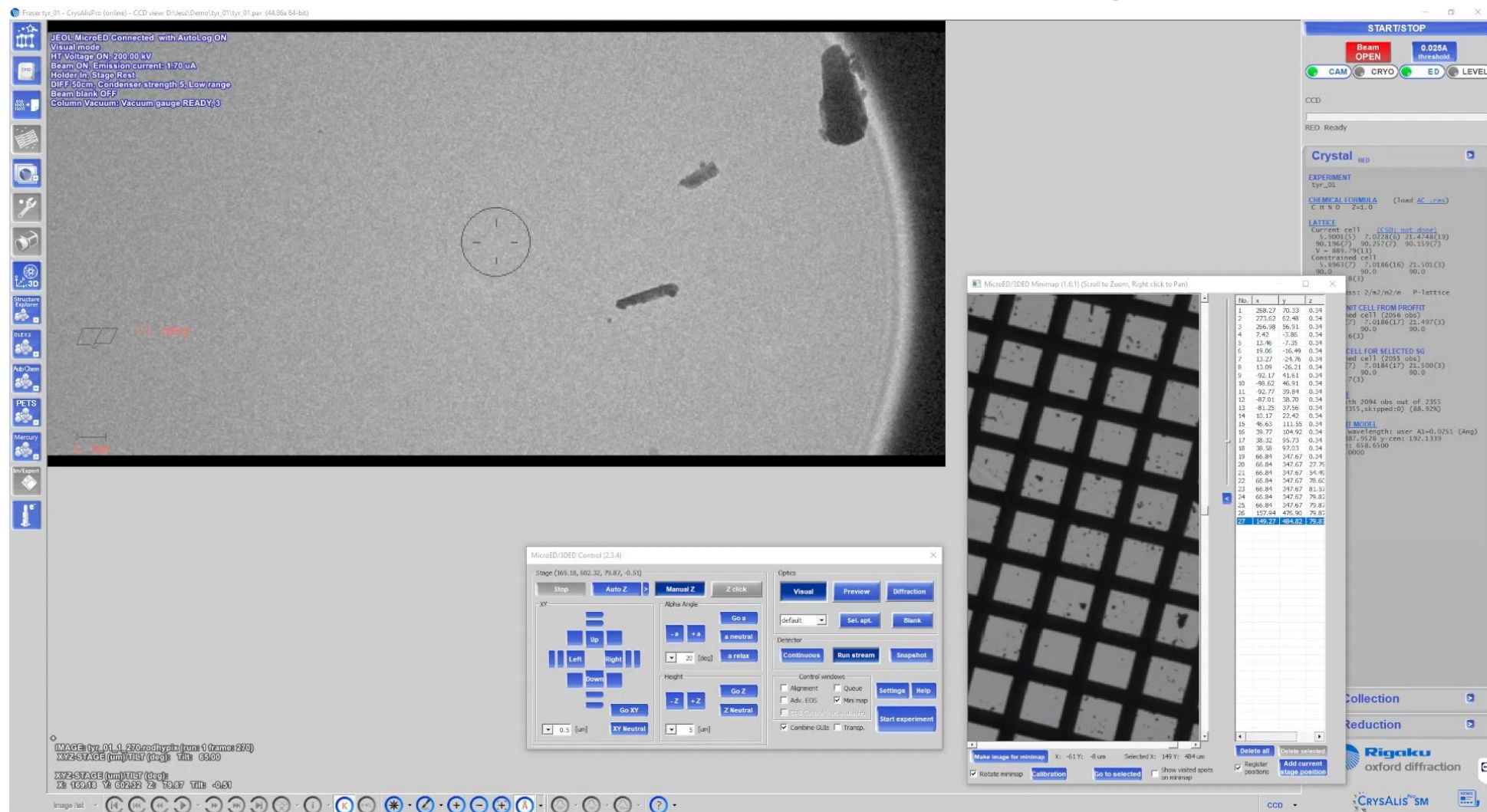


Queuing allows for unattended data collection



Results viewer streamlines decisions about data quality

Automation Workflows: Particle Screening



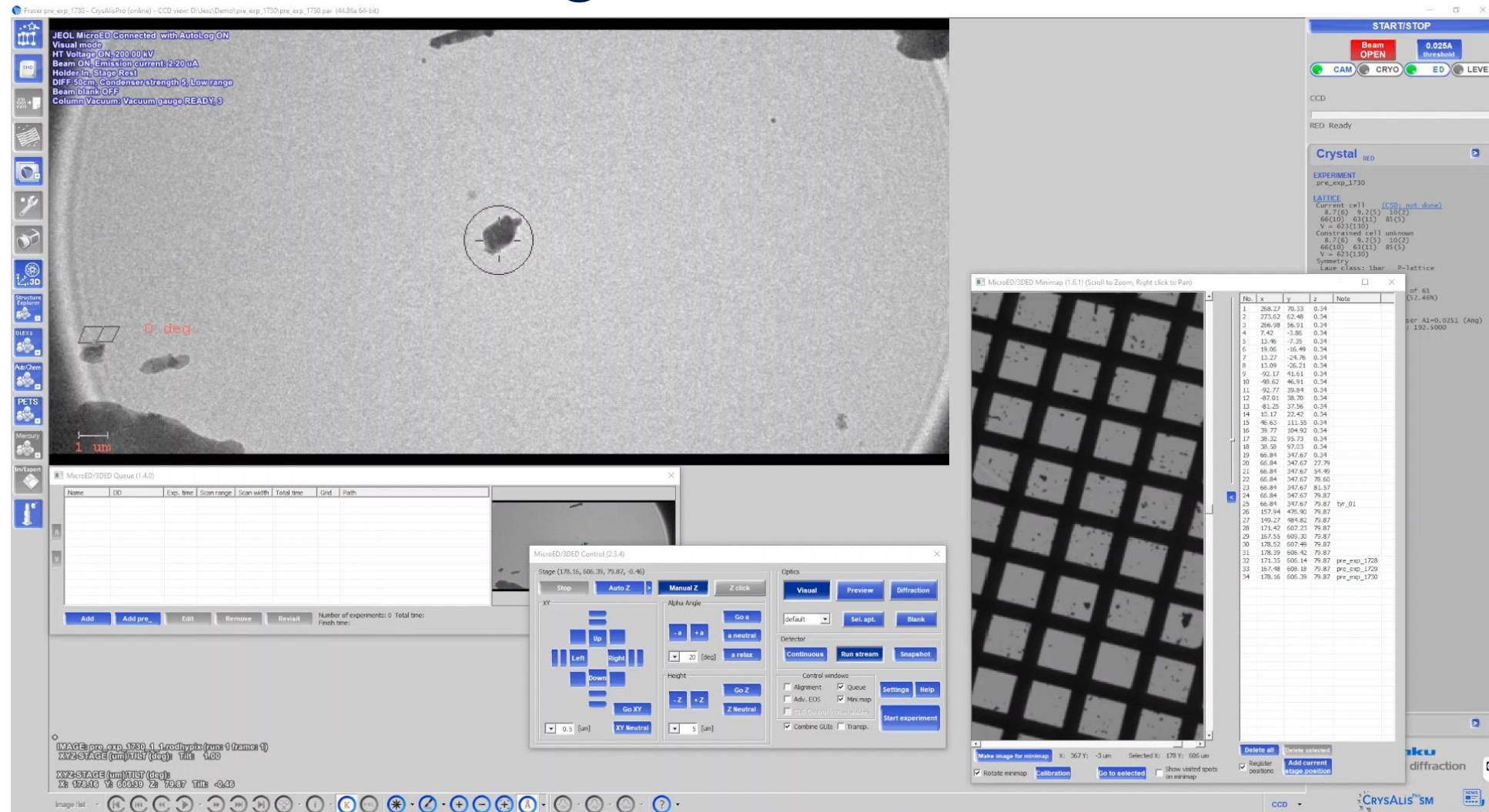
The screenshot displays the JEOL MicroED software interface, which is used for particle screening and data collection. The main window shows a large micrograph of a sample with a central crosshair. A sidebar on the left contains various control buttons for the microscope. A status bar at the bottom provides information about the current image and stage position.

Key components of the interface include:

- Top Panel:** Displays microscope status and parameters.
 - JEOL MicroED Connected with AutoLog ON
 - Visual mode
 - HT Voltage ON: 200.00 kV
 - Beam ON: Emission current: 1.70 uA
 - Holder in Stage/Rest
 - DIFF: 50cm, Condenser strength: 5, Low range
 - Beam blank OFF
 - Column Vacuum: Vacuum gauge READY, 3
- Right Panel:** Contains a 'START/STOP' section with buttons for 'Beam OPEN', '0.025A threshold', 'CAM', 'CRYO', 'ED', and 'LEVEL'. Below this is a 'Crystal' section with a 'RED' button and a table of experimental data.

No.	x	y	z
1	288.27	70.33	0.34
2	273.62	62.40	0.34
3	266.98	56.91	0.34
4	7.42	-3.86	0.34
5	15.96	-7.35	0.34
6	19.06	-16.49	0.34
7	13.57	-24.76	0.34
8	13.09	-26.21	0.34
9	-92.17	41.61	0.34
10	-80.62	46.91	0.34
11	-92.77	39.84	0.34
12	-87.01	38.70	0.34
13	-81.25	37.56	0.34
14	10.17	22.42	0.34
15	46.63	111.35	0.34
16	38.77	104.92	0.34
17	38.32	95.73	0.34
18	38.58	97.03	0.34
19	66.81	347.67	0.34
20	66.84	347.67	27.75
21	66.81	347.67	34.95
22	66.84	347.67	78.66
23	66.81	347.67	81.51
24	66.84	347.67	79.81
25	66.81	347.67	79.81
26	157.94	476.90	79.81
27	149.27	484.82	79.81
- Bottom Panel:** Contains a 'MicroED/3DED Control (2.3.4)' window with buttons for 'Stop', 'Auto Z', 'Manual Z', 'Z click', 'Go a', 'a neutral', 'a relax', 'Go XY', 'XY Neutral', 'Go Z', 'Z Neutral', 'Visual', 'Preview', 'Diffraction', 'Detector', 'Continuous', 'Run stream', 'Snapshot', 'Settings', 'Help', 'Start experiment', 'Combine GILTs', 'Transp.', 'Control windows', 'Alignment', 'Queue', 'Ads. EOS', 'Min map', 'EDS (only on sample)', 'Settings', 'Help', 'Start experiment'.

Automation: Centering and Data Collection



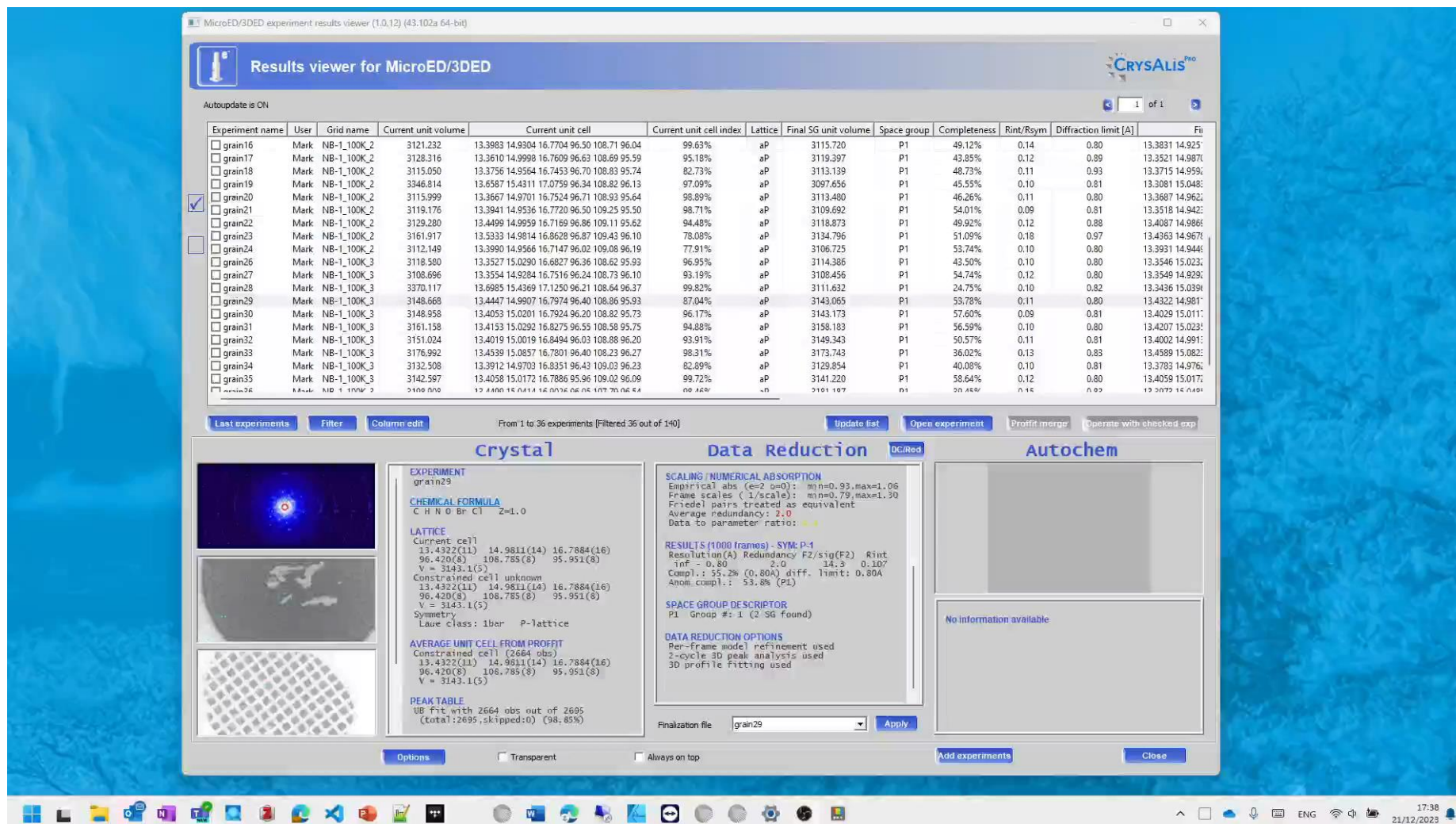
Results Viewer



Results Viewer



Structures from Merging



MicroED/3DED experiment results viewer (1.0.12) (43.102a 64-bit)

Results viewer for MicroED/3DED

Autoupdate is ON

Experiment name	User	Grid name	Current unit volume	Current unit cell	Current unit cell index	Lattice	Final SG unit volume	Space group	Completeness	Rint/Rsym	Diffraction limit [Å]	Fit
grain16	Mark	NB-1_100K_2	3121.232	13.3983 14.9304 16.7704 96.50 108.71 96.04	99.63%	aP	3115.720	P1	49.12%	0.14	0.80	13.3831 14.925
grain17	Mark	NB-1_100K_2	3128.316	13.3610 14.9998 16.7609 96.63 108.69 95.59	95.18%	aP	3119.397	P1	43.85%	0.12	0.89	13.3521 14.987
grain18	Mark	NB-1_100K_2	3115.050	13.3756 14.9564 16.7453 96.70 108.83 95.74	82.73%	aP	3113.139	P1	48.73%	0.11	0.93	13.3715 14.959
grain19	Mark	NB-1_100K_2	3346.814	13.6587 15.4311 17.0759 96.34 108.82 96.13	97.09%	aP	3097.656	P1	45.55%	0.10	0.81	13.3081 15.048
grain20	Mark	NB-1_100K_2	3115.999	13.3667 14.9701 16.7524 96.71 108.93 95.64	98.89%	aP	3113.480	P1	46.26%	0.11	0.80	13.3687 14.962
grain21	Mark	NB-1_100K_2	3118.176	13.3941 14.9536 16.7720 96.50 109.25 95.50	98.71%	aP	3109.692	P1	54.01%	0.09	0.81	13.3518 14.942
grain22	Mark	NB-1_100K_2	3129.280	13.4499 14.9959 16.7169 96.86 109.11 95.62	94.48%	aP	3118.873	P1	49.92%	0.12	0.88	13.4087 14.986
grain23	Mark	NB-1_100K_2	3161.917	13.5333 14.9814 16.8628 96.87 109.43 96.10	78.08%	aP	3134.796	P1	51.09%	0.18	0.97	13.4363 14.967
grain24	Mark	NB-1_100K_2	3112.149	13.3990 14.9566 16.7147 96.02 109.08 96.19	77.91%	aP	3106.725	P1	53.74%	0.10	0.80	13.3931 14.944
grain26	Mark	NB-1_100K_3	3118.580	13.3527 15.0290 16.6827 96.36 108.62 95.93	96.95%	aP	3114.386	P1	43.50%	0.10	0.80	13.3546 15.023
grain27	Mark	NB-1_100K_3	3108.696	13.3554 14.9284 16.7516 96.24 108.73 96.10	93.19%	aP	3108.456	P1	54.74%	0.12	0.80	13.3549 14.929
grain28	Mark	NB-1_100K_3	3370.117	13.6985 15.4369 17.1250 96.21 108.84 96.37	99.82%	aP	3111.632	P1	24.75%	0.10	0.82	13.3436 15.039
grain29	Mark	NB-1_100K_3	3148.668	13.4447 14.9907 16.7974 96.40 108.86 95.93	87.04%	aP	3143.065	P1	53.78%	0.11	0.80	13.4322 14.981
grain30	Mark	NB-1_100K_3	3148.958	13.4053 15.0201 16.7924 96.20 108.82 95.73	96.17%	aP	3143.173	P1	57.60%	0.09	0.81	13.4029 15.011
grain31	Mark	NB-1_100K_3	3161.158	13.4153 15.0292 16.8275 96.55 108.98 95.75	94.88%	aP	3158.183	P1	56.59%	0.10	0.80	13.4207 15.023
grain32	Mark	NB-1_100K_3	3151.024	13.4019 15.0019 16.8494 96.03 108.88 96.20	93.91%	aP	3149.343	P1	50.57%	0.11	0.81	13.4002 14.991
grain33	Mark	NB-1_100K_3	3176.992	13.4539 15.0857 16.7801 96.40 108.23 96.27	98.31%	aP	3173.743	P1	36.02%	0.13	0.83	13.4589 15.082
grain34	Mark	NB-1_100K_3	3132.508	13.3912 14.9703 16.8351 96.43 109.03 96.23	82.89%	aP	3129.854	P1	40.08%	0.10	0.81	13.3783 14.976
grain35	Mark	NB-1_100K_3	3142.597	13.4058 15.0172 16.7886 95.96 109.02 96.09	99.72%	aP	3141.220	P1	58.64%	0.12	0.80	13.4059 15.017

Crystal

EXPERIMENT
grain29

CHEMICAL FORMULA
C₁₂H₁₀O₂Br₂Cl₂ Z=1.0

LATTICE
Current cell
13.4322(11) 14.9811(14) 16.7884(16)
96.420(8) 108.785(8) 95.951(8)
V = 3143.1(5)
Constrained cell unknown
13.4322(11) 14.9811(14) 16.7884(16)
96.420(8) 108.785(8) 95.951(8)
V = 3143.1(5)
Symmetry
Laue Class: 1bar P-lattice

AVERAGE UNIT CELL FROM PROFIT
Constrained cell (2664 obs)
13.4322(11) 14.9811(14) 16.7884(16)
96.420(8) 108.785(8) 95.951(8)
V = 3143.1(5)

PEAK TABLE
UB fit with 2664 obs out of 2695
(total: 2695, skipped: 0) (98.85%)

Data Reduction

SCALING / NUMERICAL ABSORPTION
Empirical abs (w=2 o=0): min=0.93, max=1.06
Frame scales (1/scale): min=0.79, max=1.30
Friedel pairs treated as equivalent
Average redundancy: 2.0
Data to parameter ratio: 4.4

RESULTS (1000 frames) - SYM: P-1
Resolution(Å) Redundancy F2/sig(F2) Rint
min - 0.80 14.3 0.107
Comp.: 55.2% (0.80Å) diff. limit: 0.80Å
Anom. compl.: 53.8% (P1)

SPACE GROUP DESCRIPTOR
P1 Group #: 1 (2 SG found)

DATA REDUCTION OPTIONS
Per-frame model refinement used
2-cycle 3D peak analysis used
3D profile fitting used

Finalization file: grain29 Apply

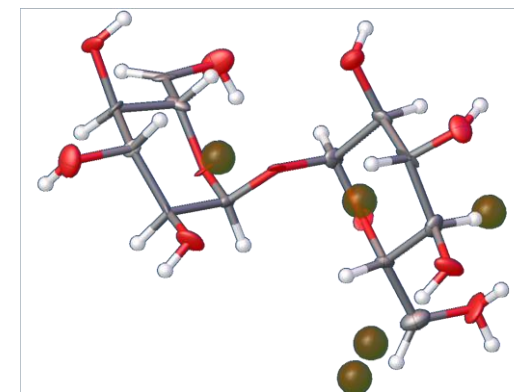
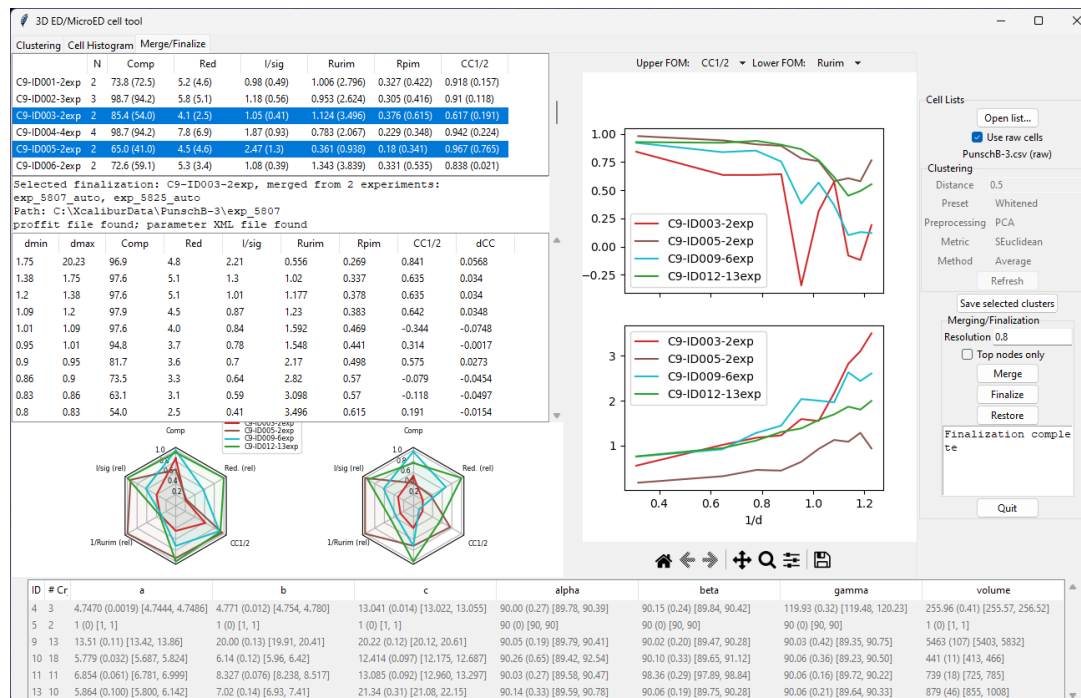
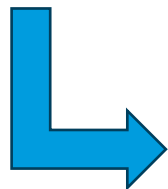
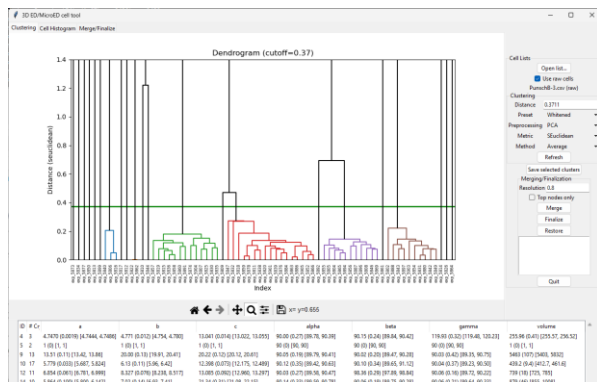
Autochem

No information available

Options
☐ Transparent ☐ Always on top

Add experiments Close

Structures from Merging

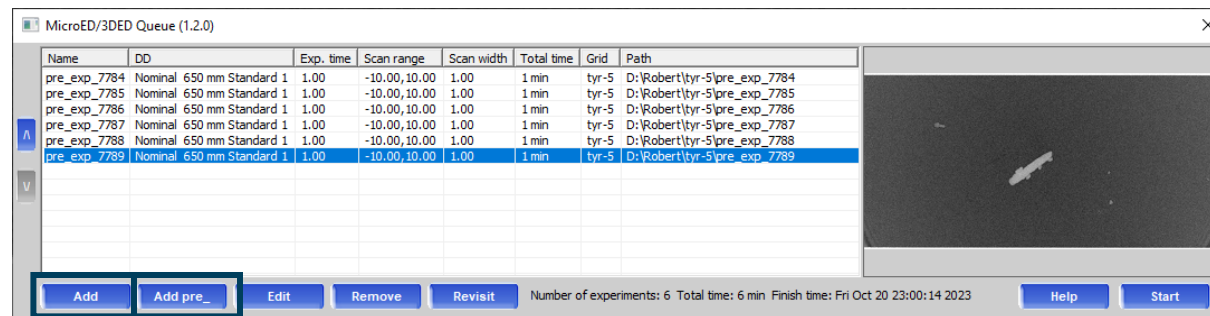
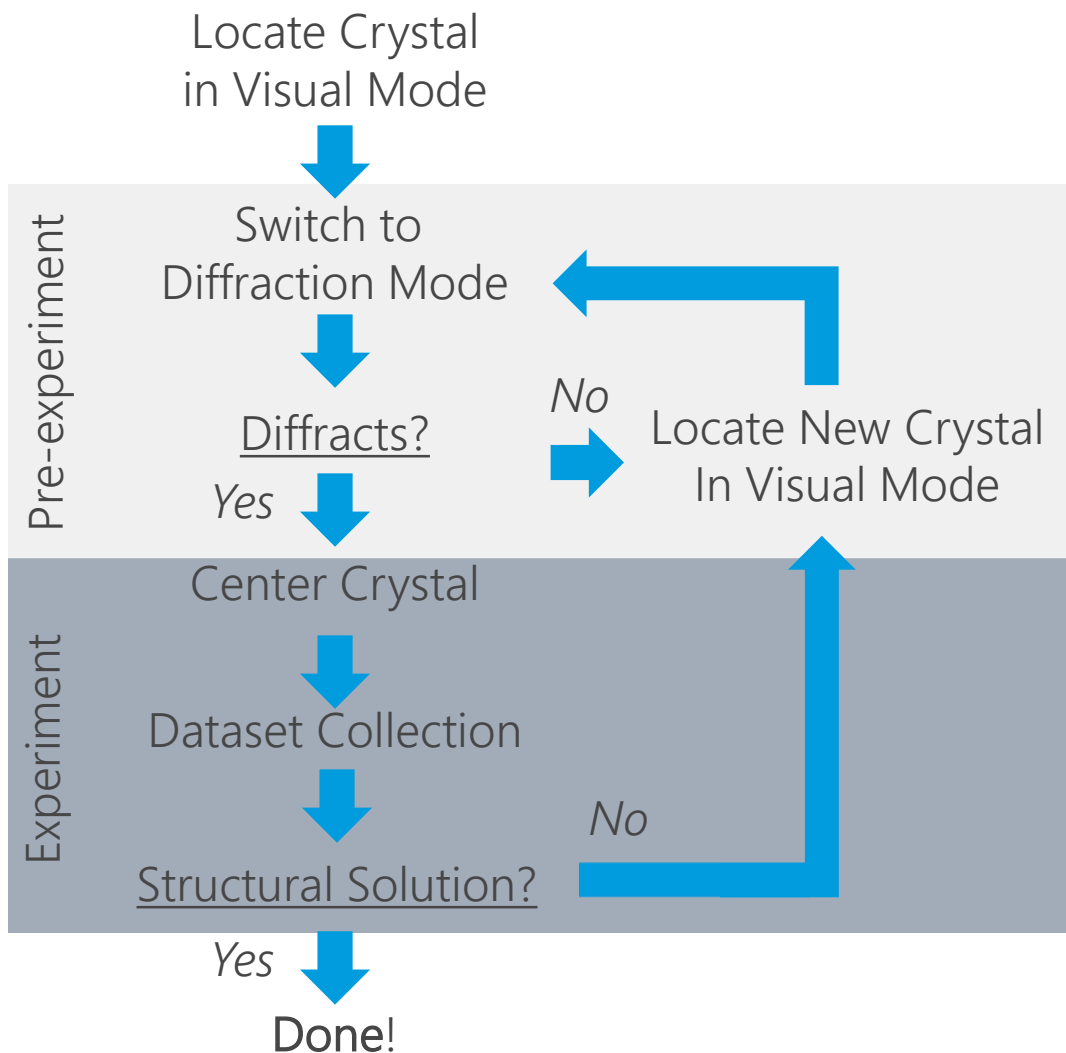


Cluster #11
11 grains

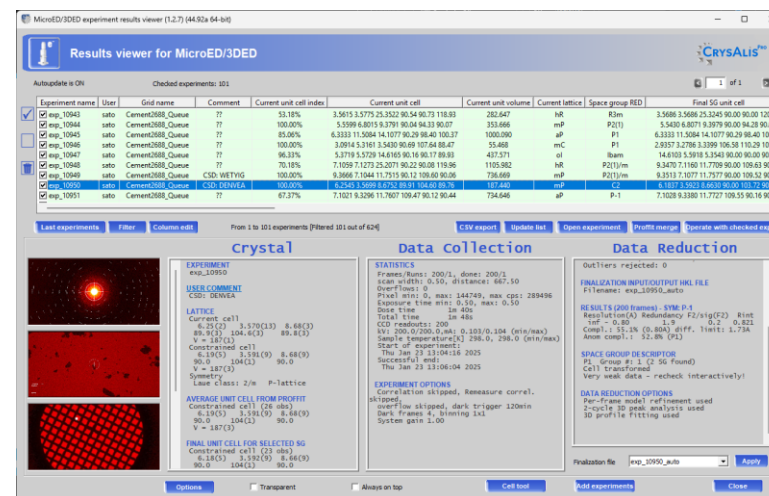
Automatic merging and scaling for each clustering tree node

Automatic structure solution with AutoChem from optimal merged solution

Workflow with Automation

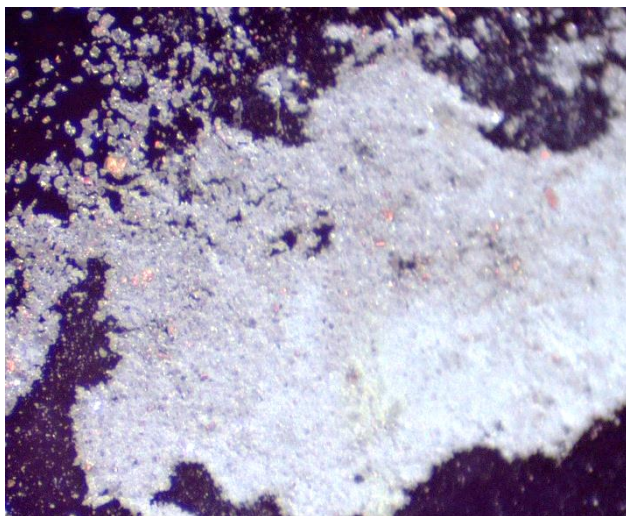


Queuing allows for unattended data collection

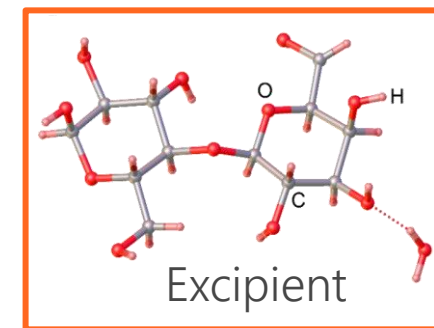
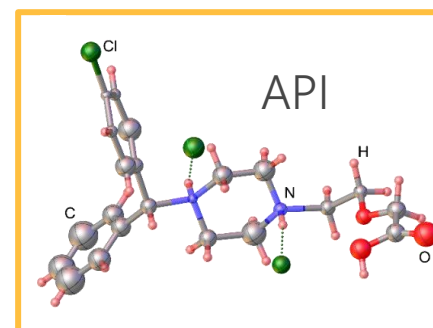
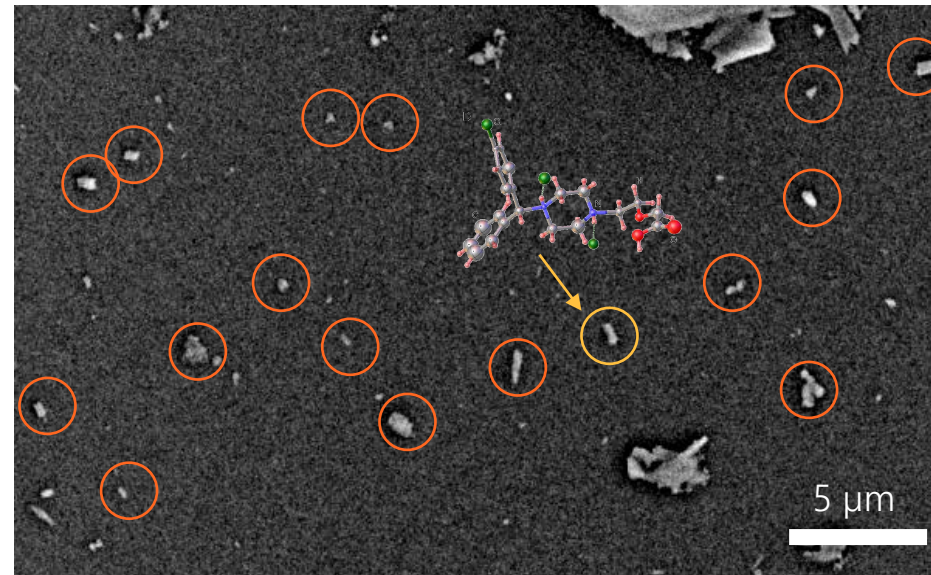
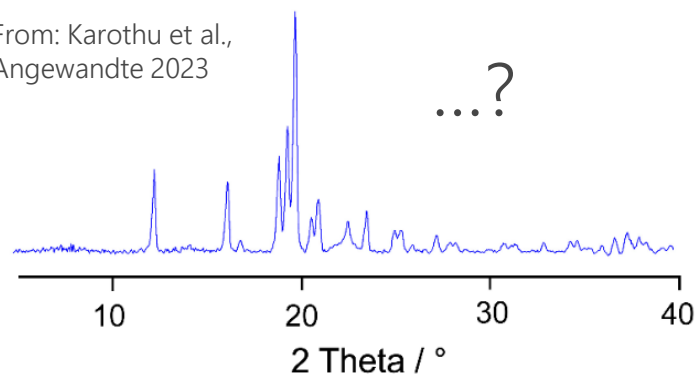


Results viewer streamlines decisions about data quality

Powders Grain-by-Grain



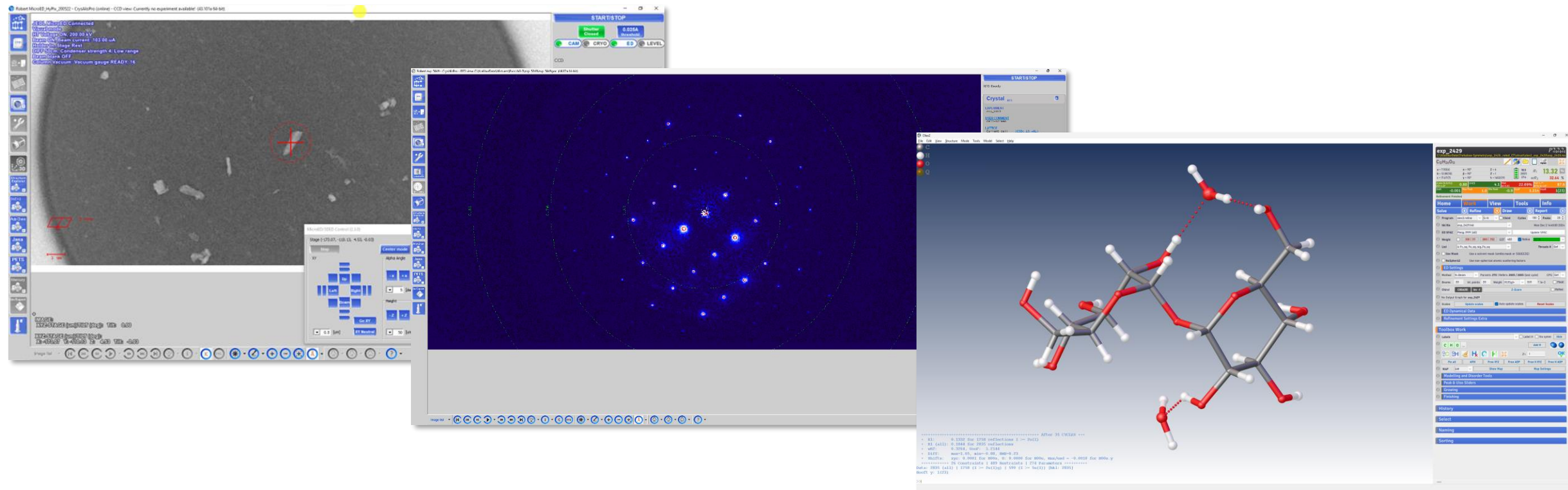
From: Karothu et al.,
Angewandte 2023



5. Summary

XtaLAB Synergy-ED: An Integrated Experience

Screen, collect, automate, process, solve, manage,... in *one* seamless interface



Screen for crystals

Collect diffraction data

Solve and refine structure

XtaLAB Synergy-ED

A dedicated electron diffractometer,
made for non-specialists

Highly optimized and integrated hardware and
software design for microED / 3D ED

Seamless workflows, from instrument control to
structure solution



Rigaku



Questions?





We'll follow up with
your questions.



Recording will be
available tomorrow.



Register for
webinar.



Webinar Series

Small Crystals, Big Insights: How Electron Diffraction is Transforming Materials, Life Science, and Chemistry Research

Episode 4 – Solving Pharma's Toughest Solid Form
Challenges with Electron Diffraction

Presenter: Simon Bates, PhD

Wednesday, June 4, 9 am CST

Don't forget to register for the next episode!

