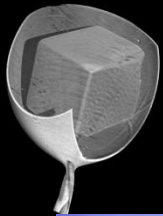


MINIKAPPA

strategies for data collection

Sandor Brockhauser

MX School, ESRF
Grenoble, France
Feb 8-11, 2010



EMBL-Grenoble & ESRF



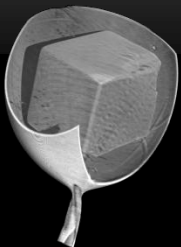


EMBL and ESRF

Beamlines

- CRG Beamlines: **BM14**, BM16, BM30
- ESRF SB Group Beamlines:
 - Fixed wavelength beamlines:
ID14-1, ID14-2, ID14-3
 - Tunable wavelength beamlines:
ID14-4, ID23-1, ID29
 - Microfocus beamline:
ID23-2

ID14-4
Developments
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smart strategies
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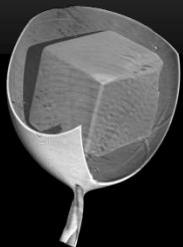
ID14-4: PDB millenary 2009

Beamlines

ID14-4

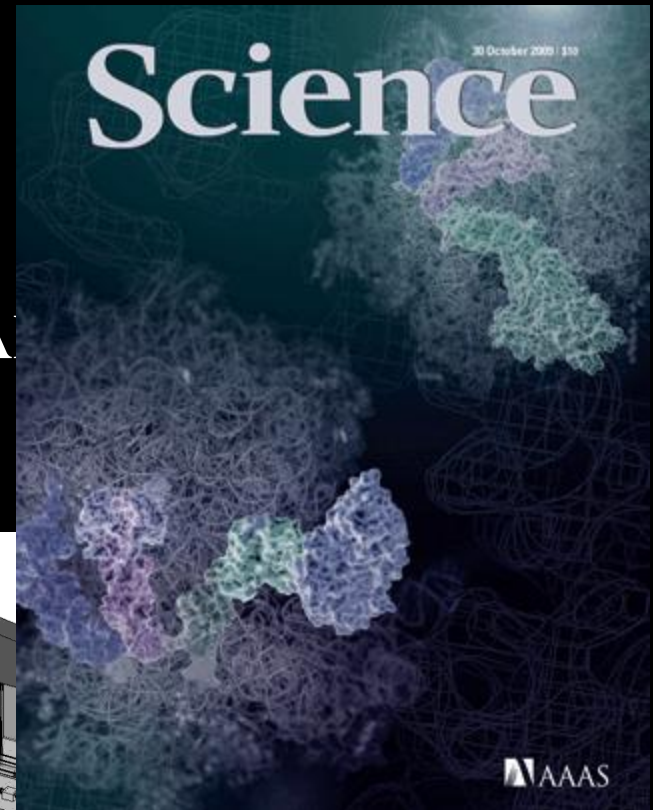
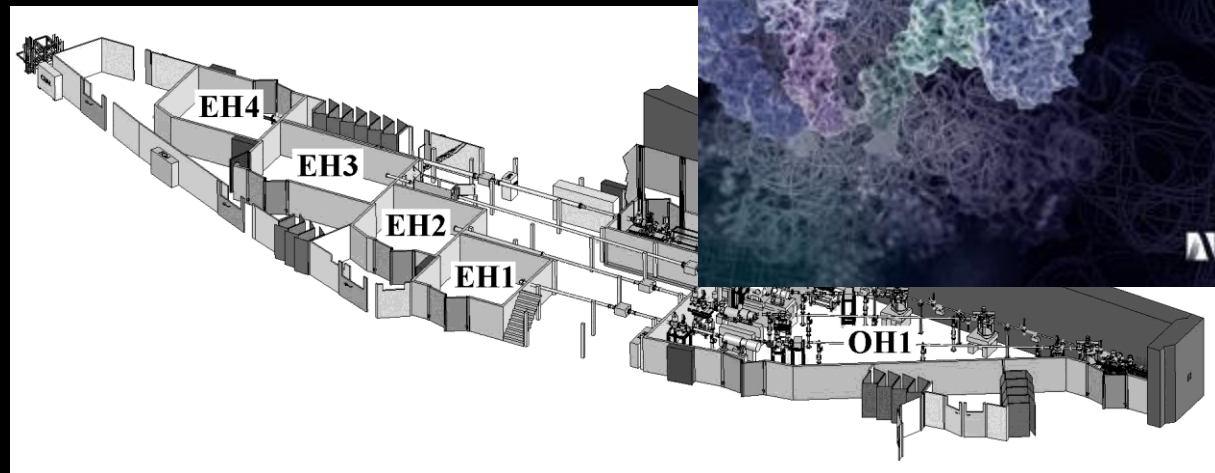
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After a decade in service

- First in Europe
- Second on the world (after A





Instrumentation Developments

Beamlines

ID14-4

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aims

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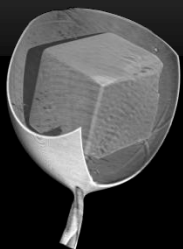
MX Tomography

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Summary

- Vibration-free Optics (monochromator, mirror)
- Beam Shaping (definition and cleaning apertures)
- Precise Goniometers (advanced centering: decreased SOC/on-axis camera/easy beam position monitor)
- Sample Changer
- New generation of Detectors
- On the fly diagnostics



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Data Collection/Processing

Beamlines

ID14-4

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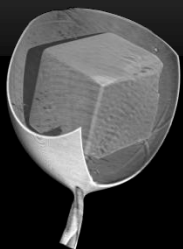
MX Tomography

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Summary

- Redundancy (empirical absorption correction/filtering out outliers)
- Screening for best crystal
- *Extended screening:*
(best place of the best crystal)
- Radiation Limitations
- *Radiation Induced Phasing*
- **Crystal Reorientation**
- *Dynamic Data Collection Strategies*





Kappa Workgroup

Beamlines

ID14-4

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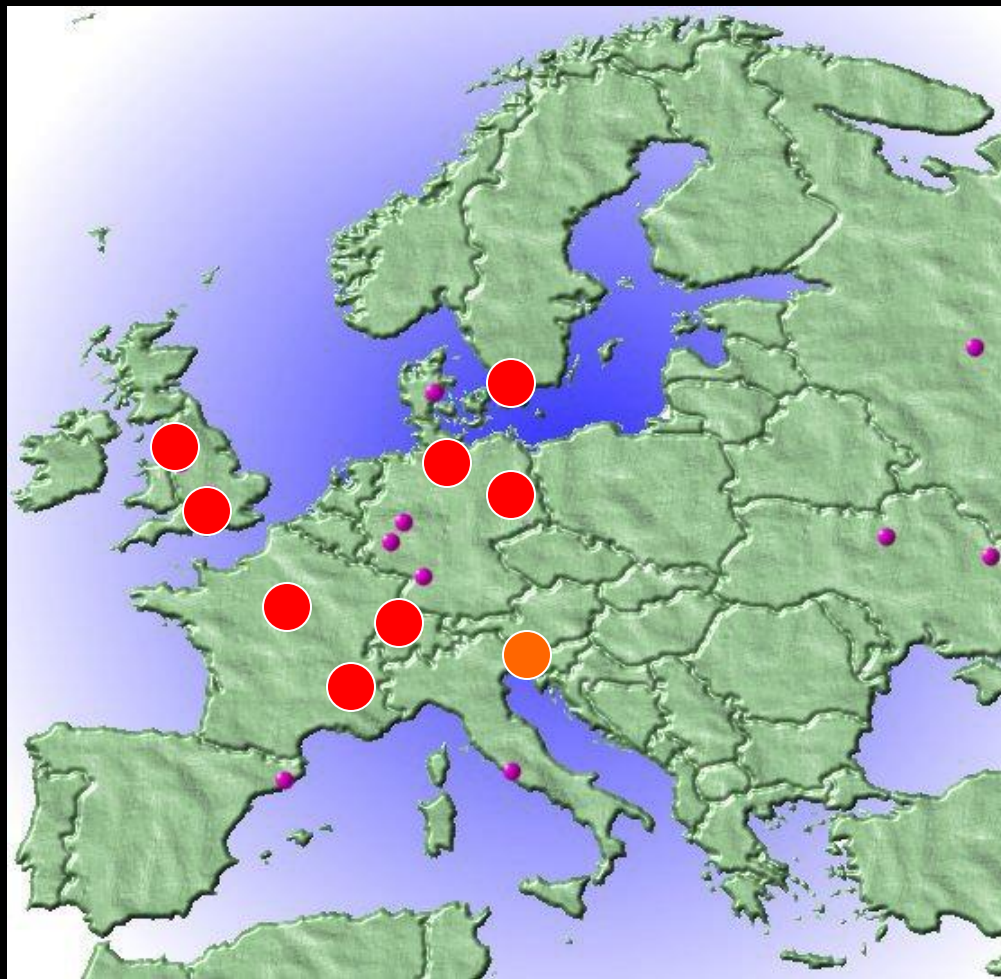
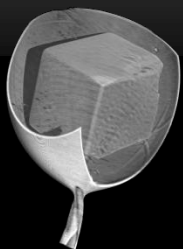
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Kappa Workgroup

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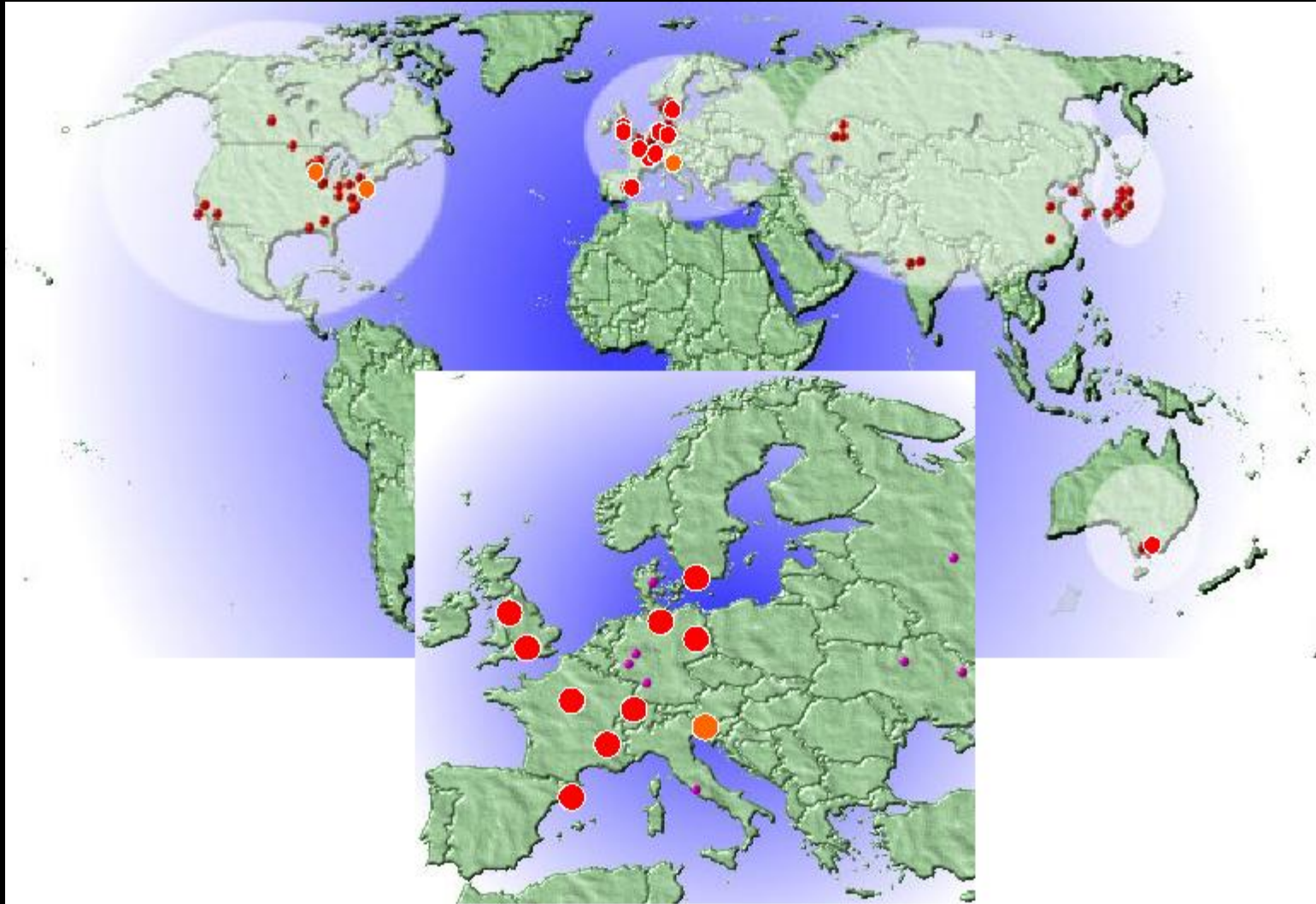
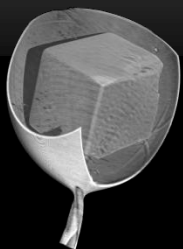
aims

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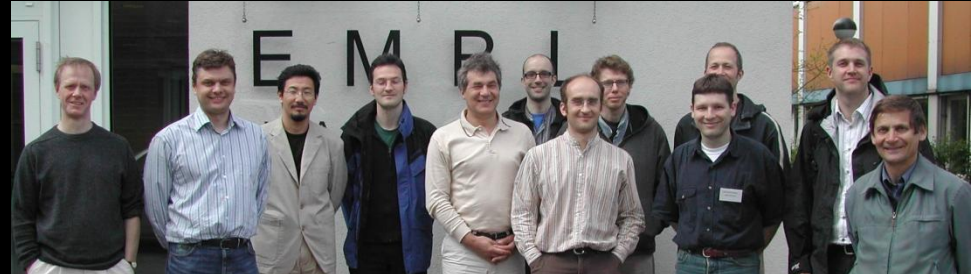


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Kappa Workgroup



Grenoble, 2004



Hamburg, 2005



Soleil, 2006



Diamond, 2007

Grenoble, 2007

Leiden, 2008

Maxlab, 2009

Berlin, 2010



Beamlines

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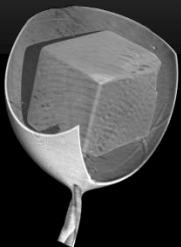
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Bijvoet Pairs on the same image

Wimberly et al. (2000). Structure of the 30S ribosomal subunit. *Nature* **407**, 327-339.

Beamlines

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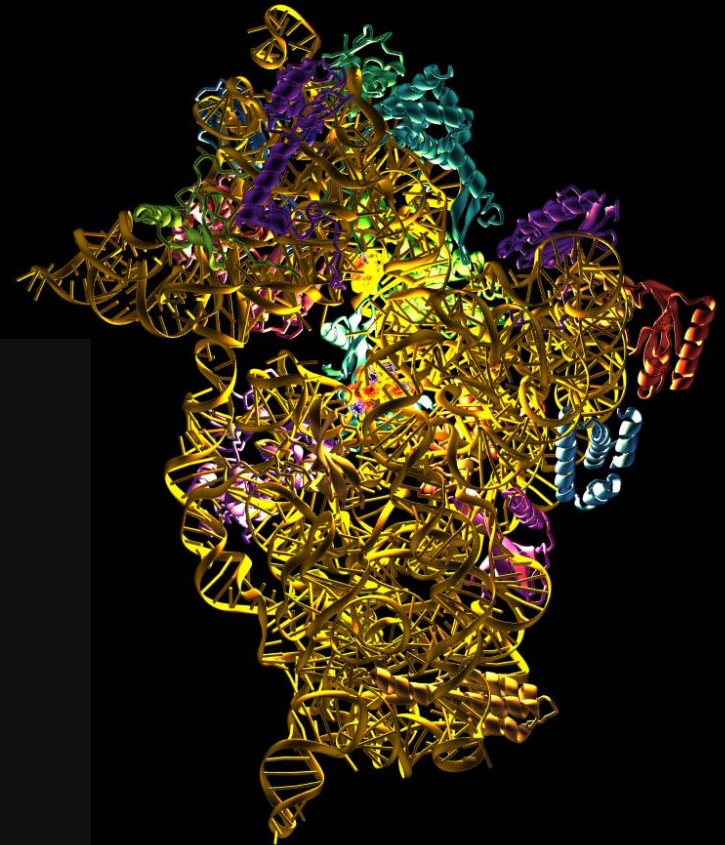
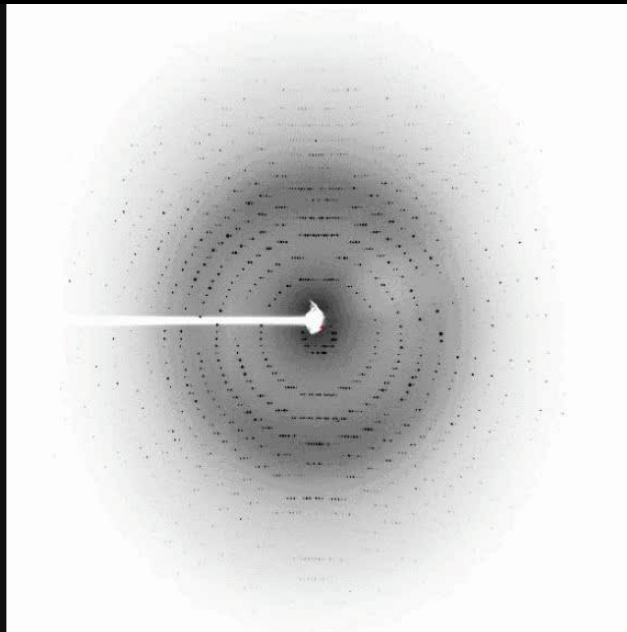
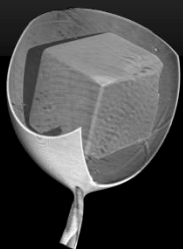
instruments

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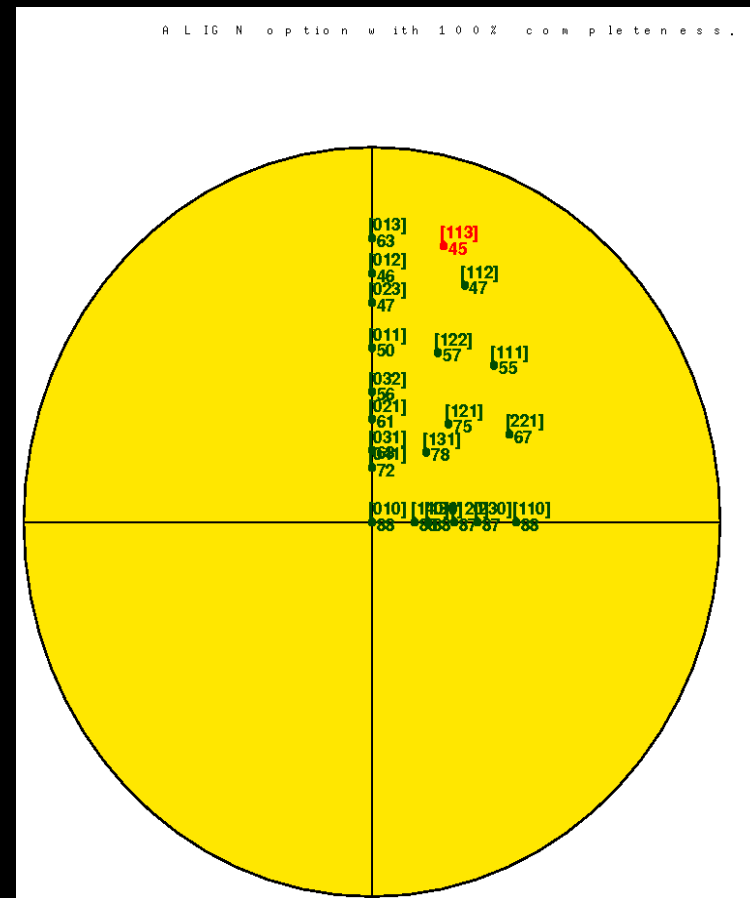
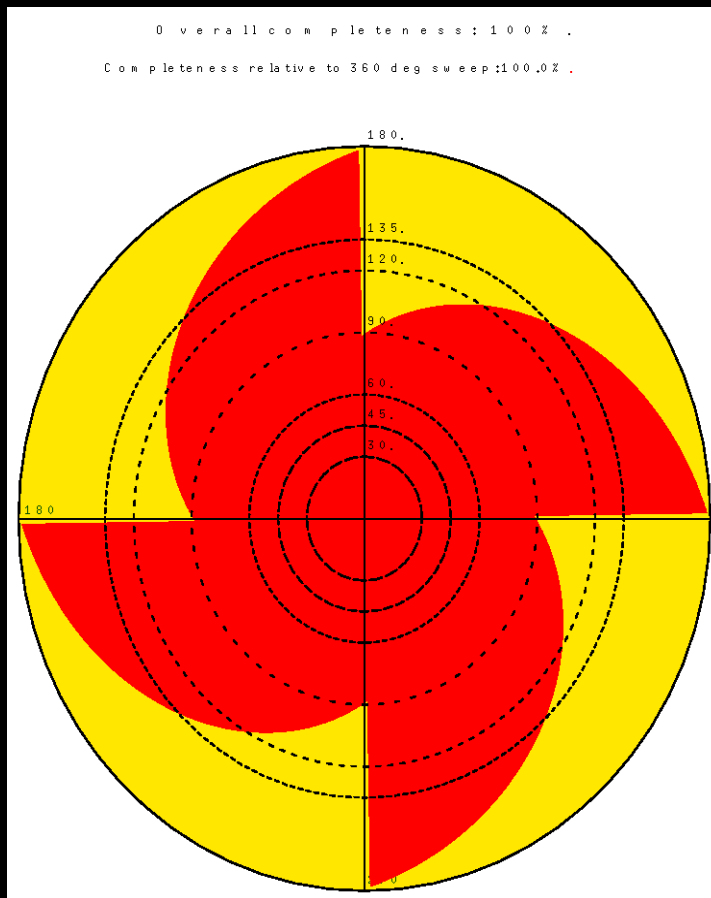
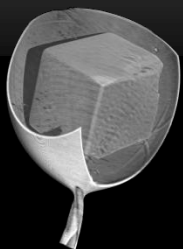
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Optimal Data Collection Oscillation Range

Beamlines

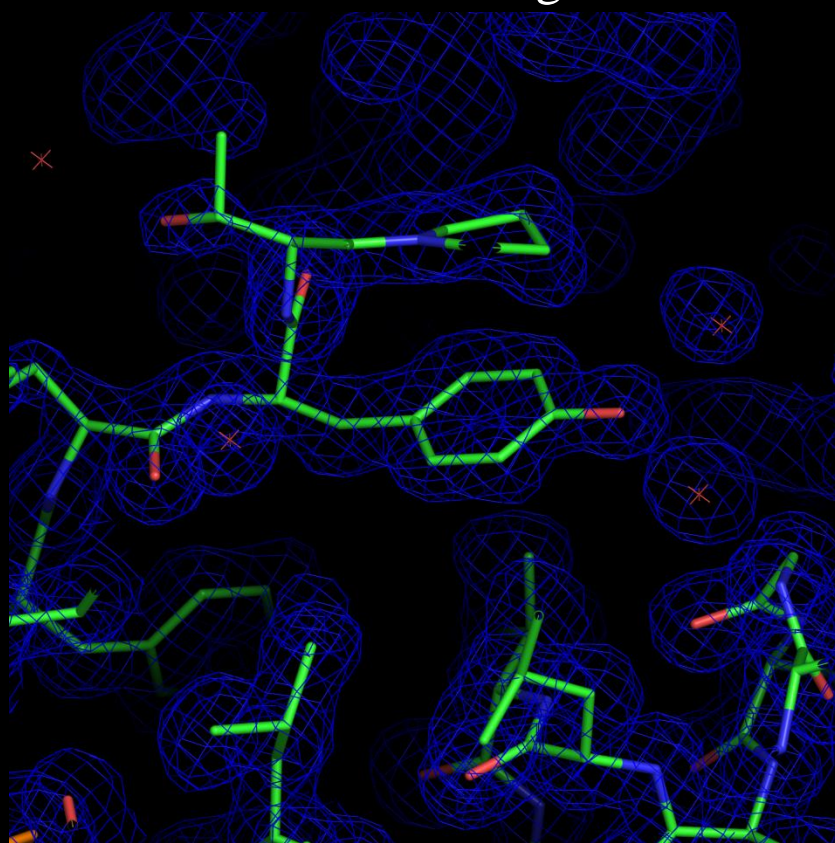
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High-energy S-SAD

4 runs, alignments: (0 0 1), (0 1 3), (1 2 8) and random
Sulphur SAD at 0.976 Å wavelength
Theoretical Anomalous Signal: <0.5%



- Sites found with SHELXD using resolution-ranges 40-3.8 .. 40-3.0

- CC(best) varies between 27% (3.0Å) and 40% (3.8Å)

- SHELXE iteration needed to resolve individual sulphur atoms

- Radiation-damage Induced Phasing is easier

Beamlines

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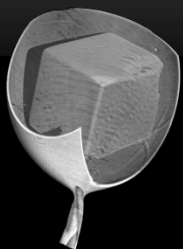
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Kappa Instruments

ESRF-MX/ BM14/ FIP/
Bessy/ Diamond/APS/
ALS/PETRAIII



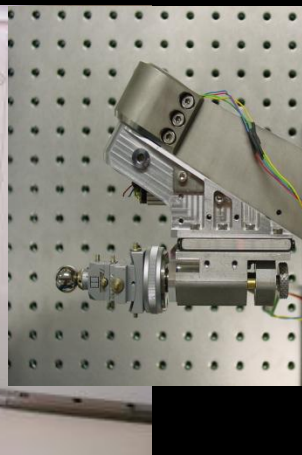
Uni Sydney



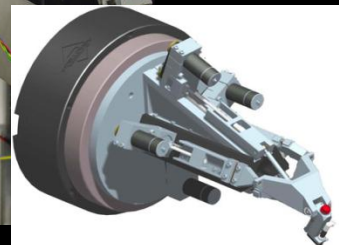
Soleil



MAX-lab



SLS



Beamlines

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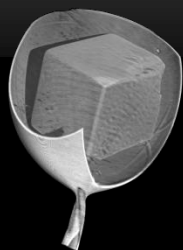
Kappa Goniometry

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Problems with Kappa Gonios

- Risk of Collision
 - » Limited use; only for resolved users
- Difficult Alignment
 - » Testing and setting up the instrument is a long procedure
- Low Overall Precision
 - » SOC ~ 20microns
- Problem of Leaving the Cryo
 - » Even determined users become upset by loosing their important samples

Beamlines

ID14-4

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Kappa Goniometry

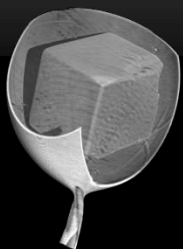
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ESRF/EMBL MiniKappa

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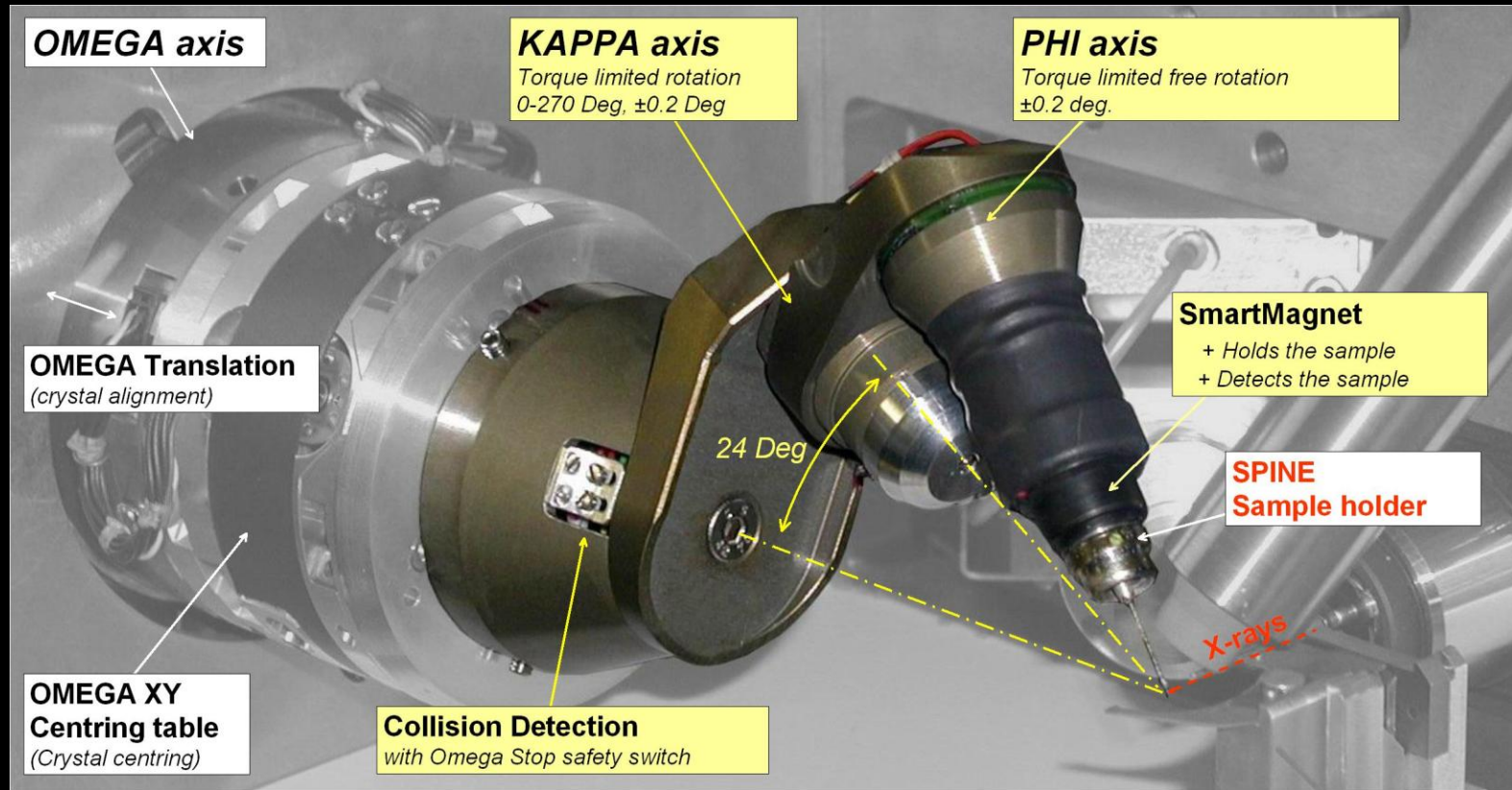
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ESRF/EMBL: 8 DIAMOND: 2 BESSY: 1

APS: 7 ALS: 1

MAXLAB/SOLEIL/ALBA/NSLS/SLS

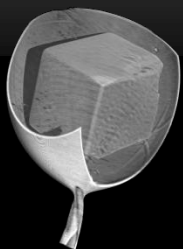
PETRAIII: 1

20 units

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STAC in DNA

DNA Expert System

File Screening

DNA - automated collection of data

Sample Screening Sample Ranking Collect Reference Images Auto Index Kappa Strategy Results

Desired orientation Datums Strategies

Multiple-Sweep Strategies: Clear Table

ID	OmegaStart	Incr	Time	Images	1st img	Resolution	Kappa	Phi	Completeness	Rank
1	11.0	1.0	34.558	97	1	2.4	71.244	130.432	99.984	3766.822 (...)
1	39.0	1.0	34.558	12 (3)	1	2.4	0.0	0.0	99.89	3766.822 (...)
2	16.0	1.0	34.558	98	1	2.4	164.093	313.687	99.988	3490.358
2	40.0	1.0	34.558	3	1	2.4	0.0	0.0	99.894	3490.358
3	64.0	1.0	34.558	90	1	2.4	127.281	213.687	99.988	3213.894
3	48.0	1.0	34.558	3	1	2.4	0.0	0.0	99.894	3213.894

Omega Kappa Phi X Y Z

Clear: Mv Sel. Mv Edit

Collect Data Collect & Integrate Data

Feedback for PROPOSAL : opd14

Collection status: Ready

Processing status: Ready

Control

Kappa Strategy ☒ On ☐ Off

Help Submit Feedback Abort

Executive Output MOSFLM Output

```

20090223 15:43:36 : Ranking resolution returned by strategy: 3.08
20090223 15:43:36 : 
20090223 15:43:36 : =====
20090223 15:43:36 : WARNING! WARNING! WARNING! WARNING! WARNING! WARNING! WARNING! WARNING!
20090223 15:43:36 : 
20090223 15:43:36 : In order to avoid radiation damage for this crystal BEST has decreased the resolution to 3.08 Å.
20090223 15:43:36 : This is the resolution that will be used if you choose to start a data collection.
20090223 15:43:36 : 
20090223 15:43:36 : Calculating the kappa strategies
20090223 15:43:36 : Starting kappa strategy calculation...
20090223 15:43:45 : Calculation performed by STAC (using strategy from Raimond Ravelli)
20090223 15:43:45 : Waiting for new command
  
```

Beamlines

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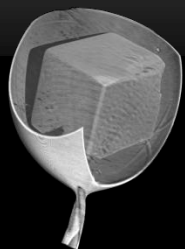
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Air Absorption

Beamlines

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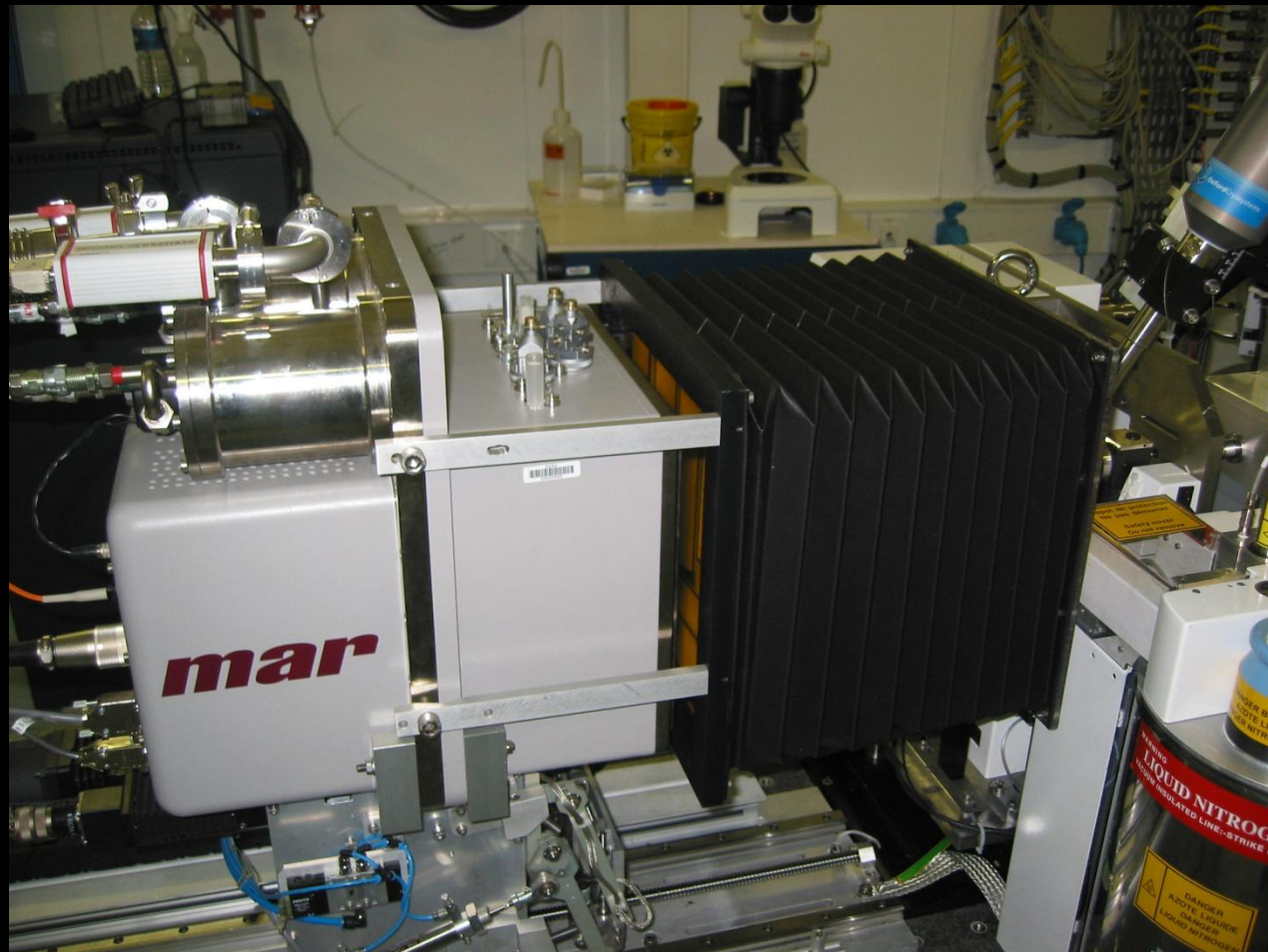
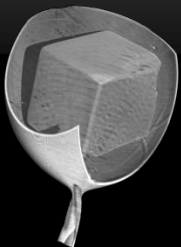
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Absorption Correction

Beamlines

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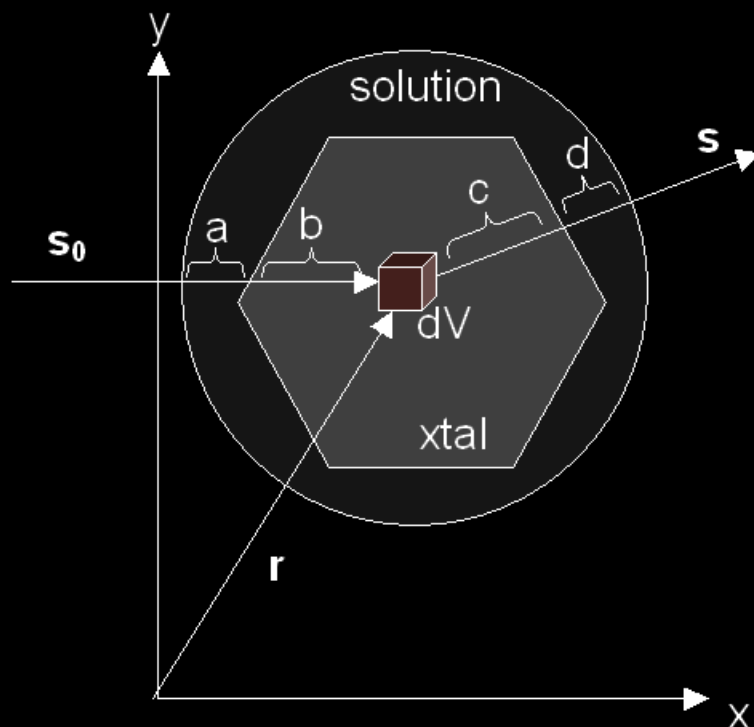
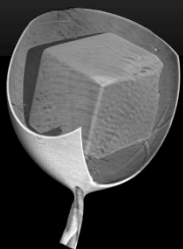
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$$A = \frac{1}{V_{xtal}} \int_{V_{xtal}} e^{-\int_{\xi \geq 0} [\mu(\vec{r} - \hat{s}_0 \xi) + \mu(\vec{r} + \hat{s} \xi)] d\xi} d^3 \vec{r}$$



Crystal Defects

Beamlines

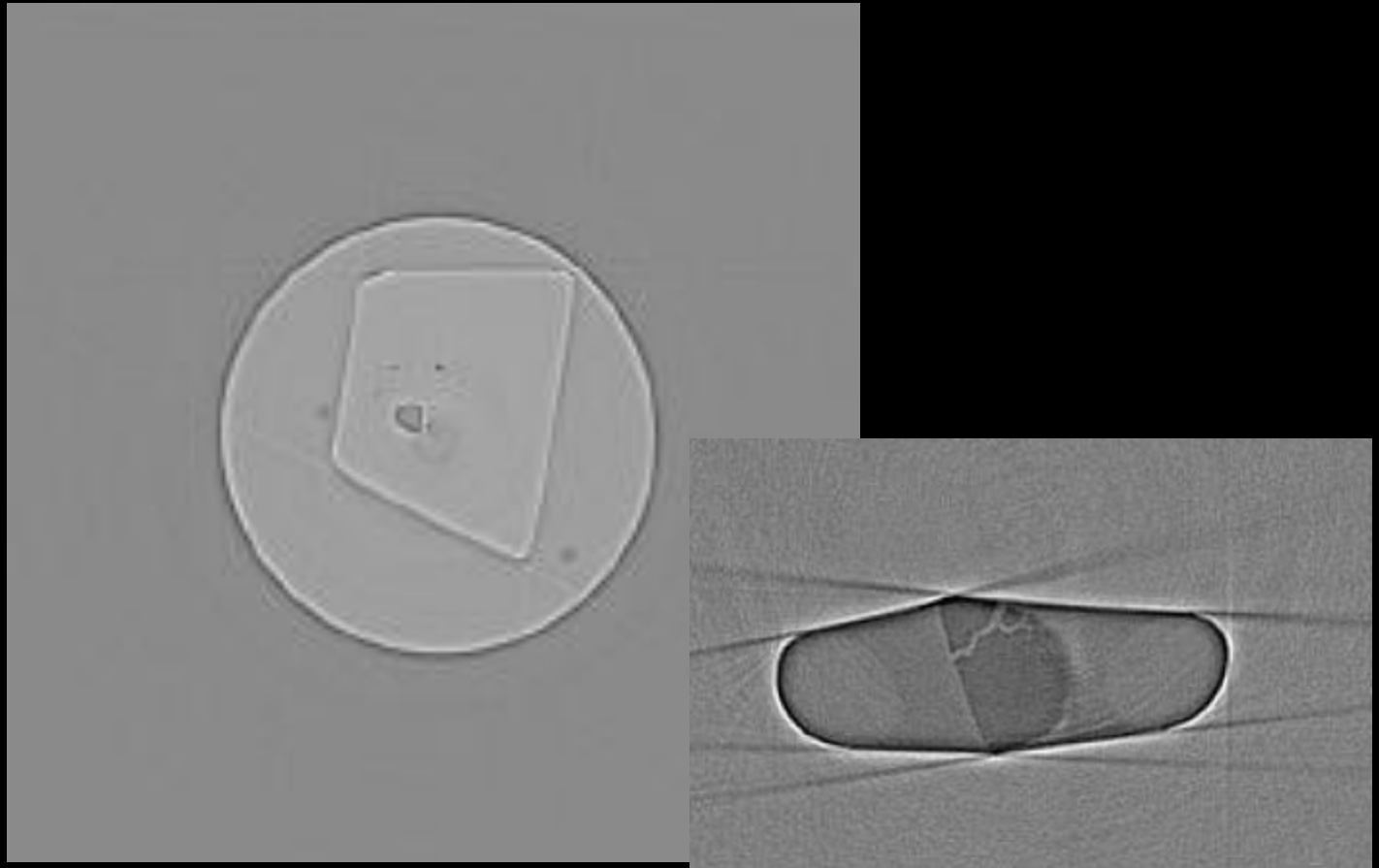
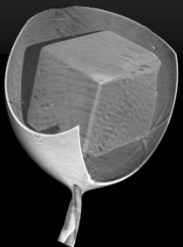
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Crystal Defects

Beamlines

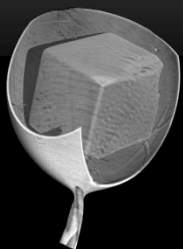
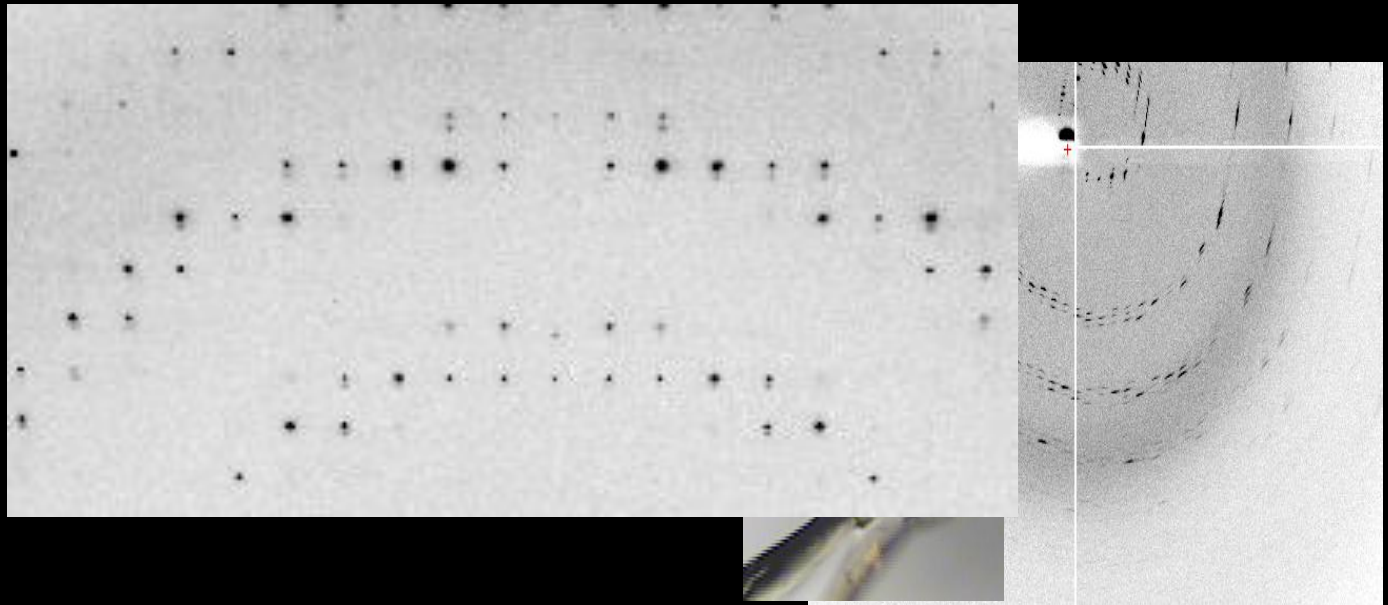
ID14-4

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Centering Microcrystals

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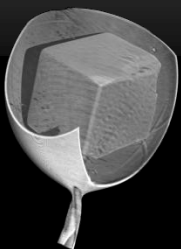
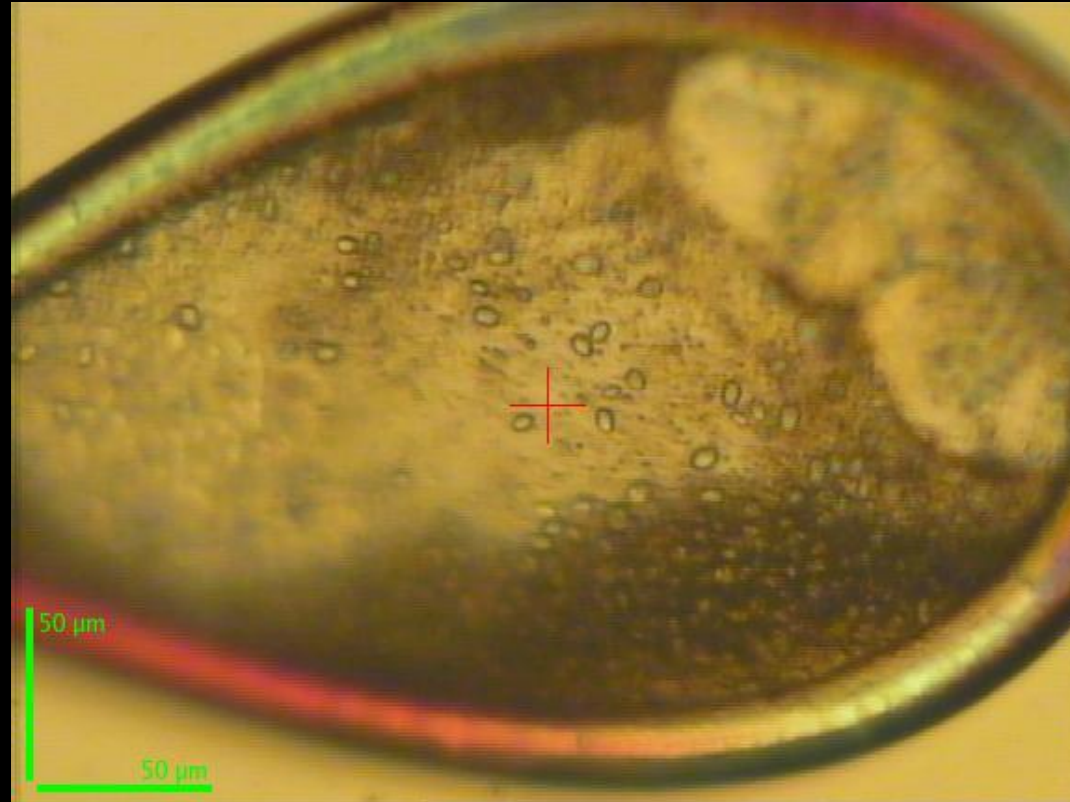
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Experimental Setup for MX Tomography

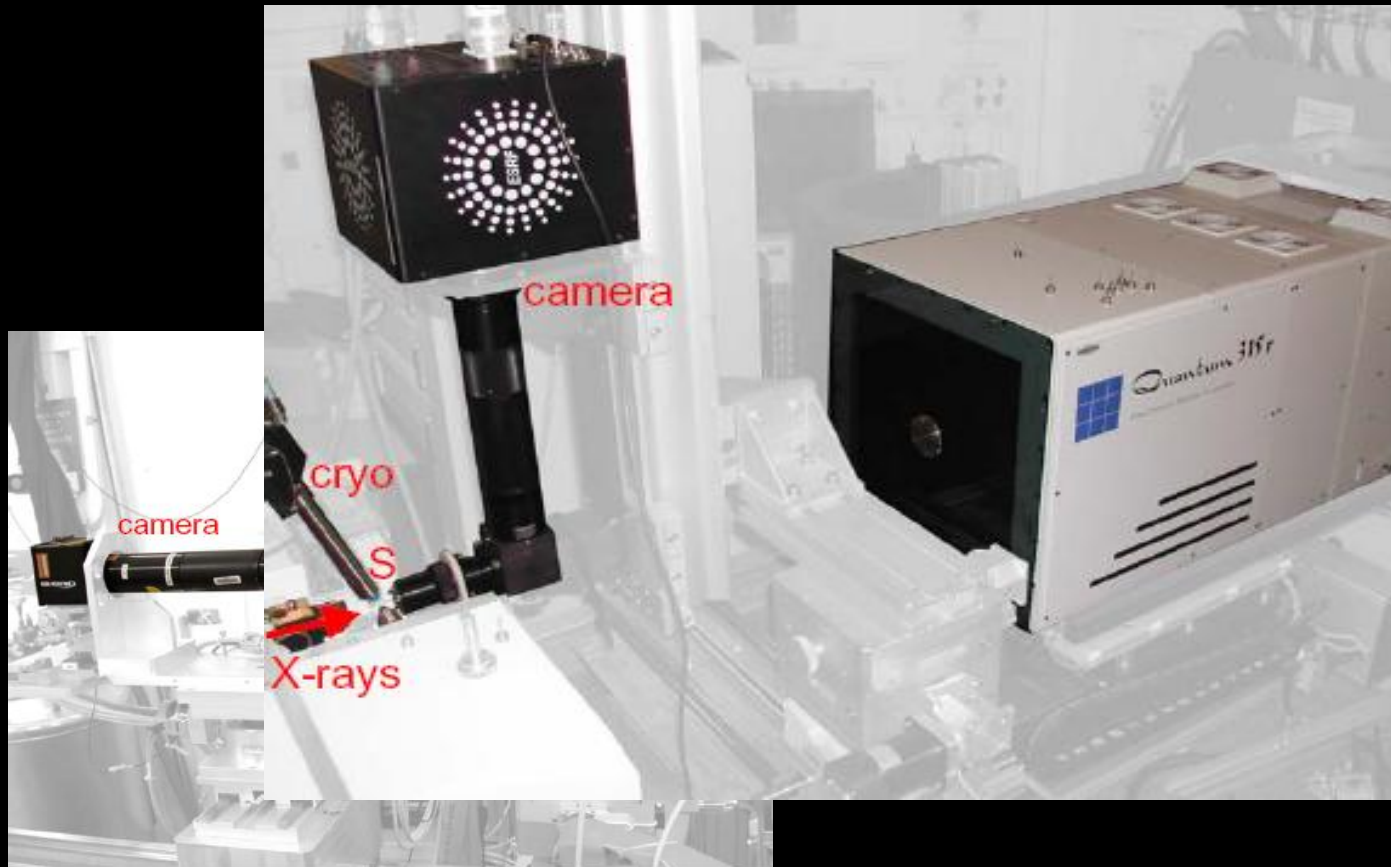
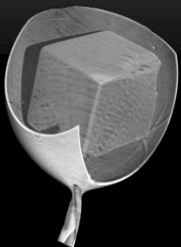


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DNA samples

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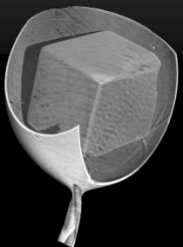
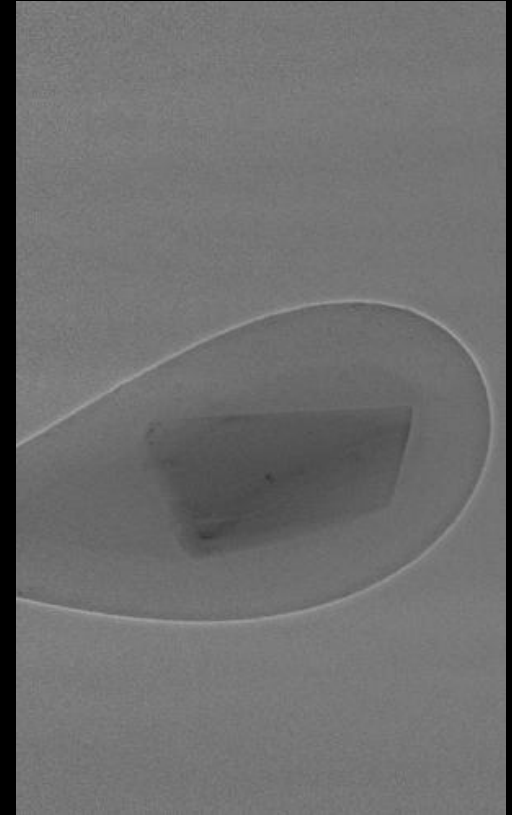
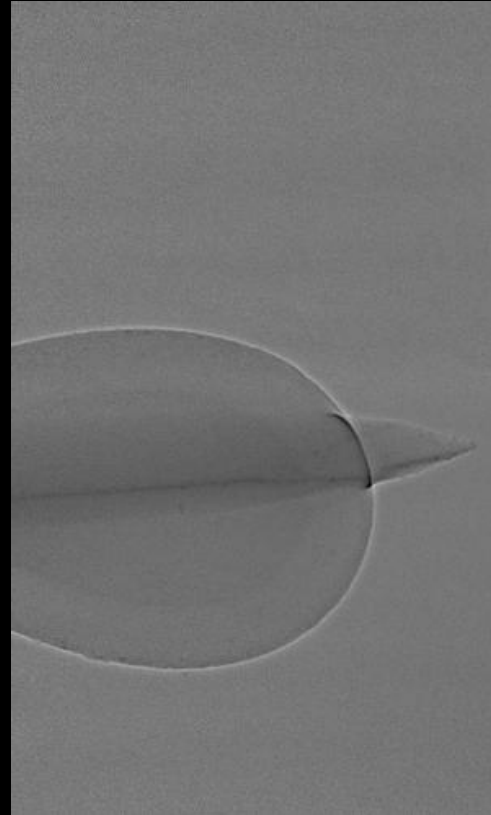
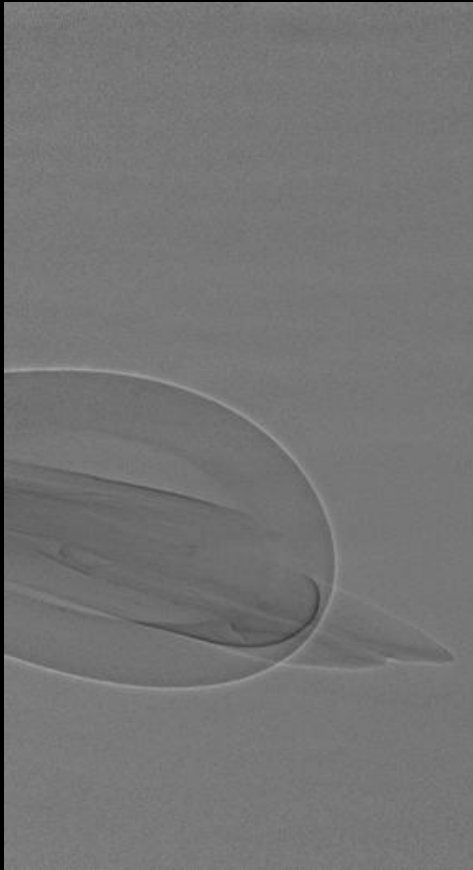
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Thermolysin

Beamlines

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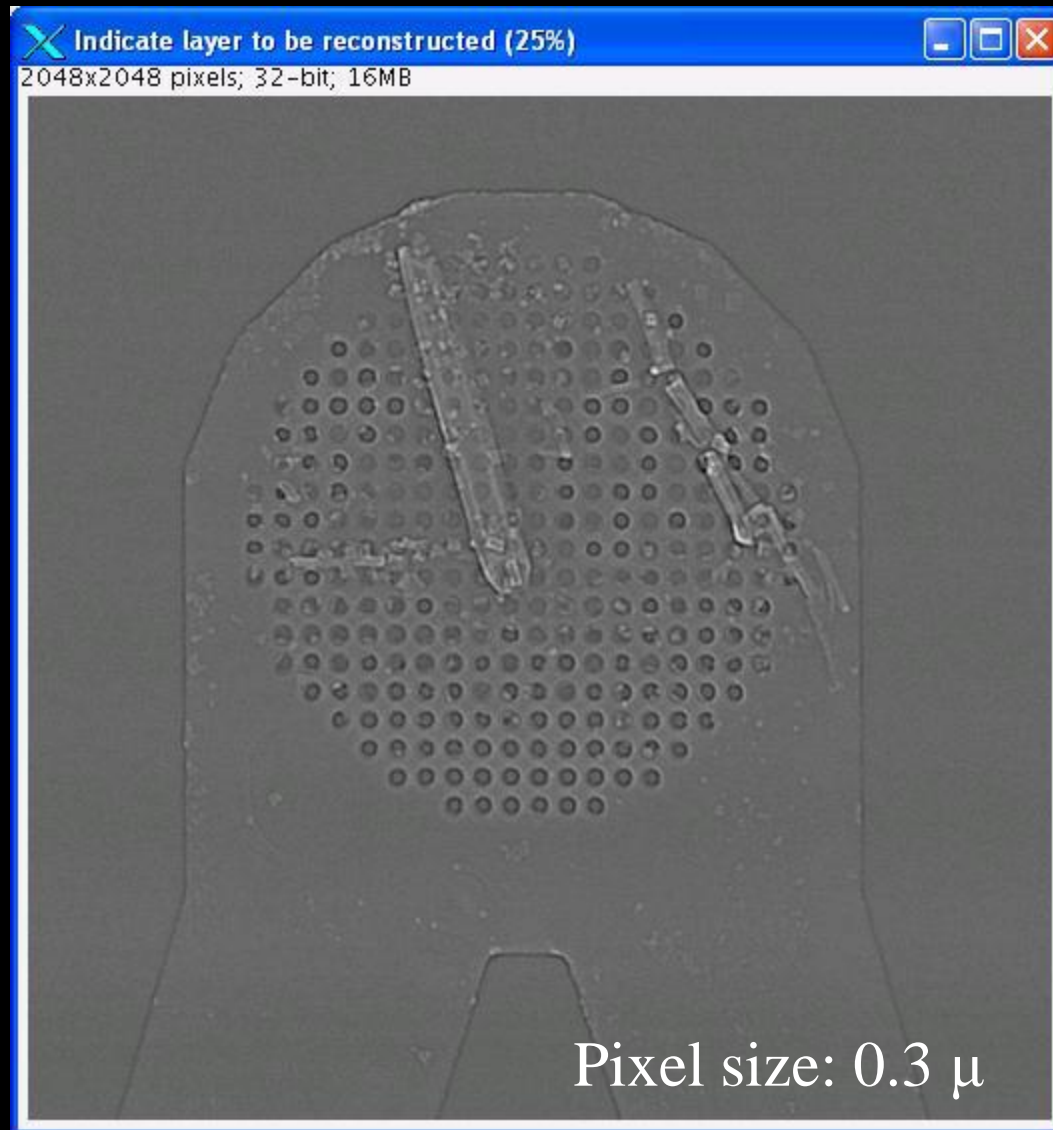
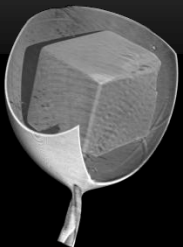
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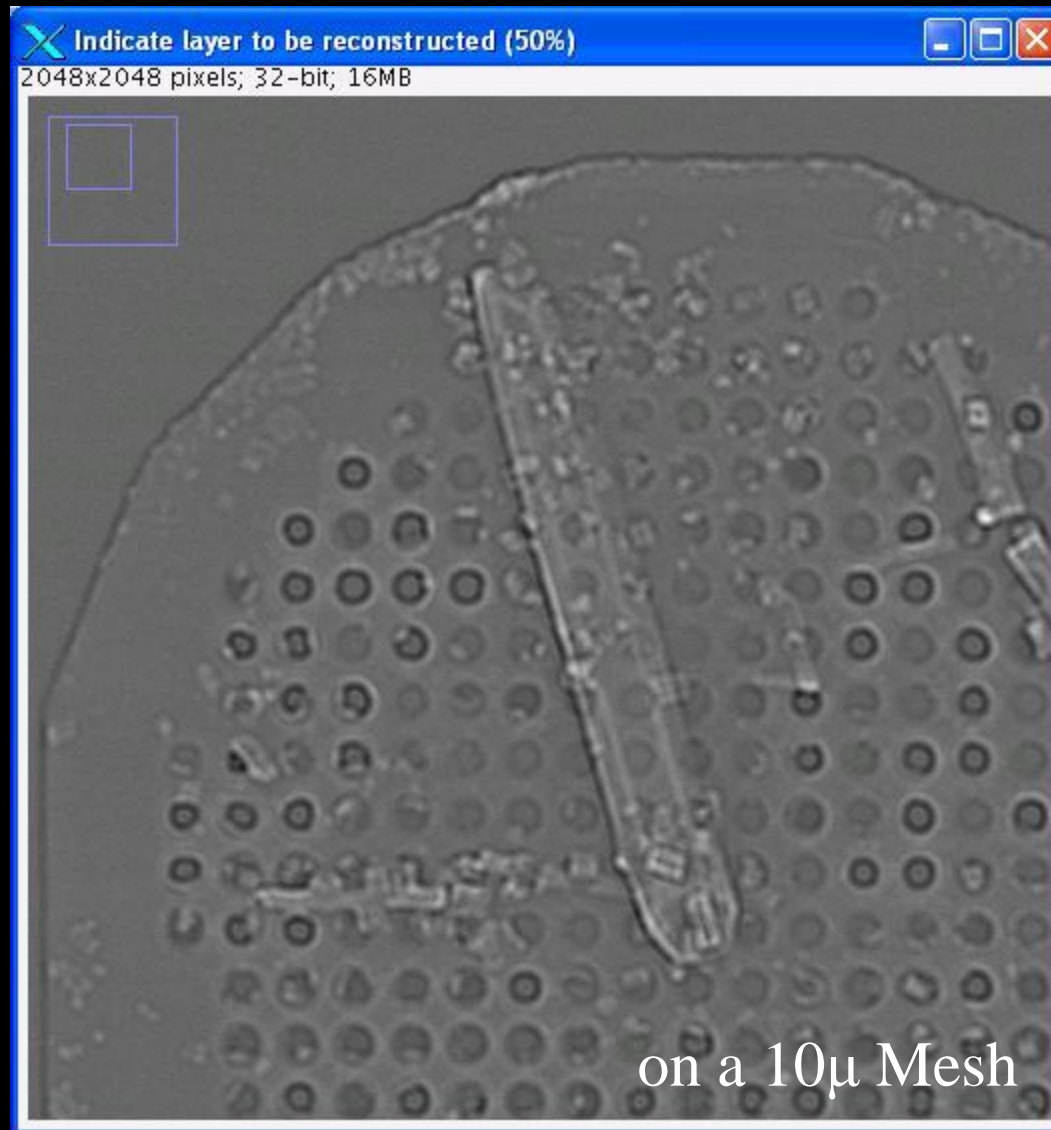
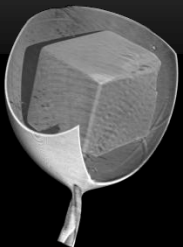
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on a 10μ Mesh



Virtual Beamline

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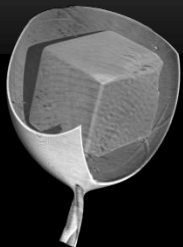
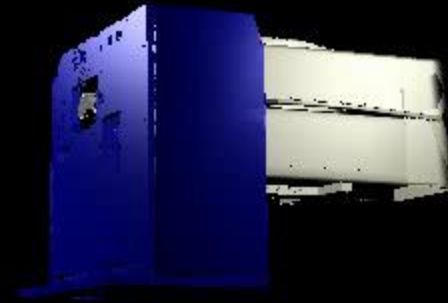
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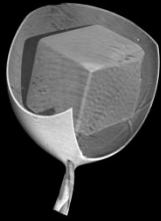
Summary



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Acknowledgements



EMBL-Grenoble

Florent Cipriani, Franck Felisaz, Julien Huet, Alexandre Gobbo, Bernard Lavault, Andrew McCarthy, Raimond Ravelli

Kappa Workgroup

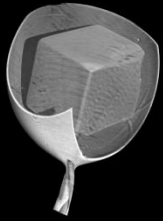
Gerard Bricogne, Gleb Burenkov, Ralf Flaig, Pierre LeGrand, Krister Larsson, Uwe Muller, Sasha Popov, Clemens Schulze-Briesse, Takashi Tomizaki, Johan Unge, Thomas Ursby

ESRF Macromolecular Crystallography

Gordon Leonard, Didier Nurizzo, Sean McSweeney, Martin Walsh

ESRF BLISS/SciSoft/... Groups

Marco DiMichiel, Claudio Ferrero, Matias Guijarro, Mario Lentini, Jens Meyer, Vicente Rey-Bakaikoa, Daren Spruce, Olof Svensson



Thank you for your attention!