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Deploying experimental strategies and workflows

A progress report

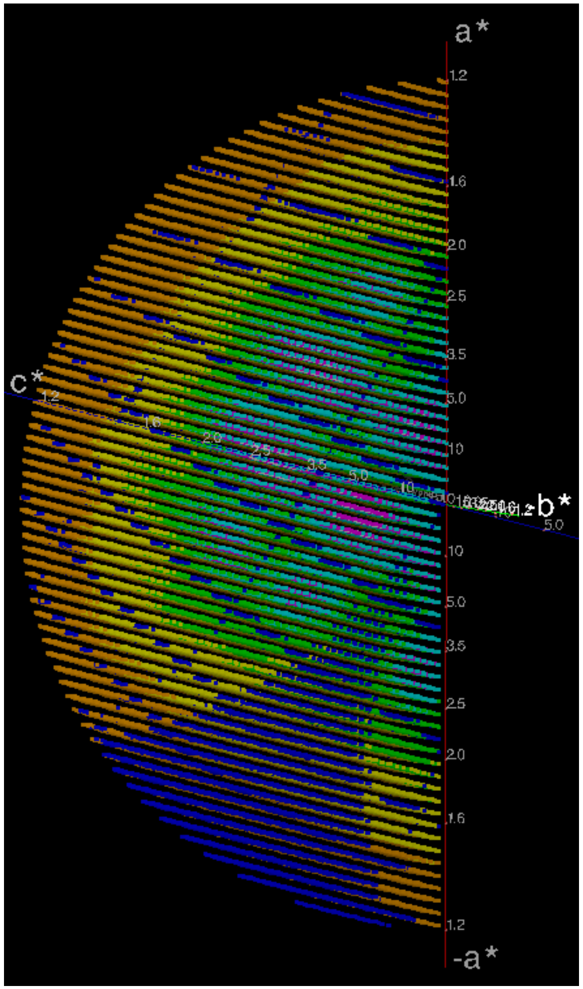
Rasmus Fogh
Global Phasing Ltd, Cambridge, UK

MXCuBE meeting, virtual ESRF
May 2022

- **Introduction – the GΦL Workflow**

- Development and deployment
  - EMBL-HH P14 collaboration
  - ESRF MASSIF1 collaboration
  - Centring issues

The problem - example



6R6F

P21

93% complete, redundancy 6

(highest shell: 90%, 6.1)

1.20Å resolution

Missing observations (blue) from module gaps and cusp

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On-line strategy calculation and execution

- Calculation of optimised multi-orientation acquisition strategies in real-time, from characterisation results
- Workflow* includes characterisation, indexing, pre-centring, parameter setting, and data acquisition
- Choice of strategies
  - Full, for high-quality data
  - OR quick, for fast-but-guaranteed-complete data
- Integrated into MXCuBE

** With acknowledgements to the EDNA precursors*

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Workflow capabilities

- Native, MAD or SAD collection strategies
  - Adapted to resolution, symmetry, crystal orientation, and accessible (re)orientations on the available goniostat
  - Use full range of κ values with minimal goniostat shadowing
  - Bespoke processing, allowing for residual shadowing
- Set default transmission*, depending on resolution
  - Uses pre-calculated radiation sensitivity values
  - Assumes B-factor increase of 1Å^2/MGy
  - ‘*Top hat*’: Based on mean flux density = flux / beam area

** With acknowledgements to the EDNA precursors*

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Strategy types

- Native, full
  - Optimised for most uniform distribution of redundancy
  - 360-960°, 2-4 orientations, depending on symmetry
- Native, quick
  - Optimised for completeness and speed (avoiding cusps)
  - 180-360°, 1-2 orientations, requires characterisation
- Phasing
  - Acquire Bijvoet pairs at similar dose and scaling, using alignment of even-order symmetry axes or inverse-beam
  - SAD, 360-540°, up to 4 orientations
  - MAD, 180-360°, up to 3 orientations, wavelength interleaving

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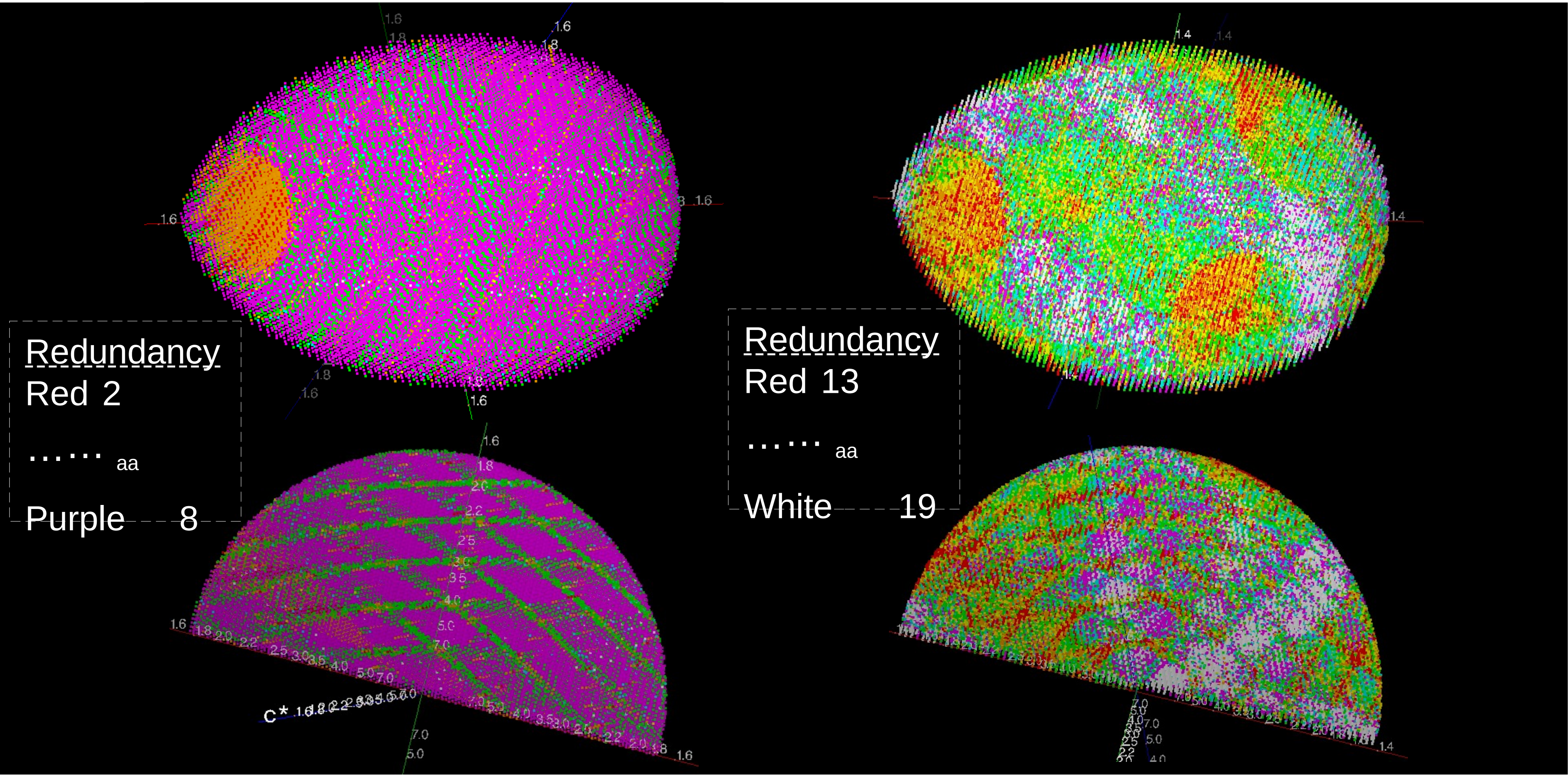
Monoclinic native - redundancy



Reflections for 1 * 360° strategy (left) *v.* 4 * 220° strategy (right)

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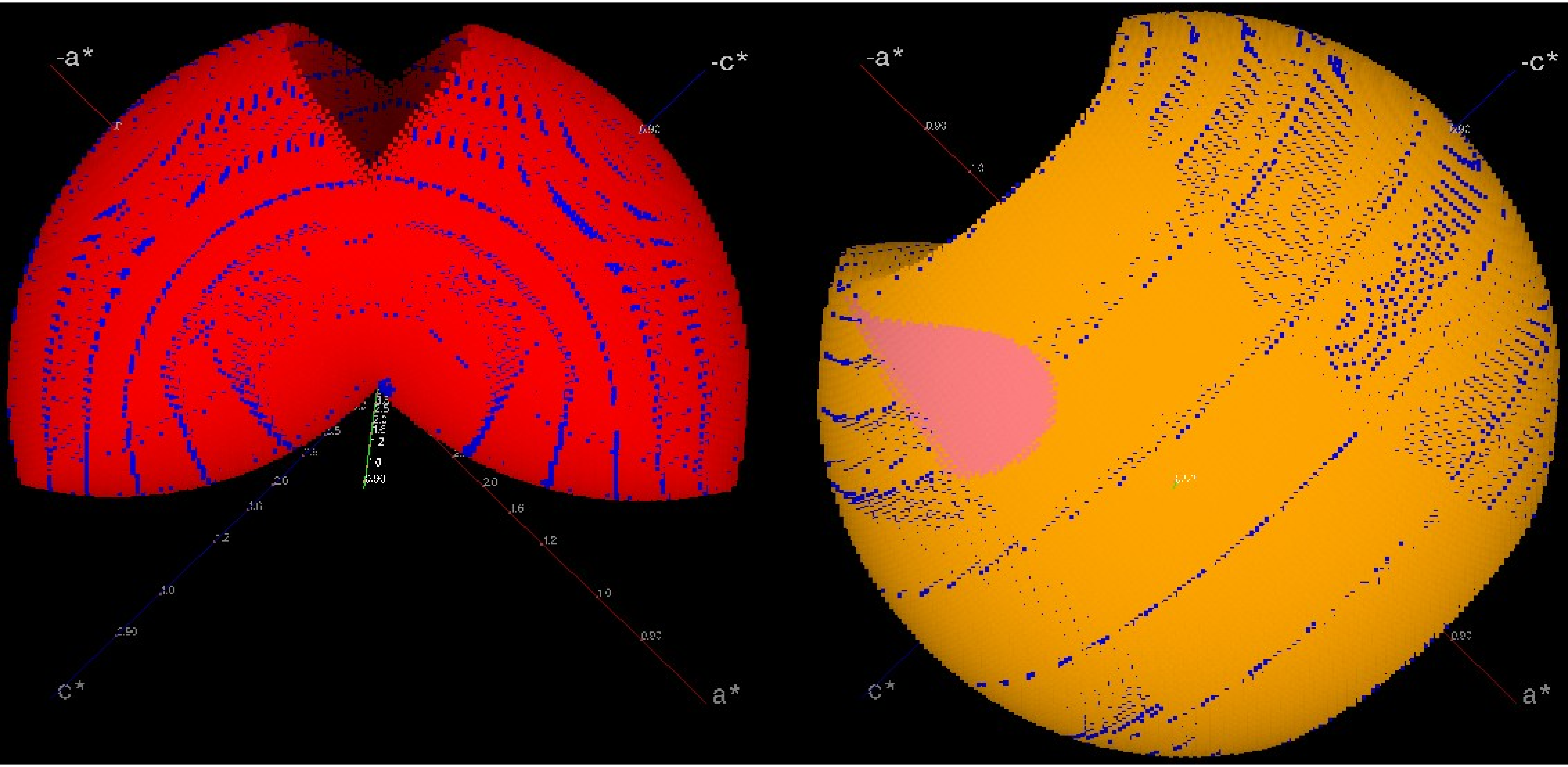
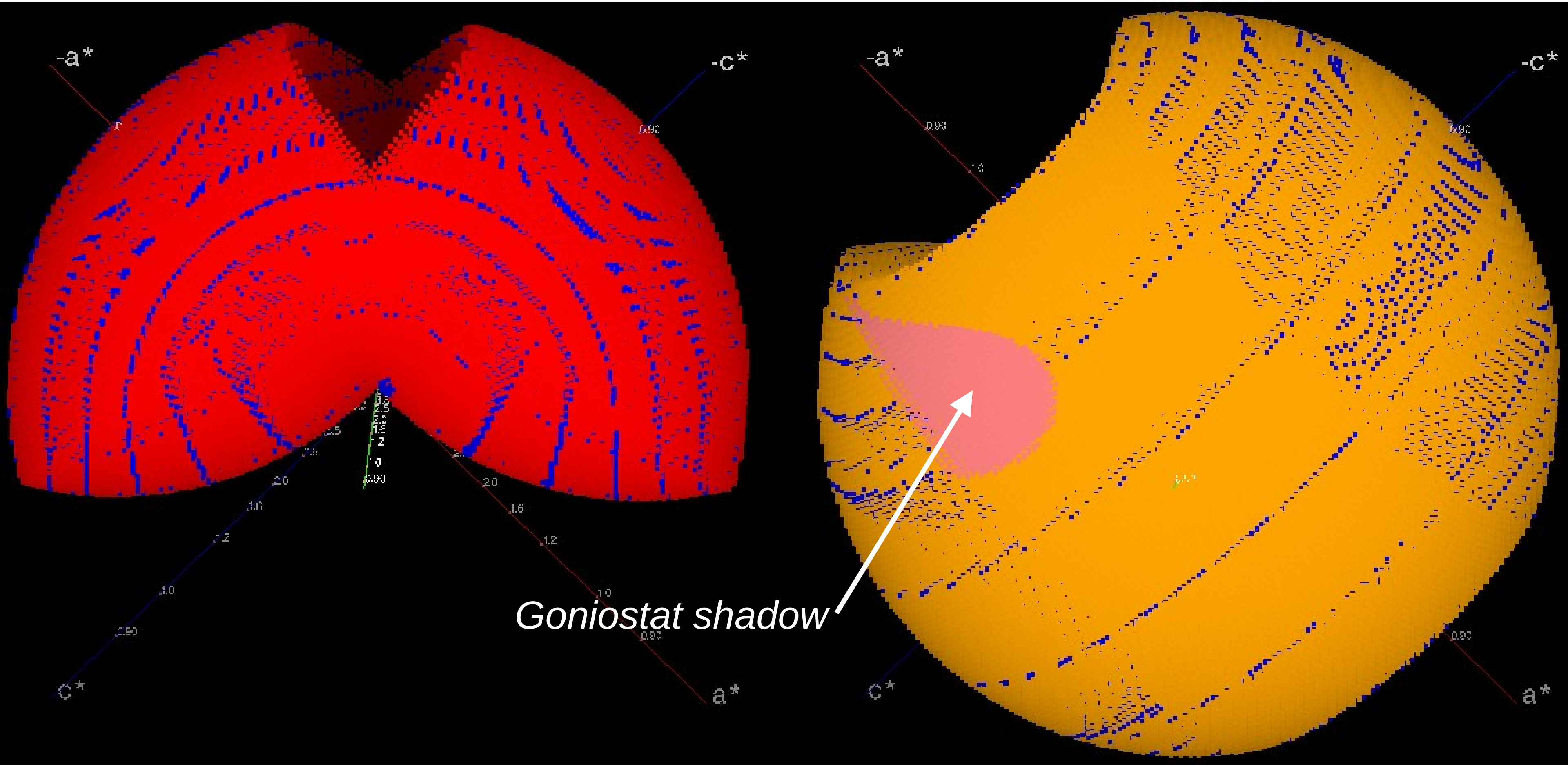
Orthorhombic phasing strategy



90° and 180 ° sweeps, aligned on different axes for mutual cusp-filling

Orthorhombic phasing strategy



90° and 180 ° sweeps, aligned on different axes for mutual cusp-filling

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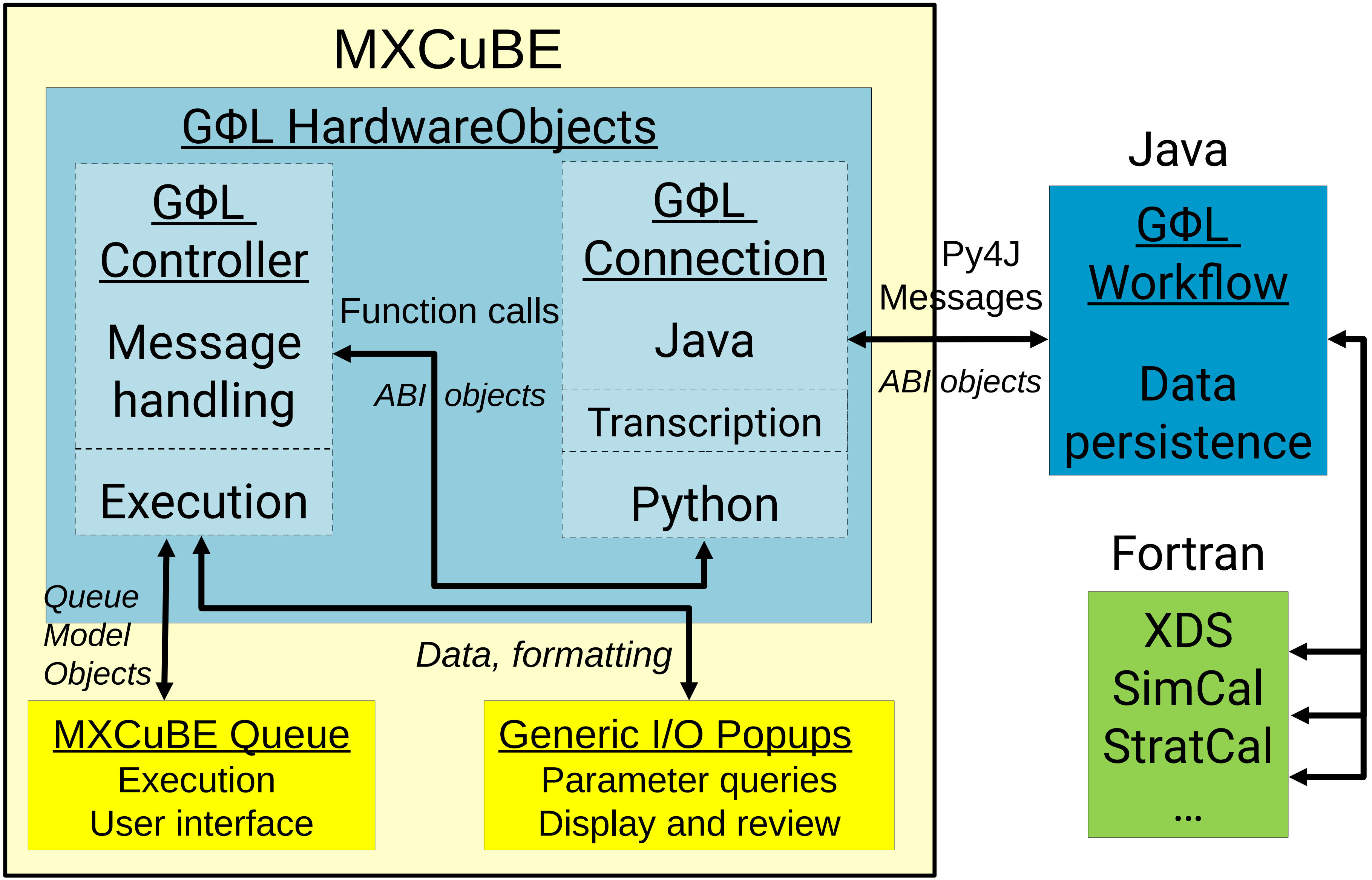
Workflow integration with MXCuBE



- Introduction – the GΦL Workflow

- **Development and deployment**
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Where are we in terms of deployment?

- Workflow installed on P14 at PETRA III
  Collaboration on improvements.
  In regular use by power users

- Porting workflow to MXCuBE3.
  Being installed at MASSIF-1, integrating with MASSIF-1 EDNA automation workflow

- Workflow installed and tested at SOLEIL, under further testing by beamline scientists

- Workflow installed on BL13 at ALBA
  under testing by beamline scientists

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Multi-beamline coding

- Each beamline needs working code for workflow integration
- Two different user interfaces, web and Qt – *well, OK*.
- Beamline production branches are **far apart**, requiring maintaining a permanent branch for each
  - Impossible to transfer or compare code using git repository operations
  - Improvements must be ported from version to version by copy-and-diff. Hard to keep consistency and avoid errors.
  - Be *nice* if we could work off a repository branch.

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Collaboration with P14

- Workflow installed and under full local control, in production use by external power users
- Improving user interface from feed-back.
- Contributing to collaboration with Gleb Bourenkov and Ashwin Chari on acquiring highest-quality data. Collaboration also includes:
    - Acquisition at 26.7 keV
    - Eiger2 16M CdTe detector
    - Width-adjustable top-hat beam
    - Ultra-high-quality crystals

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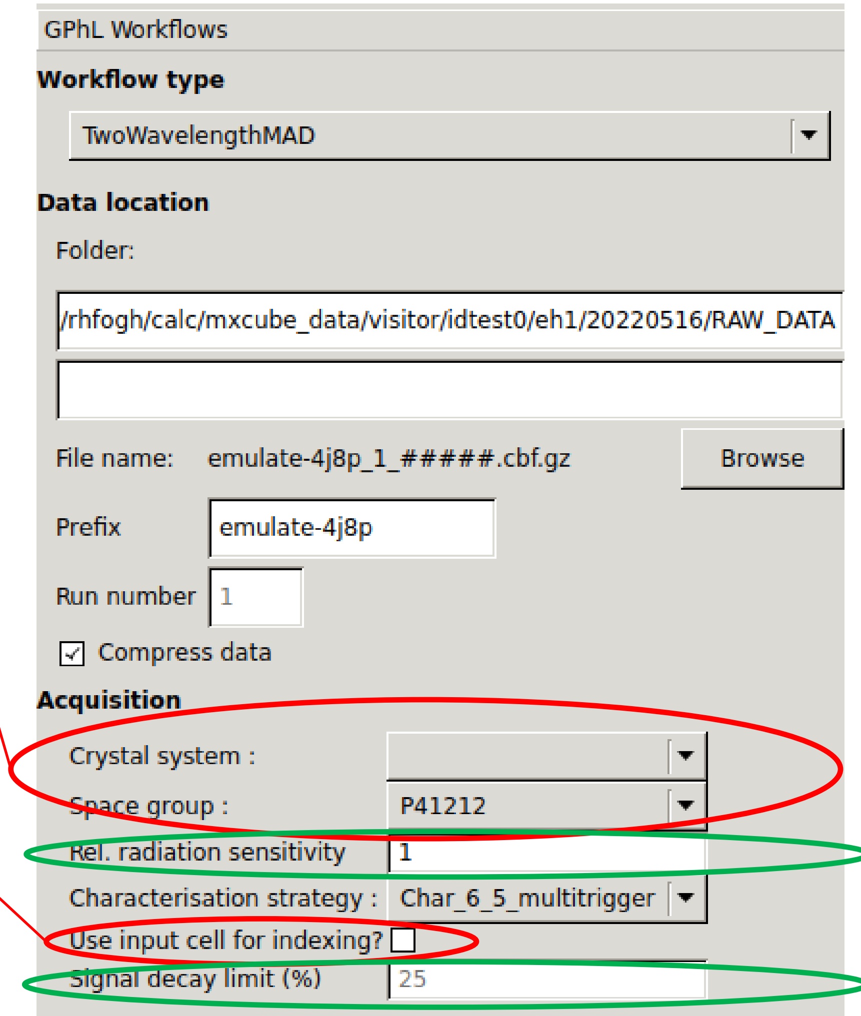
UI – starting panel



Select indexing solution and strategy symmetry

Enforce input symmetry in indexing calculation

Determine dose budget and transmission

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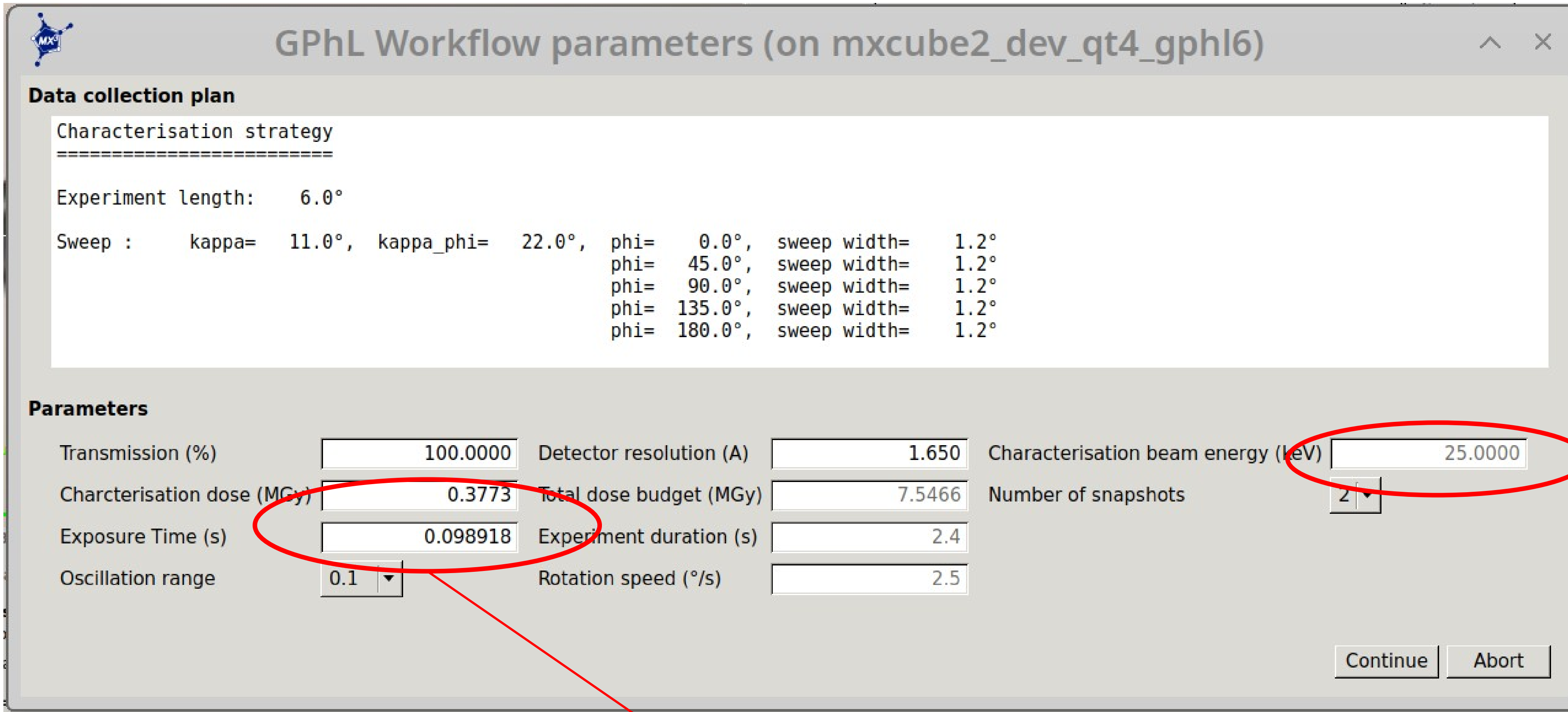
UI - Characterisation



Characterisation dose defaults to 5% of budget (configurable)
You would probably want to reset this value here.

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UI – Indexing solution

Select indexing solution:

	LATTICE-CHARACTER		BRAVAIS-LATTICE	QUALITY OF FIT	a	b	c	alpha	beta	gamma
1	*	44	aP	0.0	56.3	56.3	102.4	90.0	90.0	90.0
2	*	31	aP	0.0	56.3	56.3	102.4	90.0	90.0	90.0
3	*	14	mC	0.1	79.6	79.6	102.4	90.0	90.0	90.0
4	*	33	mP	0.1	56.3	56.3	102.4	90.0	90.0	90.0
5	*	35	mP	0.1	56.3	56.3	102.4	90.0	90.0	90.0
6	*	10	mC	0.1	79.6	79.6	102.4	90.0	90.0	90.0
7	*	34	mP	0.1	56.3	102.4	56.3	90.0	90.0	90.0
8	*	32	oP	0.1	56.3	56.3	102.4	90.0	90.0	90.0
9	*	13	oC	0.2	79.6	79.6	102.4	90.0	90.0	90.0
10	*	11	tP	0.2	56.3	56.3	102.4	90.0	90.0	90.0
11		37	mC	250.0	212.4	56.3	56.3	90.0	90.0	74.6

(UNIT CELL CONSTANTS (ANGSTROEM & DEGREES): a, b, c, alpha, beta, gamma)

Parameters

Detector resolution (A): 1.9
Main aquisition energy (keV): 20.0000
Strategy variant: full
Prior space group: P41212
Prior point group:
Prior crystal system:

☐ Use cell and symmetry for processing?

Continue | Abort

Adjust resolution and energy before strategy calculation

Space group from indexing solution and prior information

UI - Acquisition

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Data collection plan

```
TwoWavelengthMAD strategy, variant 'full'
-----------------------------------------

Experiment length: 2 *  540.0°

Sweep :     kappa=    0.0°,  kappa_phi=    0.0°,  phi= -130.0°,  sweep width=  180.0°

Sweep :     kappa=  101.1°,  kappa_phi=  149.7°,  phi=  -30.7°,  sweep width=  360.0°
```

Parameters

Transmission (%)	50	Detector resolution (A)	1.900	Peak beam energy (keV)	20.0000
Total dose (MGy)	46.5062	Total dose budget (MGy)	10.0090	Remote beam energy (keV)	20.0100
Exposure Time (s)	0.0810000	Experiment duration (s)	874.8	Number of snapshots	2
Oscillation range	0.1	Rotation speed (°/s)	1.2	Re-centre when orientation changes	
		Wedge width (deg)	15		

Continue Abort

Dose adjusts automatically when transmission, exposure time, or oscillation range change.
Dose-over-budget highlighted.

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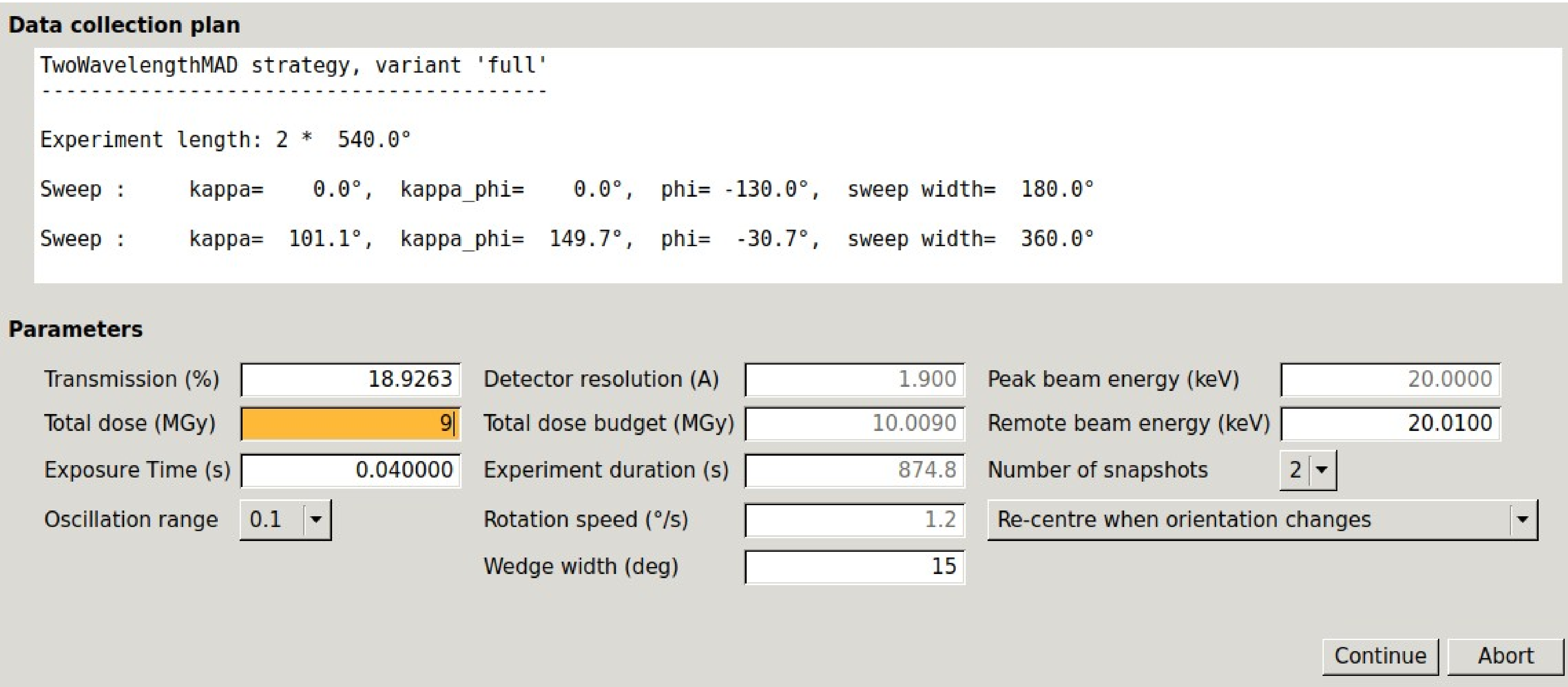
UI – Acquisition 2



Reset to max transmission and default exposure time when dose is changed.

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  - **ESRF MASSIF1 collaboration**
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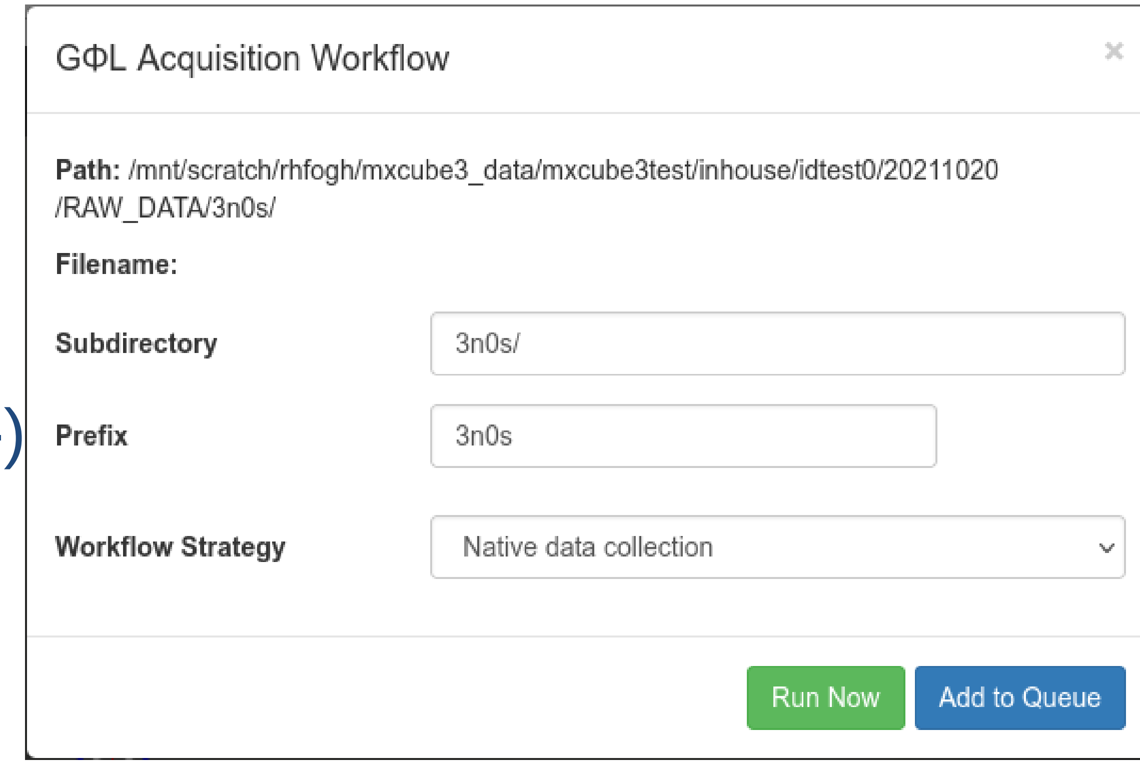
Integrating workflow in MXCuBE3

- Refactoring workflow for MXCuBE3 / MASSIF-1 and improved interface
- Acquisition tested in full-auto mock mode; beginning to test on MASSIF1
- Learning JavaScript ;-)



Special thanks to Marcus Oscarsson

MXCuBE3 interface popup for workflow

Fully automatic execution on MASSIF-1

- **MASSIF-1 workflow drives GΦL workflow**
  - GΦL workflow enqueued on MXCuBE queue
  - No UI – all parameters must be passed at start
  - Automatic selection of indexing solution
  - Automatic transmission determination

- **Characterisation done at MASSIF-1 level**
  - Use multiple thin-wedge characterisation (4 x 1.0 ° as 0.1° images) for greater robustness, as in GΦL workflow
  - MASSIF-1 does characterisation and first XDS analysis
  - GΦL skips characterisation and analyses SPOT.XDS file

- **X-ray centring (precise, fully automatic) for each orientation**
  - Implemented through an EDNA workflow task
  - GΦL calls centring through new MXCuBE queue entry

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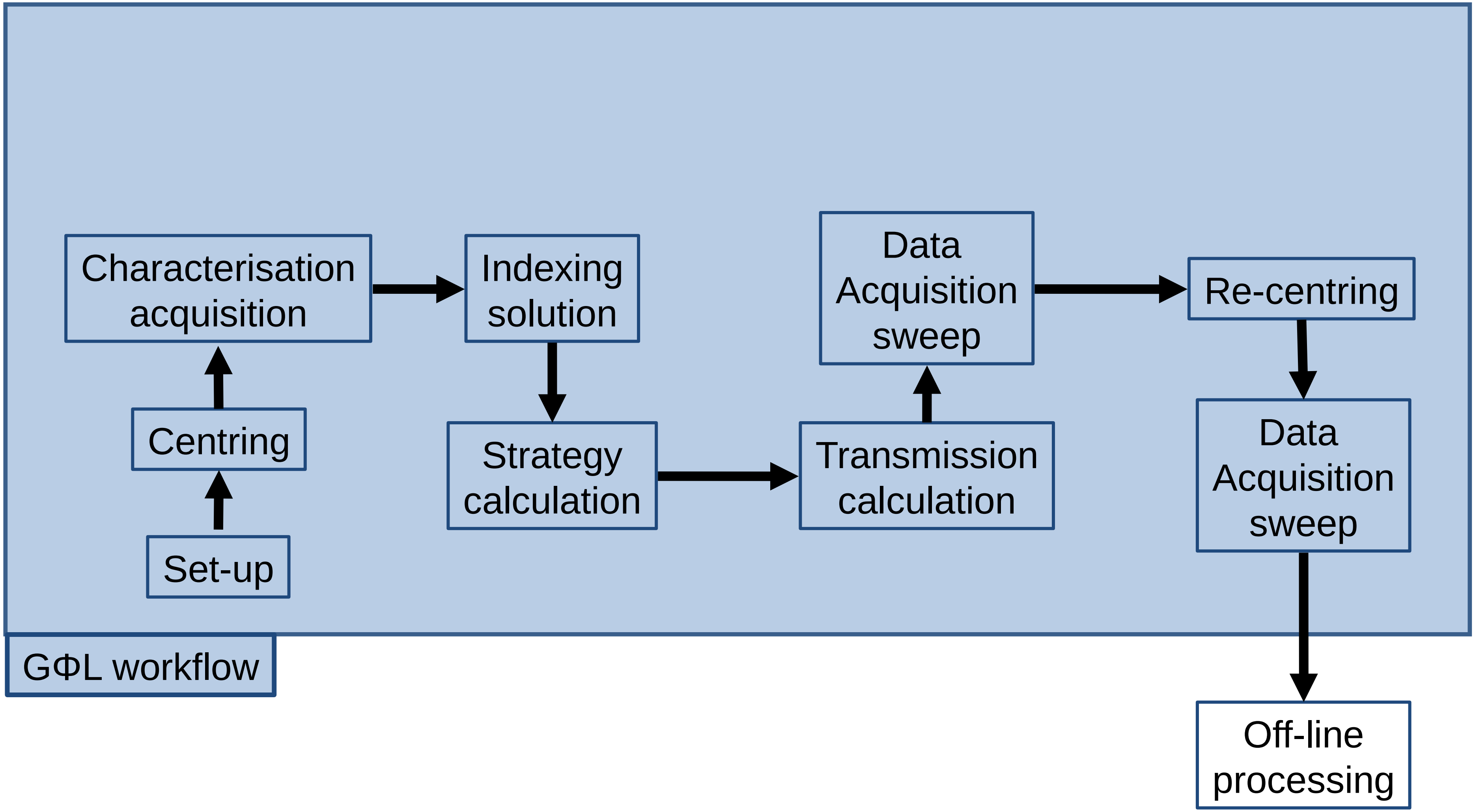
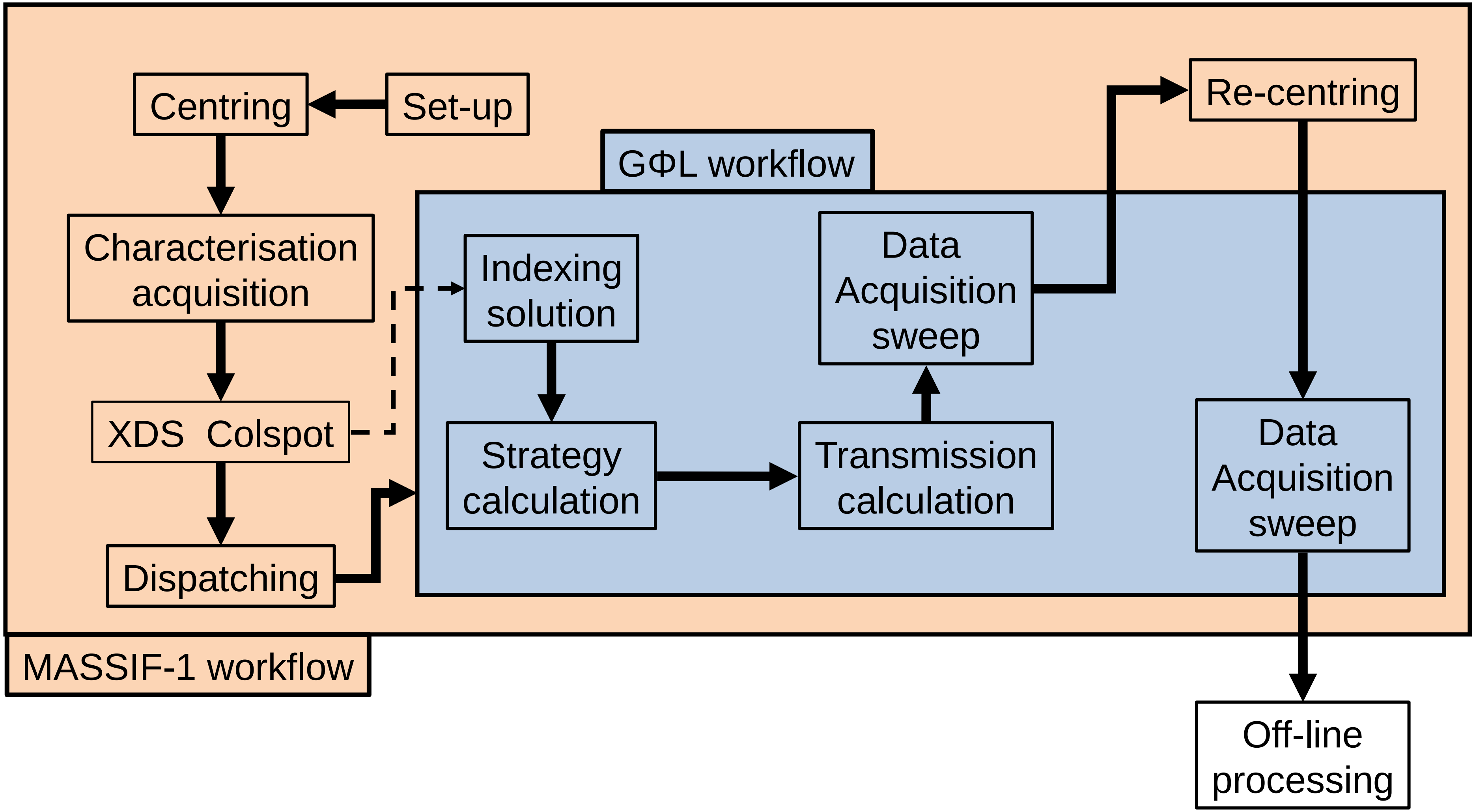
GΦL workflow actions



GΦL workflow on MASSIF-1



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Centring for multi-orientation experiments

- Each sweep uses different κ,φ settings of the goniostat
- Sample and instrumentation factors limit the recentring precision to no better than 10-20μm, even after careful goniostat calibration
- ‘Top-up’ recentring is therefore required for each orientation to ensure that the crystal remains in the beam
- Calculated pre-centring should allow any auto-centring step to be short, by requiring only a very small search grid

- Once centred at one orientation, it should be possible to calculate (approximate) centring at another orientation.

- GΦL release has routines for pre-calculating centring positions
    - Workflows for translational and diffractometer calibration
    - **STOP PRESS**: MASSIF1 has automated translational calibration using ball-tipped tungsten pin.
      Recentring shown to work well.

- EMBL P14 has its own recentring routines
    - "*Part of the MXCuBE code for ten years now*"

- WIP: MXCuBE-wide recentring code

X-ray centring

- New AbstractXrayCentring Hardware object, with MockXrayCentring, XrayCentringQueueEntry. etc.

- Implemented at MASSIF1 using calls to EDNA centring workflow
  - Initial tests done

- ‘Top-up’ centring for additional orientations (e.g. for GΦL workflow) should be a lot faster than the initial centring
  - Provided we can do reliable recentring calculations

- WIP: MXCuBE-wide automatic (X-ray) centring HardwareObject?

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Acknowledgements

- Global Phasing colleagues
  - **Peter Keller**
  - Rasmus Fogh
  - Wlodek Paciorek
  - **Claus Flensburg**
  - Clemens Vonrhein
  - Andrew Sharff
  - Ian Tickle
  - Gerard Bricogne
- Diamond Light Source
  - Funding under Collaboration Agreement COL0044 re. I23
  - Armin Wagner, Kamel El Omari
- EMBL-Grenoble
  - Josan Marquez, Irina Cornaciu
- **EMBL-Hamburg / PETRA III**
  - **Gleb Bourenkov**
- **Max Planck Institute, Göttingen**
  - **Ashwin Chari**
- **ESRF – MASSIF-1**
  - **Olof Svensson, Matthew Bowler, Marcus Oscarsson**
- ALBA synchrotron
  - Roeland Boer, Jordi Andreu
- SOLEIL
  - Martin Savko, Bill Shephard
- **The MXCuBE Collaboration**
  - **‘All for one and one for all’**
- The Global Phasing Consortium
  - Funding and much more