Detectors for single-crystal area detector diffractometers

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RIGAKU OXFORD DIFFRACTION

Rigaku, founded in 1951, are well respected for the high performance and stable rotating anodes around the world.

- Our first rotating anode was built in 1952

Oxford Diffraction, in its many guises (Kuma, Varian, Agilent) has always retained they key people responsible for success in software, CCD design and the worlds first dual source systems

- The Gemini was launched at the IUCr in Florence 2004

A NEW BEGINNING

The joining of Rigaku and Oxford opens up a realm of possibilities, the merging of superior hardware, software and expertise will allow for an exciting future in single crystal diffraction. The merging of the two groups is represented in our new logo which takes elements from both.

Expertise from both R&D groups is now shared providing an exciting future for single crystal X-ray diffraction

Roots PhD at UNIL 1992…

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Frustration 1993: incommensurates

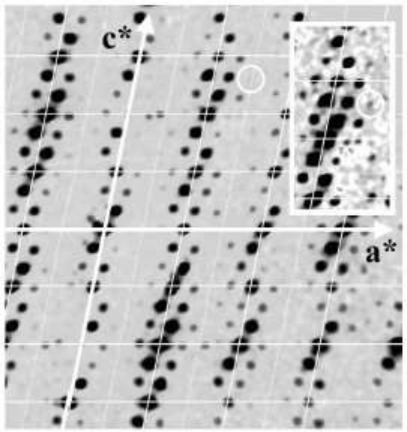
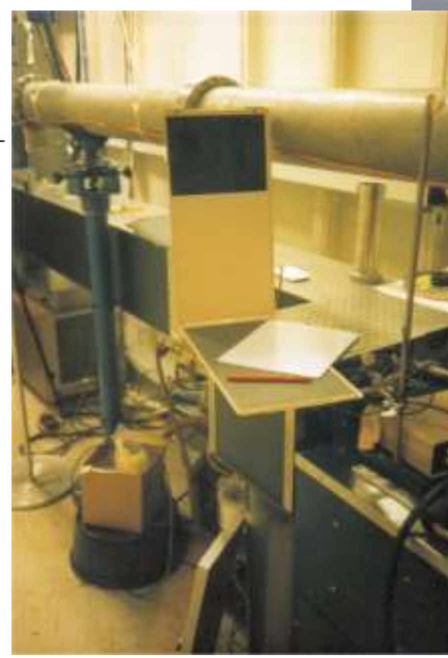
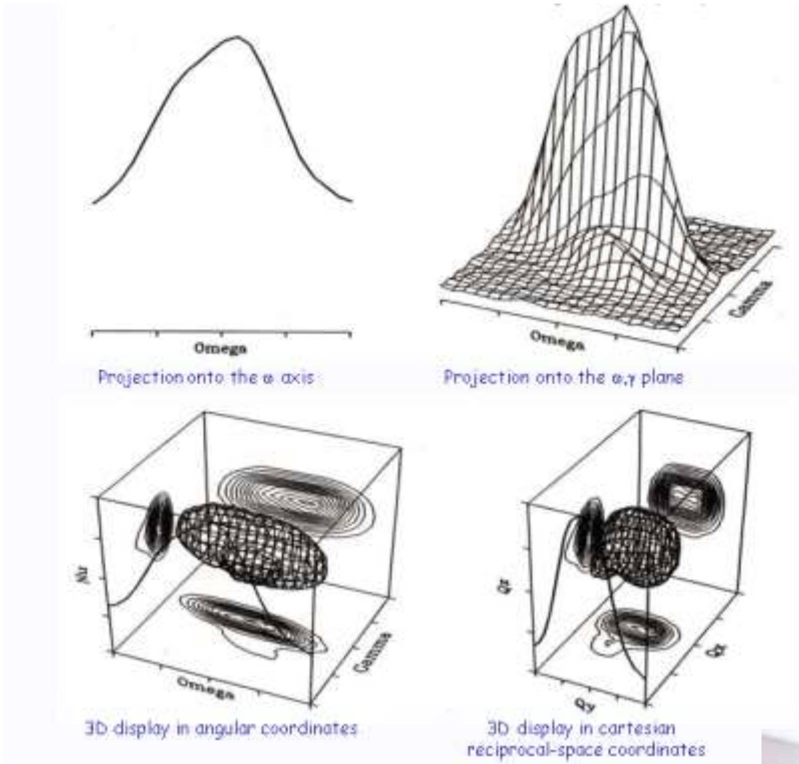


Figure 2
Reconstruction of the reciprocal plane *h2l* in γ-sodium carbonate. The intersections of dotted and solid white lines correspond to main reflections systematically absent as a result of the C centering. The white circle shows the position of the fifth-order satellite 2215. This reflection is visible in the insert showing the corresponding area on a different scale.

340 Michal Dusek *et al.* • Sodium carbonate revisited



Efficiently measure incommensurate samples?



(McIntyre, Neutron News 2, 1992, 15)

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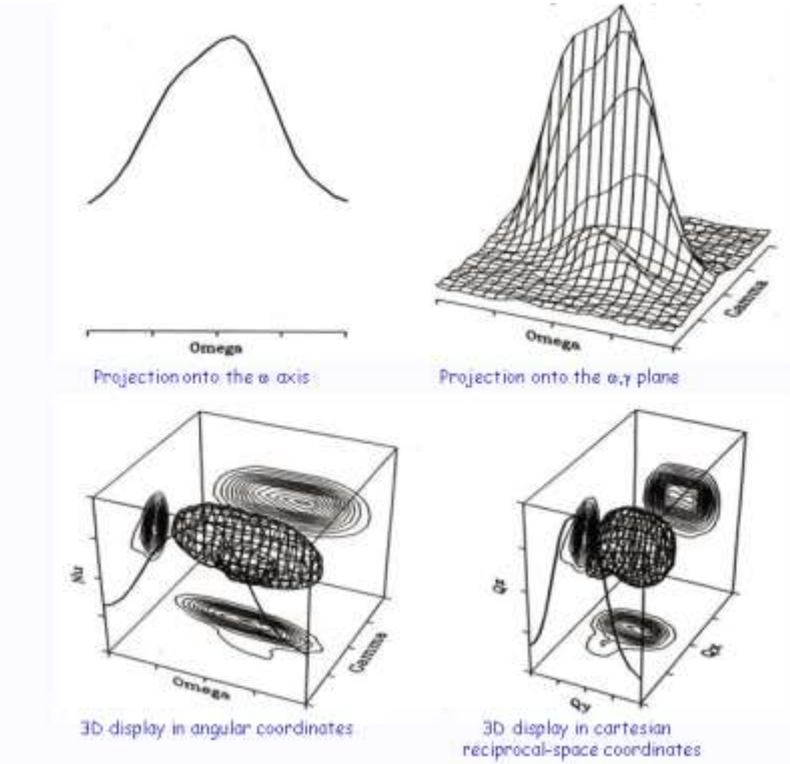
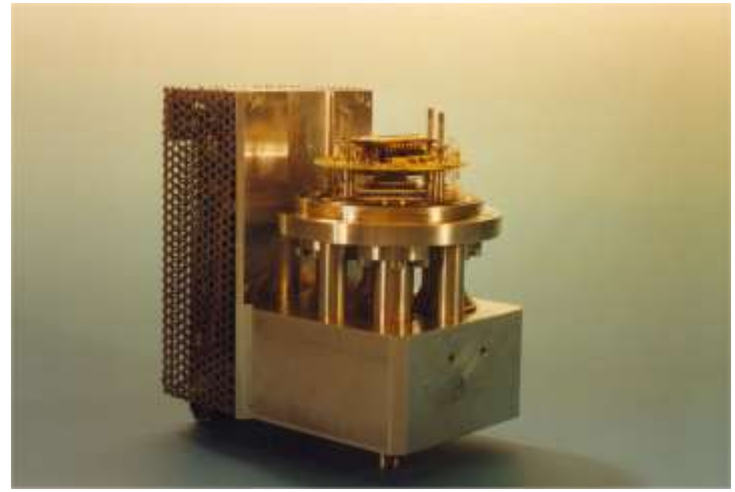
Efficiently measure incommensurate samples?



(McIntyre, Neutron News 2, 1992, 15)



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A ‘Gedankenexperiment’

- We build an area detector diffractometer
- Have source
- Have goniometer
- Have an ‘ideal detector’

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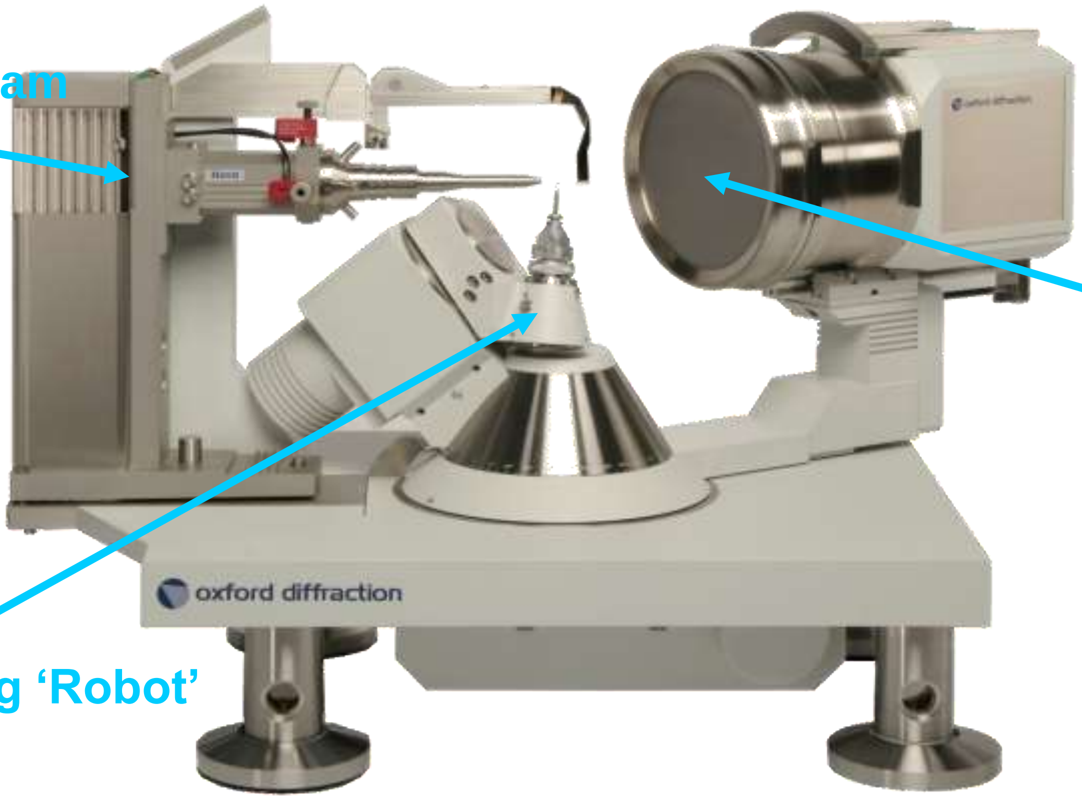
A 'Gedankenexperiment'



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The ‘ideal’ detector

The 'ideal' detector

- What should it do?
  - Experiments at Cu, Mo, Ag, synchrotron?
  - Samples 1μm to 1mm
  - Fast, precise

- What kind of properties
  - Size
  - Resolution
  - 'Color': Energy
  - Detectivity
  - Speed
  - Practical operation
  - Price

Jim Pflugrath once said:
"The ideal detector tells you where every photon landed and when."

XtaLAB Synergy S/R with HyPix6000HE

- ACA 2016 launch HyPix 6000
  - 100 microns resolution with top-hat PSF
  - 100Hz – shutterless with near 0 dead-time
  - 10 deg/sec top dc speed, very fast positioning
  - PhotonJet sources
  - WIT in 17s
  - Full mmm data set in ~2min
  - P1 = full sphere in <15min

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Synergy R and HyPix6000HE:

100µm pixel, 100Hz operation, 10deg/s scans

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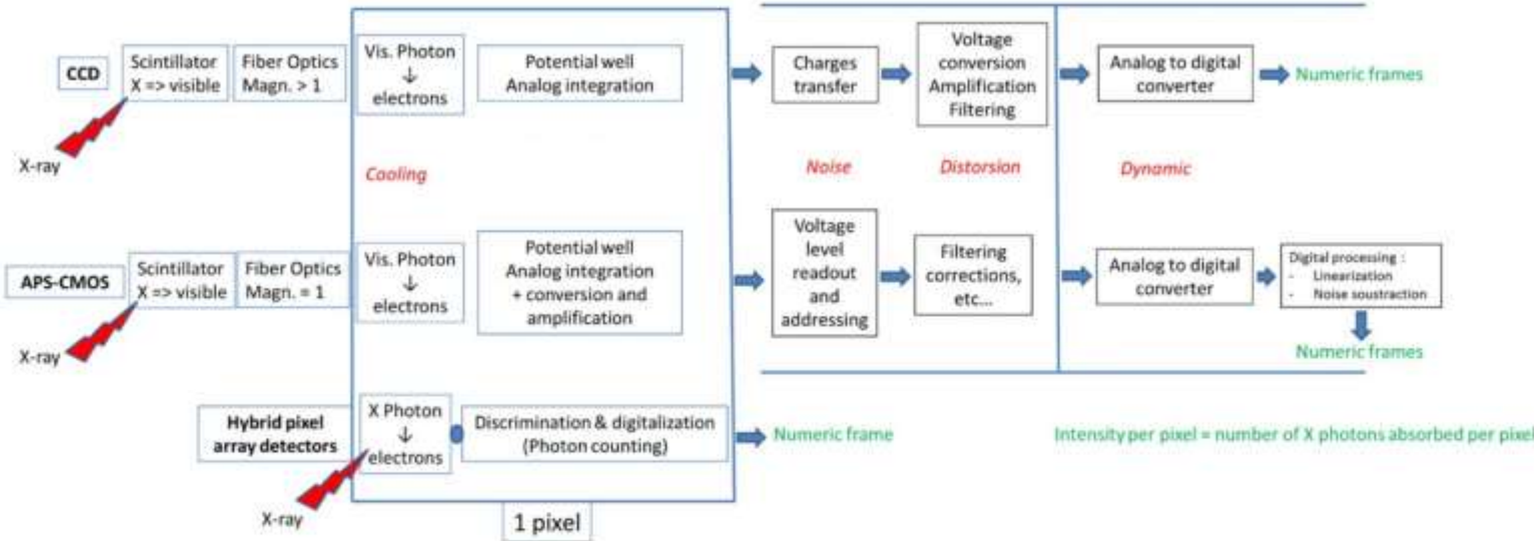
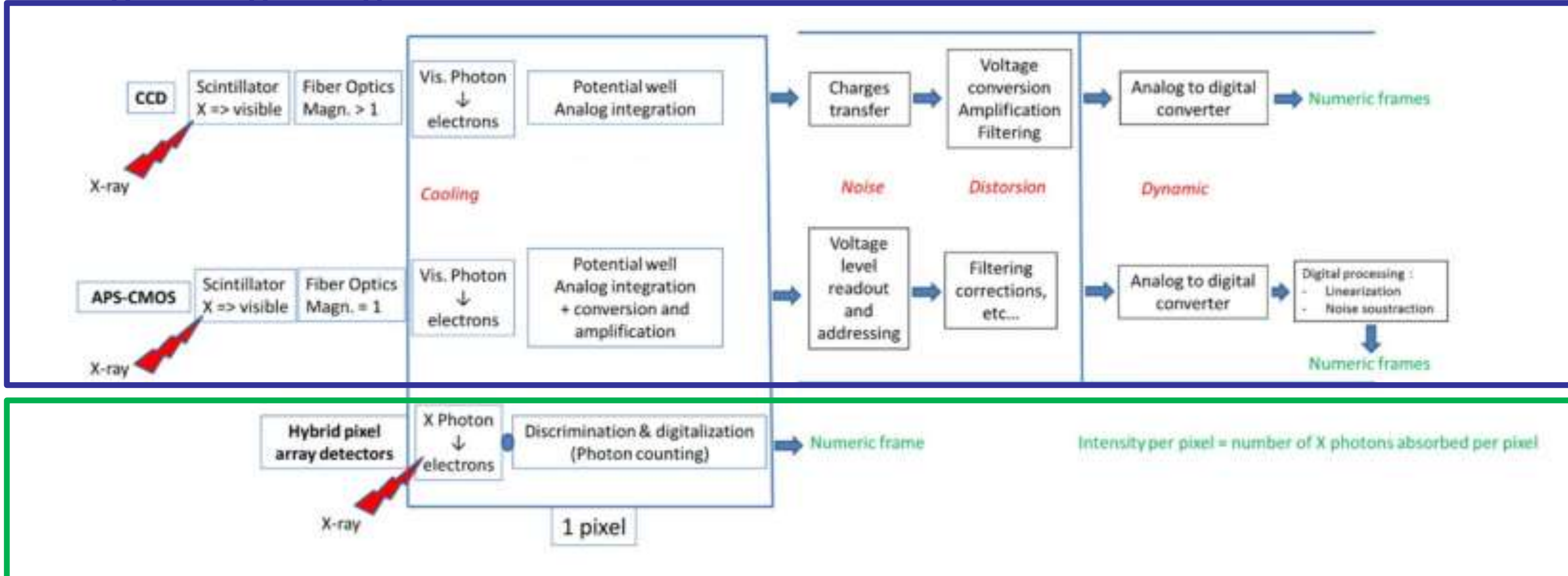
The 'real' detectors SX AD



*adapted from: P Allé, E Wenger, S Dahaoui, D Schaniel and C Lecomte: 'Comparison of CCD, CMOS and Hybrid Pixel x-ray detectors: detection principle and dataquality' Phys. Scr. **91** (2016) 063001

The 'real' detectors SX AD

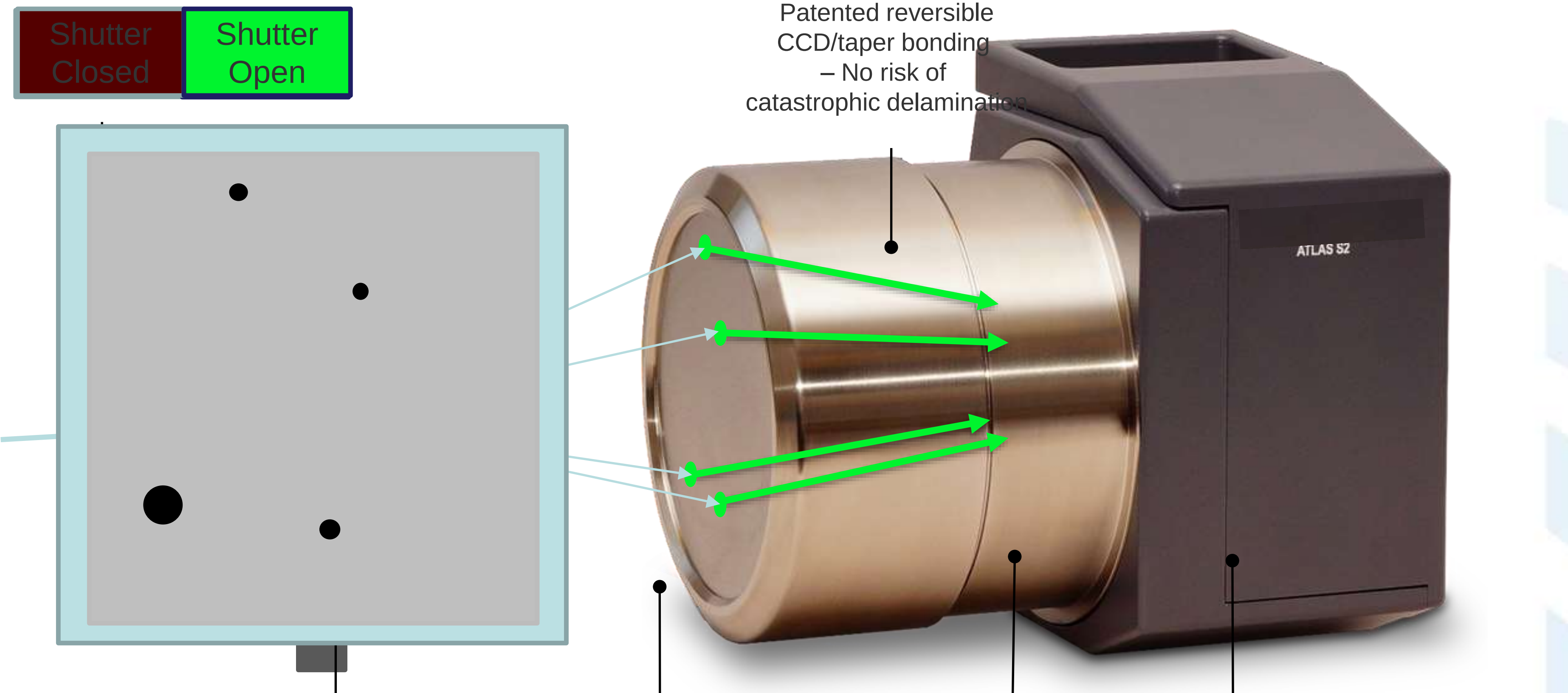
Charge integrating detectors



Event counters: HPAD, HPC – Hybrid pixel counters

*adapted from: P Allé, E Wenger, S Dahaoui, D Schaniel and C Lecomte: 'Comparison of CCD, CMOS and Hybrid Pixel x-ray detectors: detection principle and dataquality' Phys. Scr. **91** (2016) 063001

Detecting X-rays with a CCD Integrative detector



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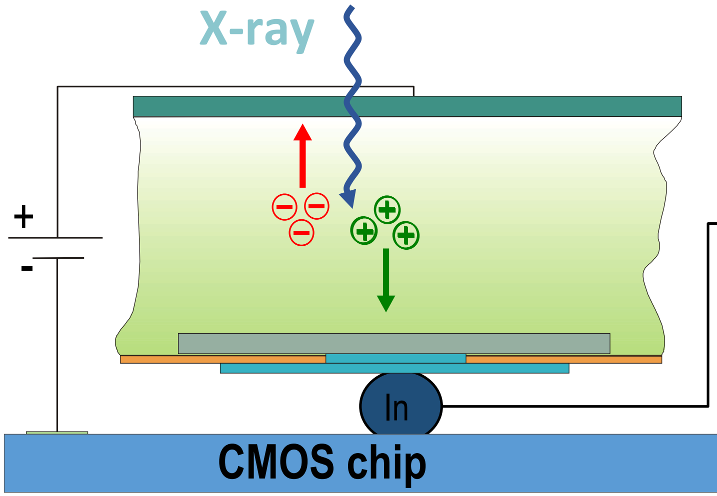
The closest thing to an ideal detector…

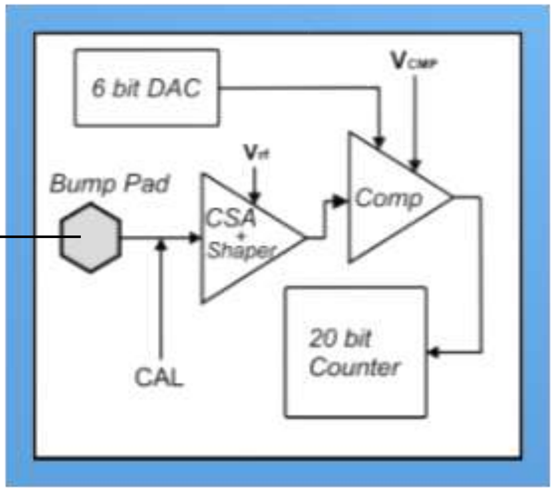
Sensor pixel



Direct Detection of X-rays in silicon sensor

→ Point Spread Function of 1 pixel

Readout pixel



Single Photon-counting in CMOS

→ no readout noise & dark current

→ high dynamic range (20 bit)

→ fast readout

CMOS: Complementary metal–oxide–semiconductor

- CMOS is only a production technology
- CMOS based detectors can be very different

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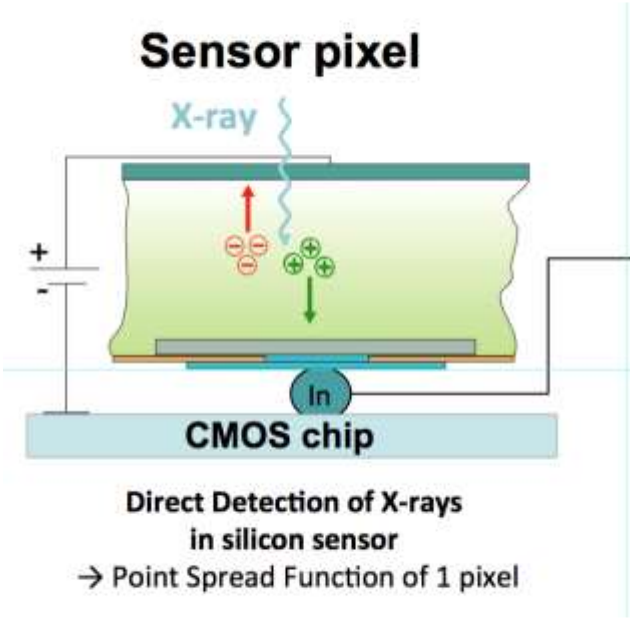
adapted from: Dectris

Key Features of HPC Detectors

- Direct detection of X-ray photons – no conversion to light
- Excellent point spread function – top hat

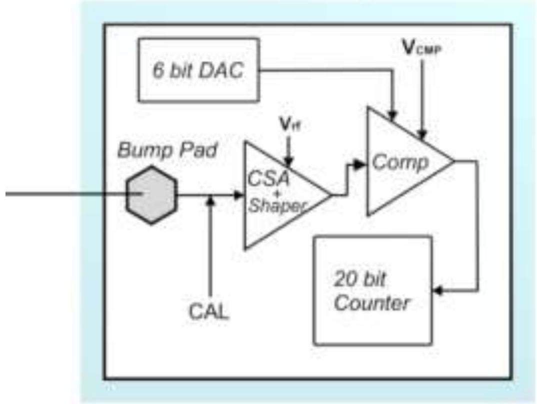


adapted from: Dectris

Key Features of HPC Detectors (Pilatus)

- Excellent signal-to-noise ratio via single photon counting
- Adjustable threshold to suppress fluorescence
- High dynamic range: 1:1,048,576 photons *per pixel*
- High counting rages: up to 2 x 10^6 photons per second *per pixel*
- Short readout time: 7 ms
- Frame rate up to 20 images per second

Readout pixel



Single Photon-counting in CMOS

→ no readout noise & dark current
→ high dynamic range (20 bit)
→ fast readout

adapted from: Dectris

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The ‘real’ detectors SX AD

Integrative detectors ‚CPAD‘		Event counters ‚HPAD‘ HPC – Hybrid pixel counters
Indirect detection via X-ray scintillator		Direct detection via photo-electric effect
Light conduction via taper/fiber glass		-
Light detection ‚Integrating‘		Charge detection ‚Photon counting‘
CCD	CMOS	-
FET->ADC->memory		Memory
No energy discrimination		Energy thresholds

The ‘real’ detectors SX AD

Integrative detectors ‚CPAD‘		Event counters ‚HPAD‘ HPC – Hybrid pixel counters
Indirect detection via X-ray scintillator		Direct detection via photo-electric effect
Light conduction via taper/fiber glass		-
Light detection		Charge detection Photon counting
CCD	CMOS	-
FET->ADC->memory		Memory
No energy discrimination		Energy thresholds






What is data quality?

$$\frac{I}{\sigma}$$

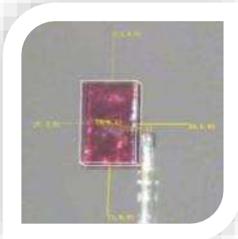
$$I$$

$$\left(\sum \frac{I}{\sigma}\right) \Big/ time$$

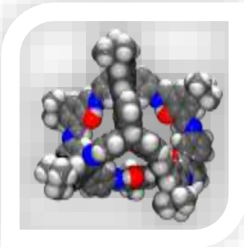
$$\sigma^2 = \sigma_{Inet}^2 + 2\sigma_{back}^2 + \sigma_d^2 + \sigma_{sys}^2$$

The single crystal diffraction experiment

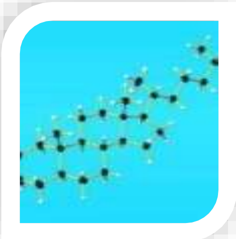

Inorganic Materials


Absorbing Samples

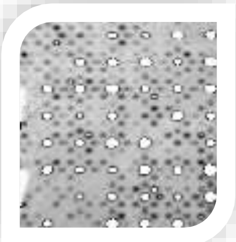

Mo vs. Cu

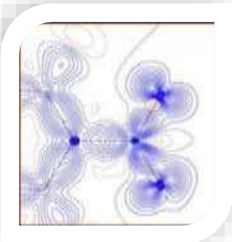

Benefits of Cu


Small Crystals


Absolute Configuration

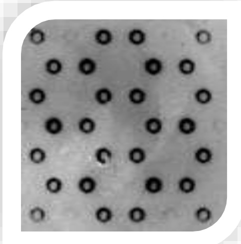

Twinning

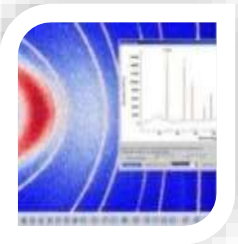

Incommensurates


Charge Density


High Pressure


Large Crystals

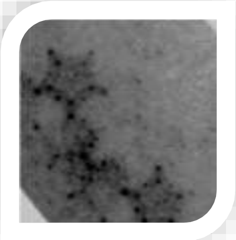

Diffuse Scatter


Powder Diffraction


Photocrystallography


Novel Applications

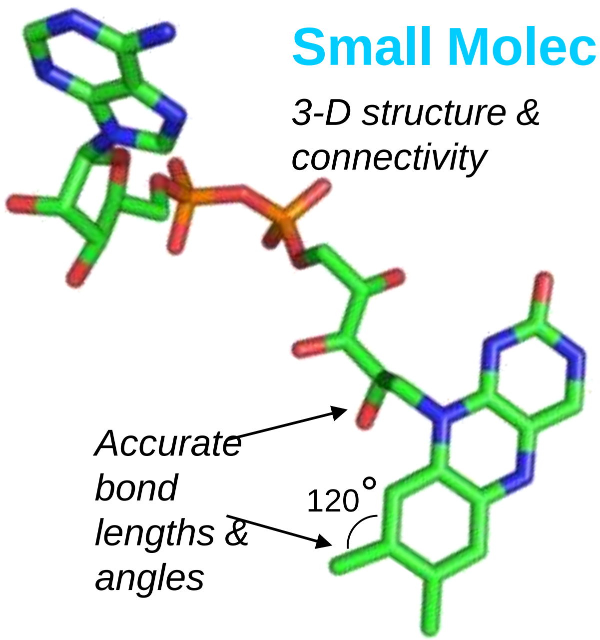

Multi-Temperature


Quasi-Crystals

The single crystal diffraction experiment

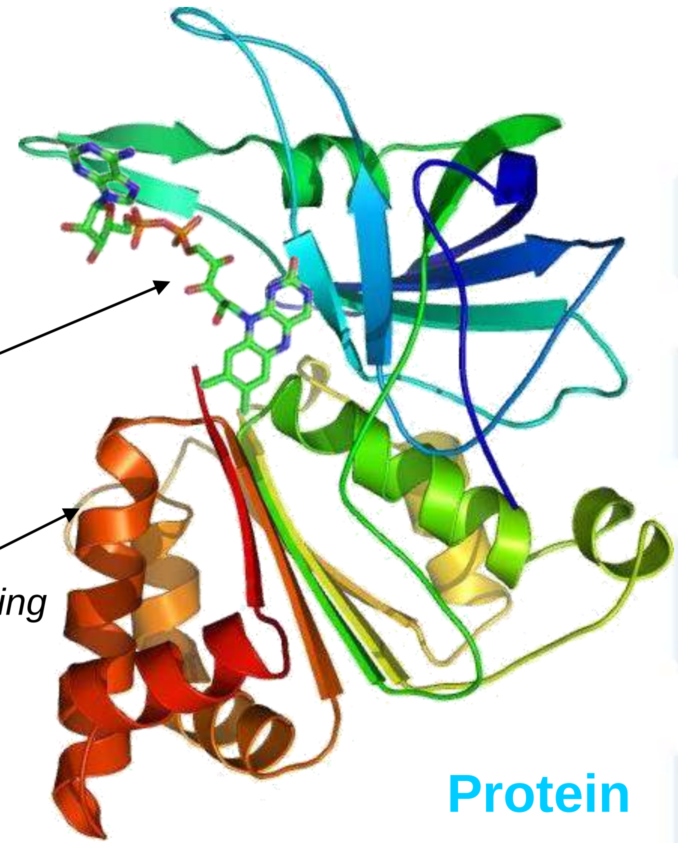
Small Molecule

3-D structure & connectivity



Shape and chemical structure (specificity / activity) of active site

3-D structure / folding



Protein

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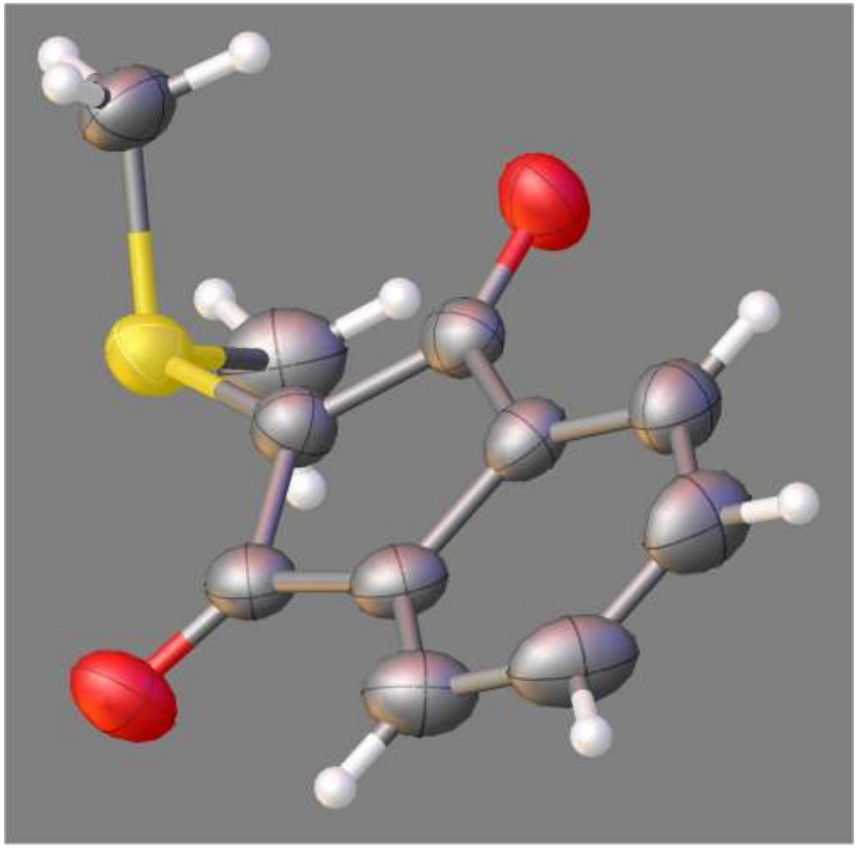
Importance of weak data



- Make a typical experiment
  - Cu radiation
  - Resolution 0.78Ang
  - $(I/sig)_{mean}$ = 15 to 0.837Ang (IUCR)

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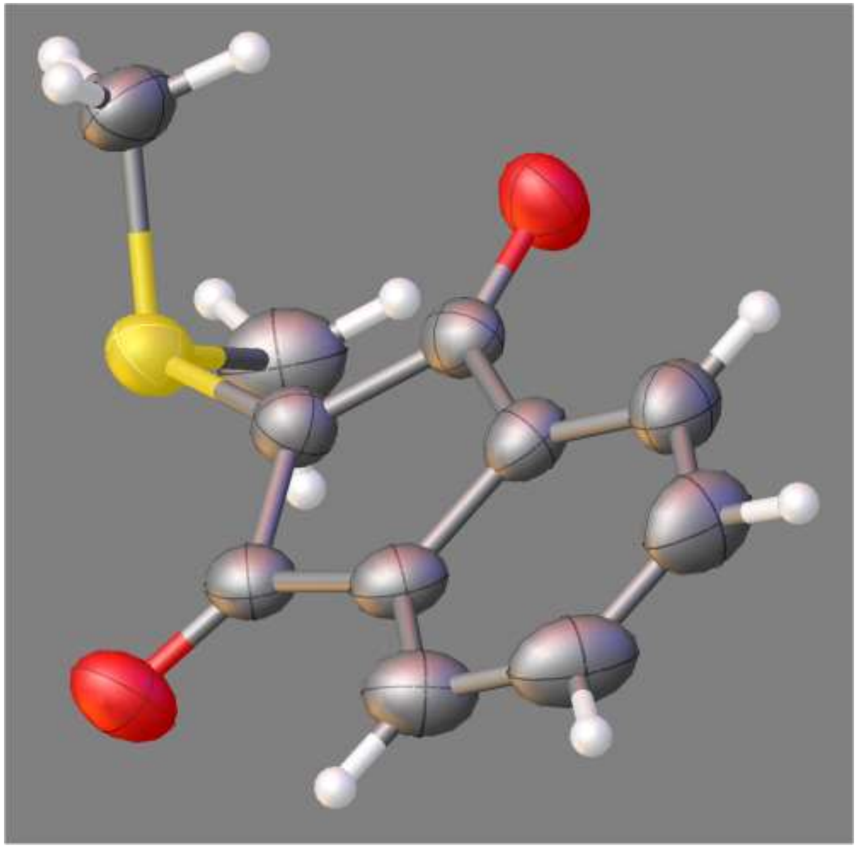
Importance of weak data



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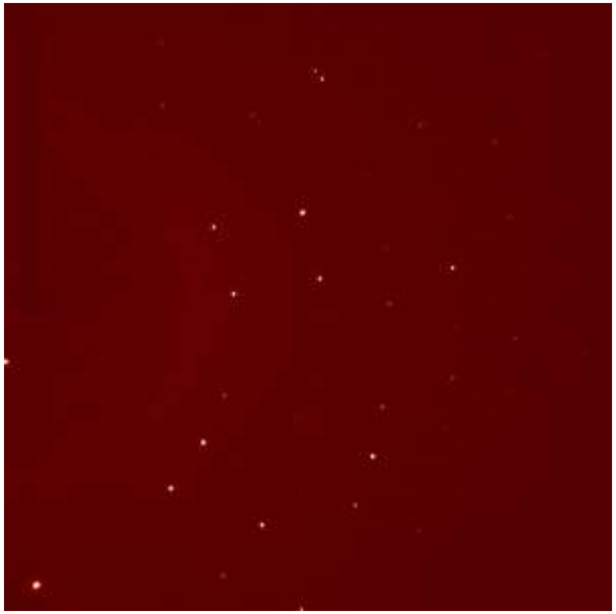

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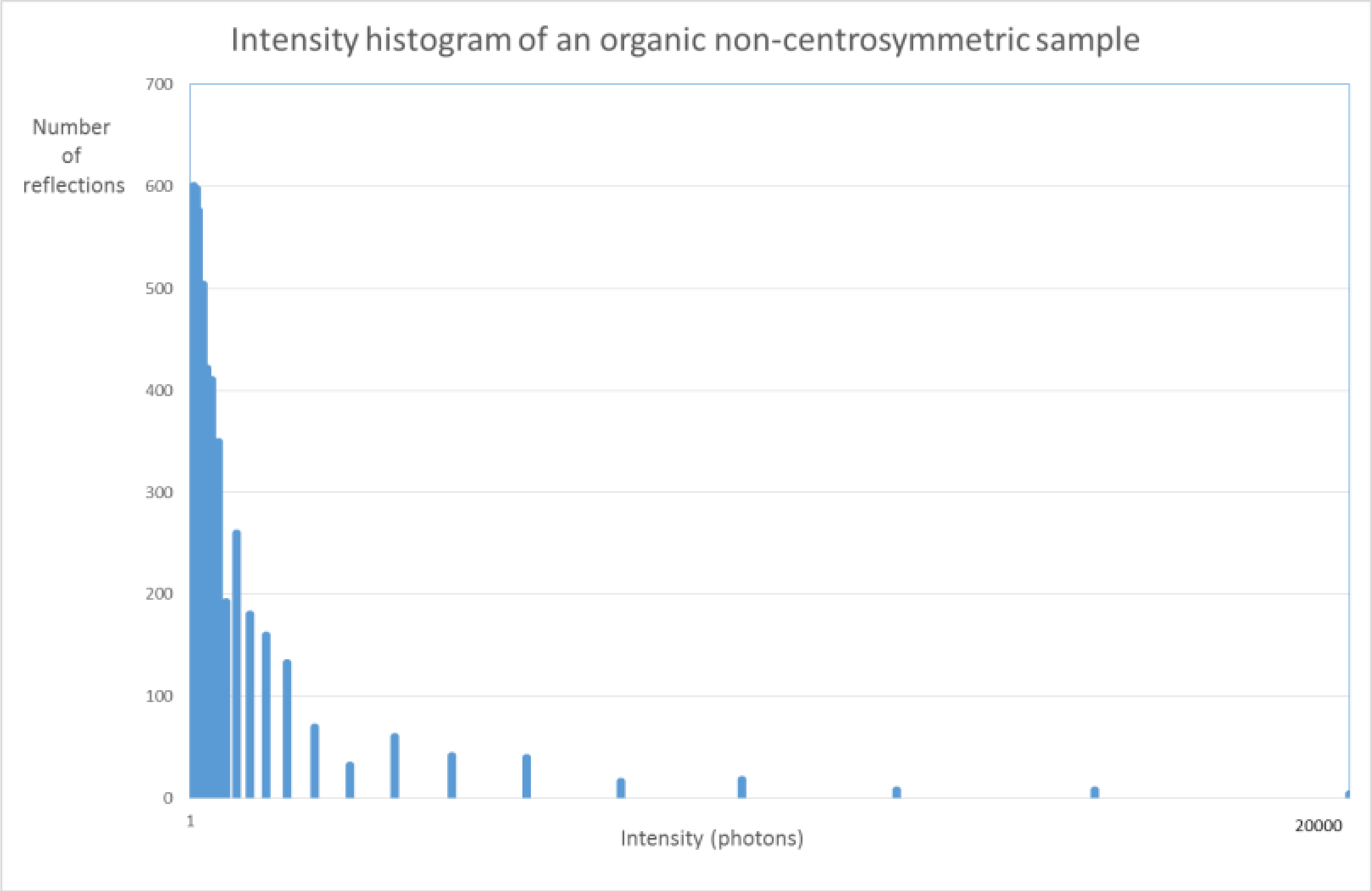
Importance of weak data

Histogram of data



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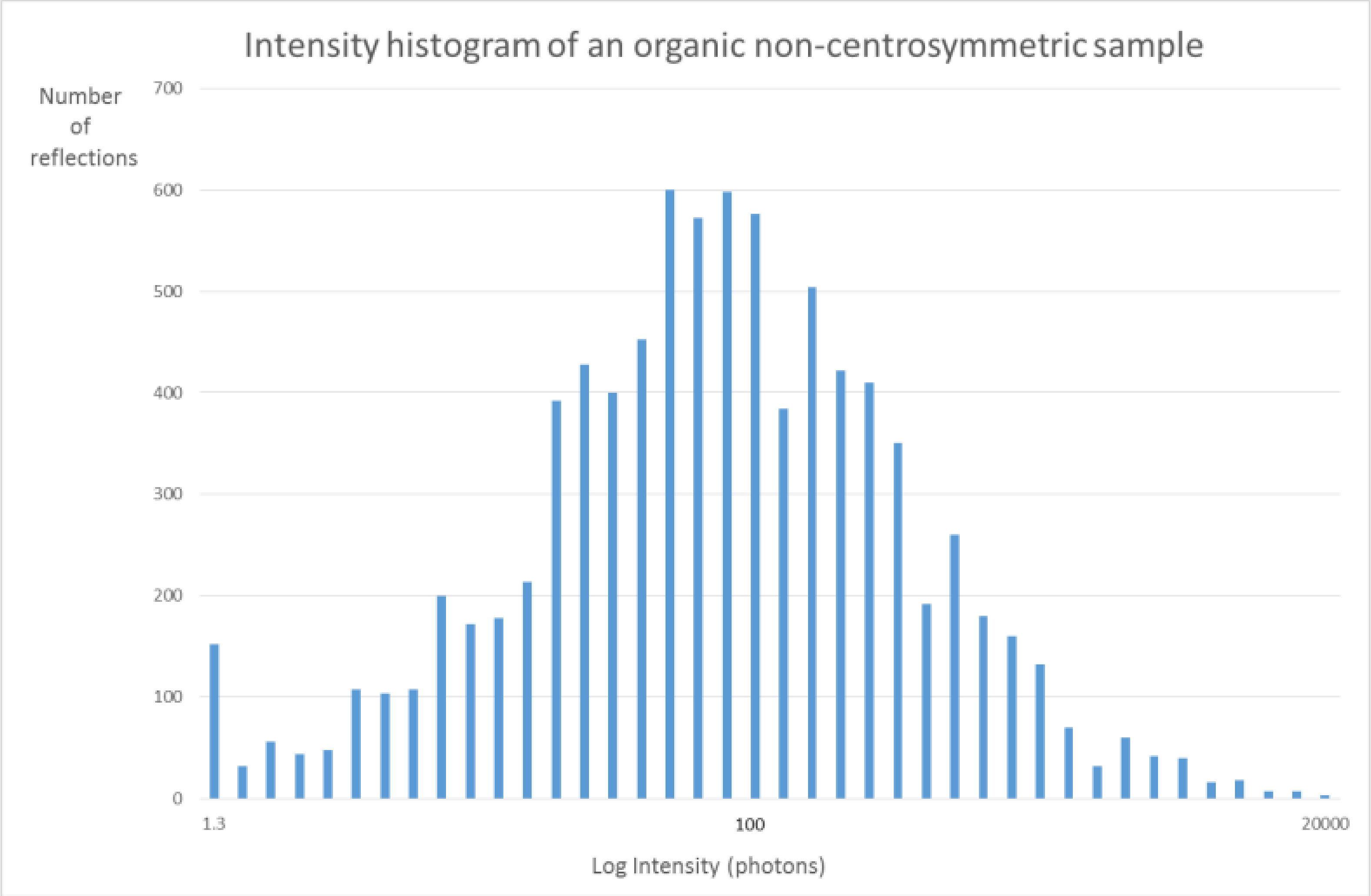
Importance of weak data

Histogram of data



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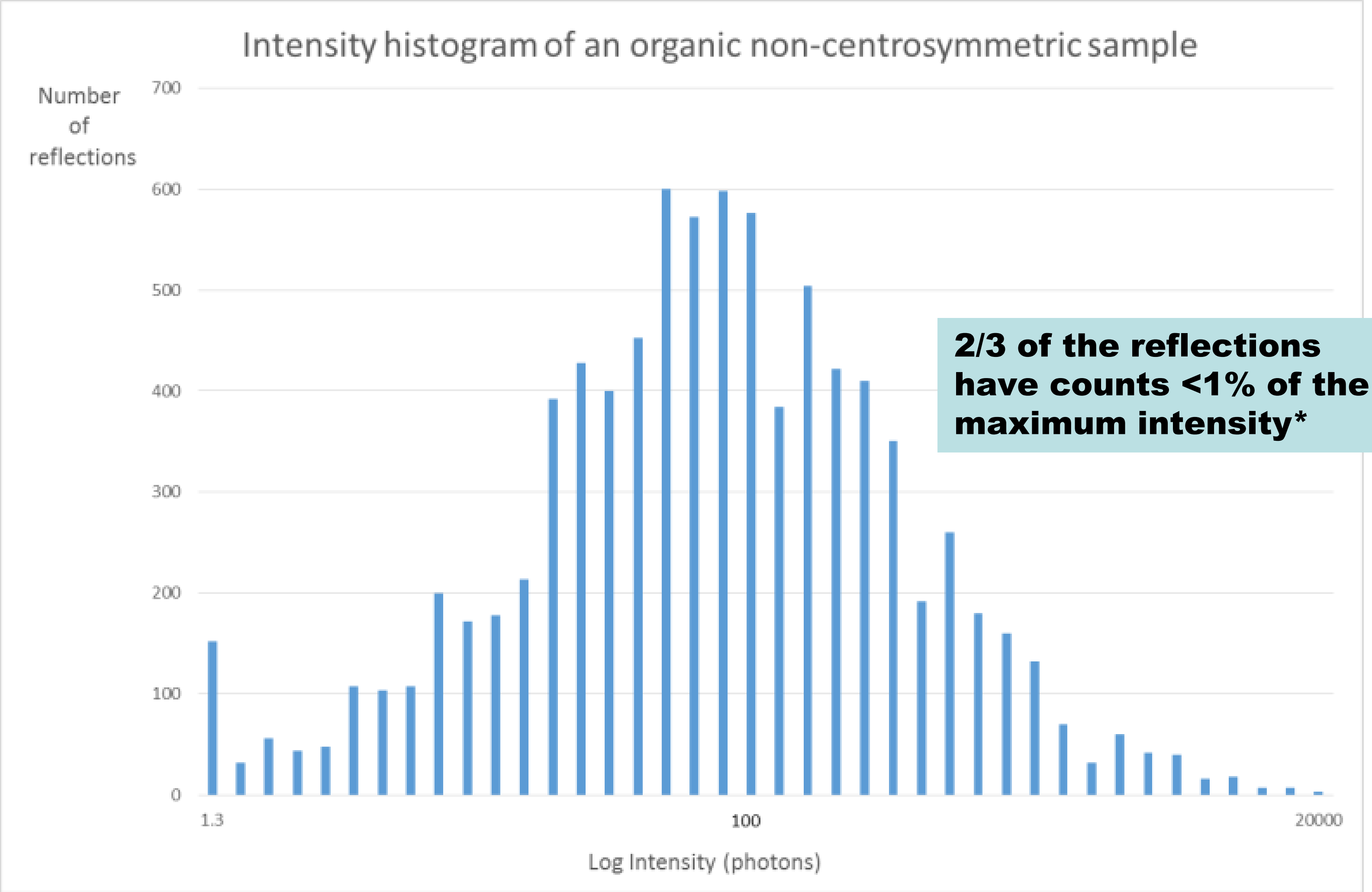
Histogram of data



2/3 of the reflections have counts <1% of the maximum intensity*

*Hirshfeld, F.L.; Rabinowich, D. *Treating Weak Reflexions in Least-Squares Calculations.* Acta Crystallogr. 1973, **A29**, 10–513.;Arnberg, L.; Hovmöller, S.; Westman, *S. On the Significance of 'Non-Significant' Reflexions.* Acta Crystallogr. 1979, **A35**, 497–499.

Detective Quantum Efficiency (DQE)

$$DQE = \frac{T_w \eta_{ph}}{1 + \frac{1}{g} + \frac{A(n_r^2 + i_d t)}{I T_w \eta_{ph} g^2}}$$

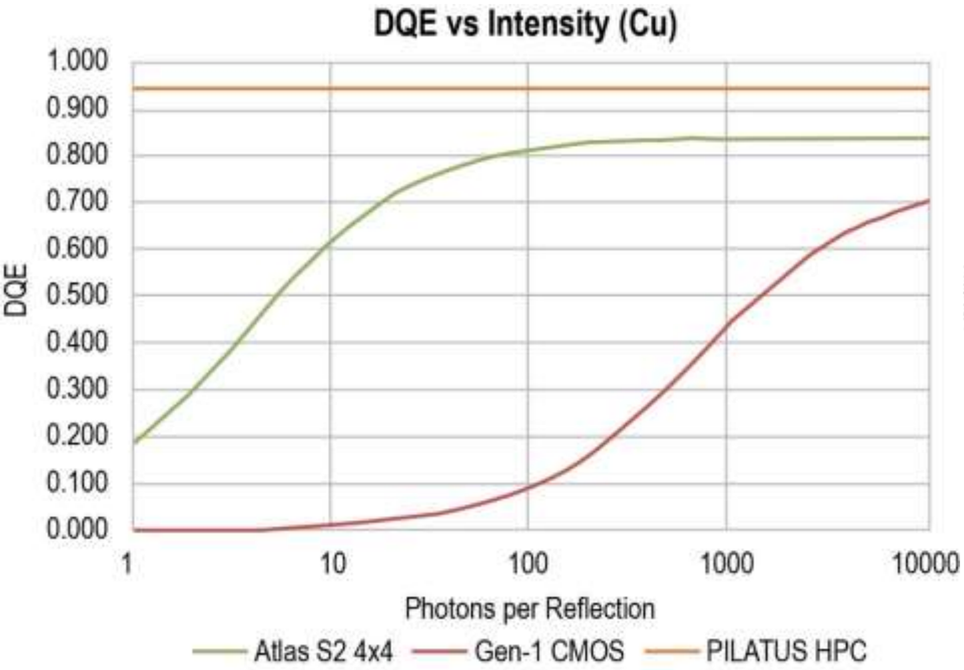
Window
Scintillator

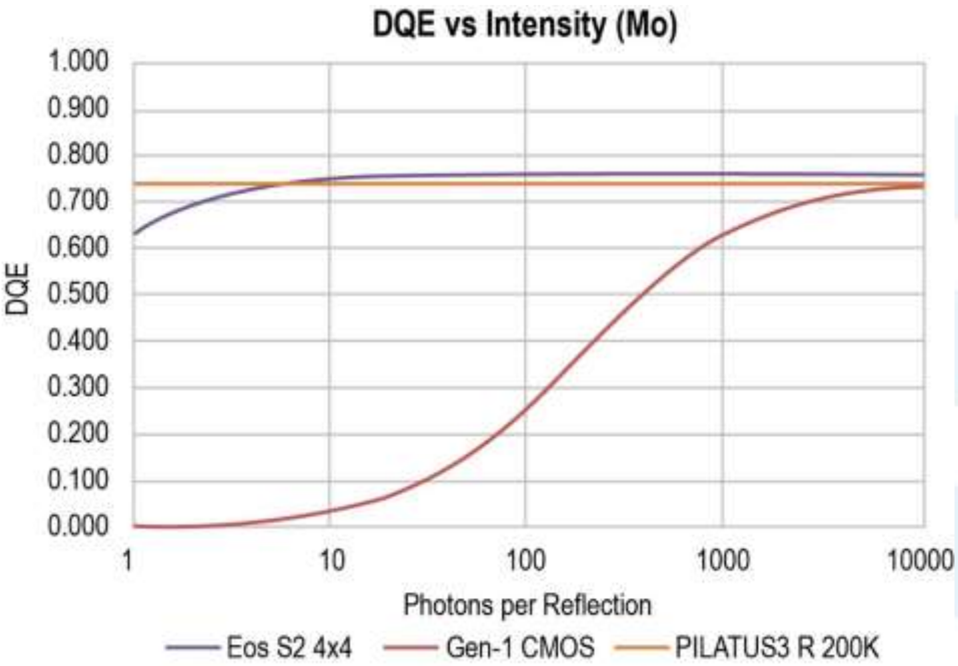
Gain

Noise:
Read and dark

M. Stanton et al., J. Appl. Cryst. (1992). 25, 638-645

DQE: Cu and Mo





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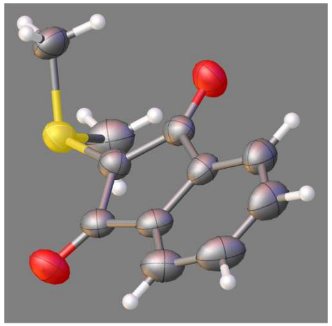
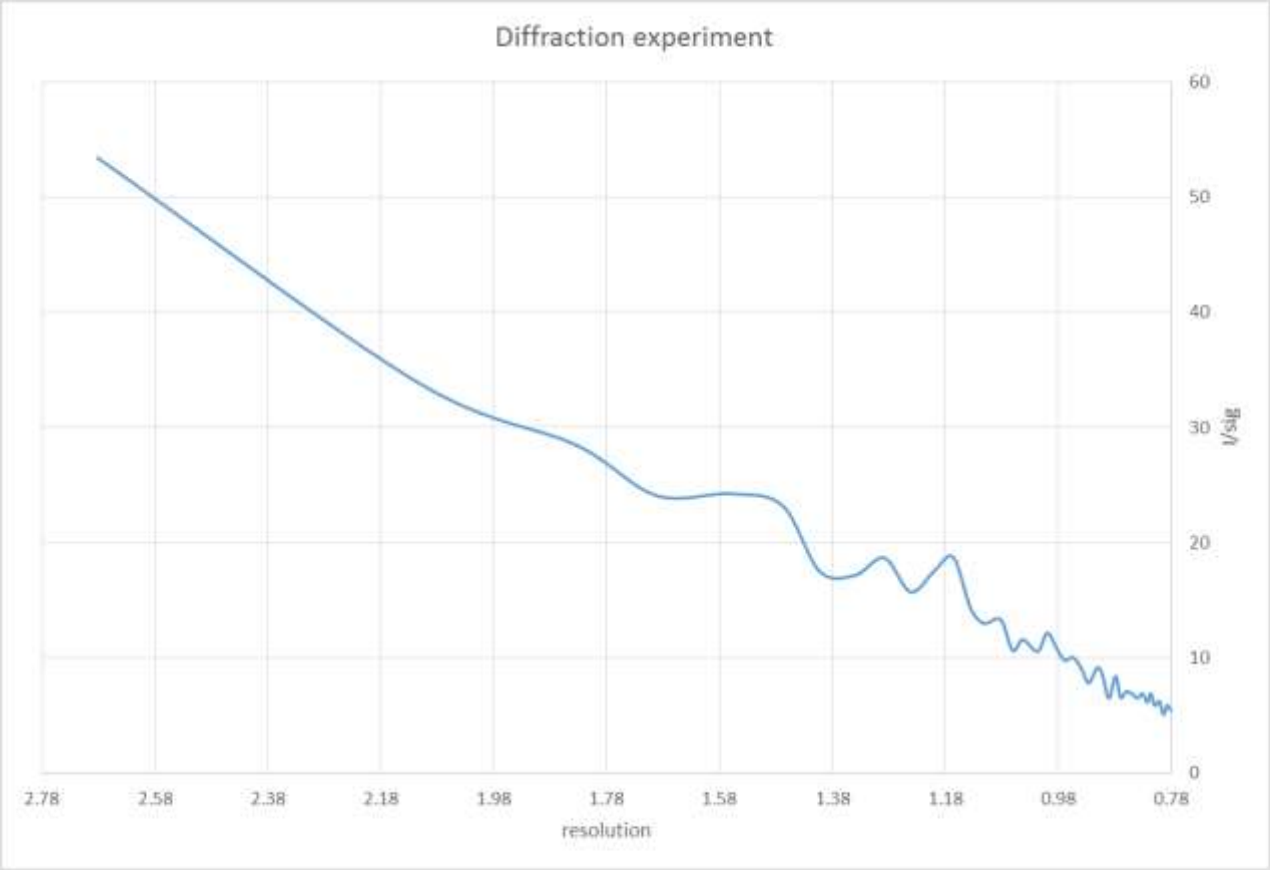


Importance of weak data

Comparison of tech



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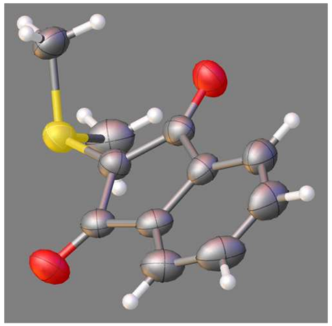

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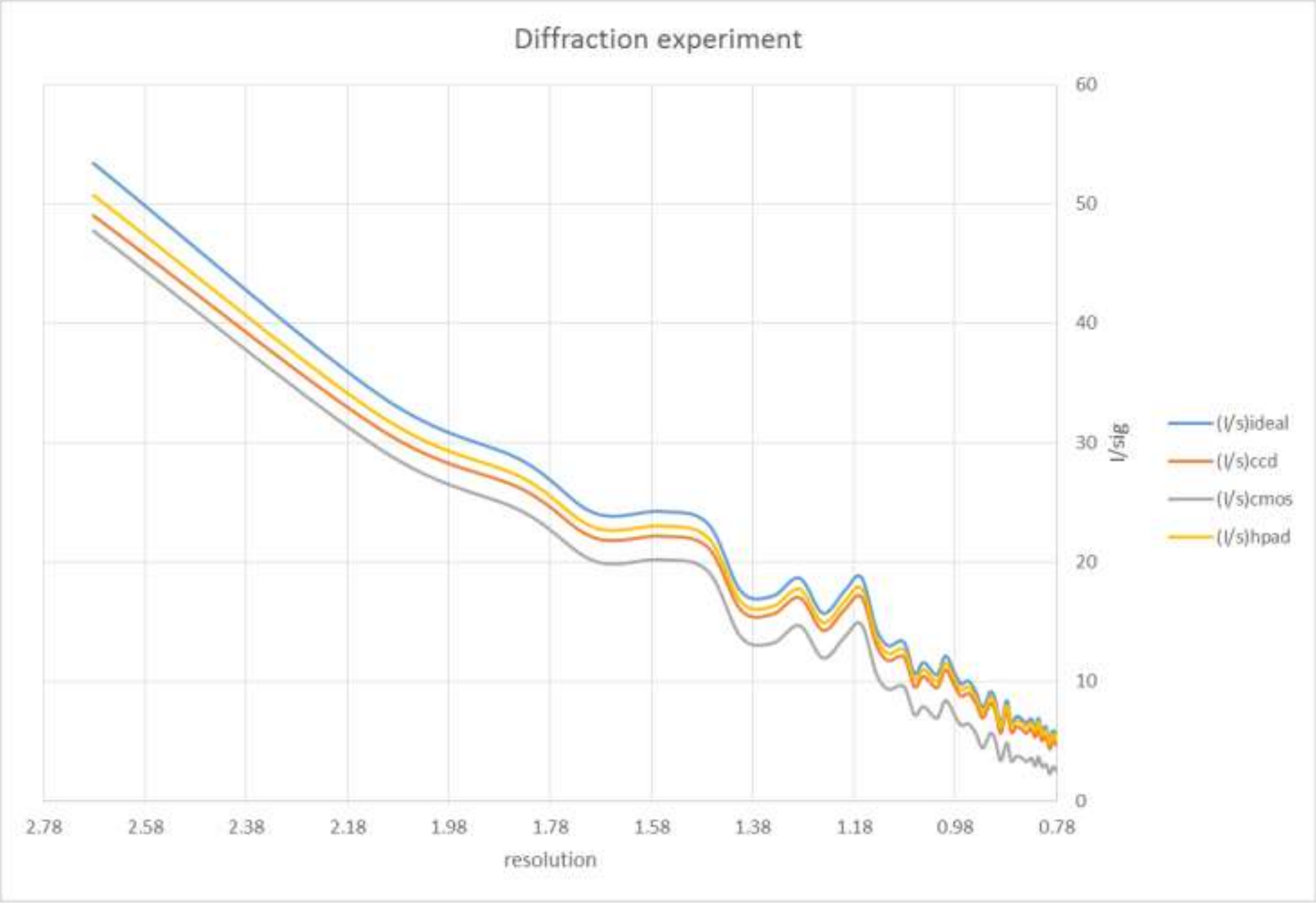
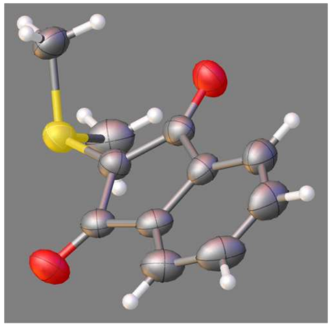
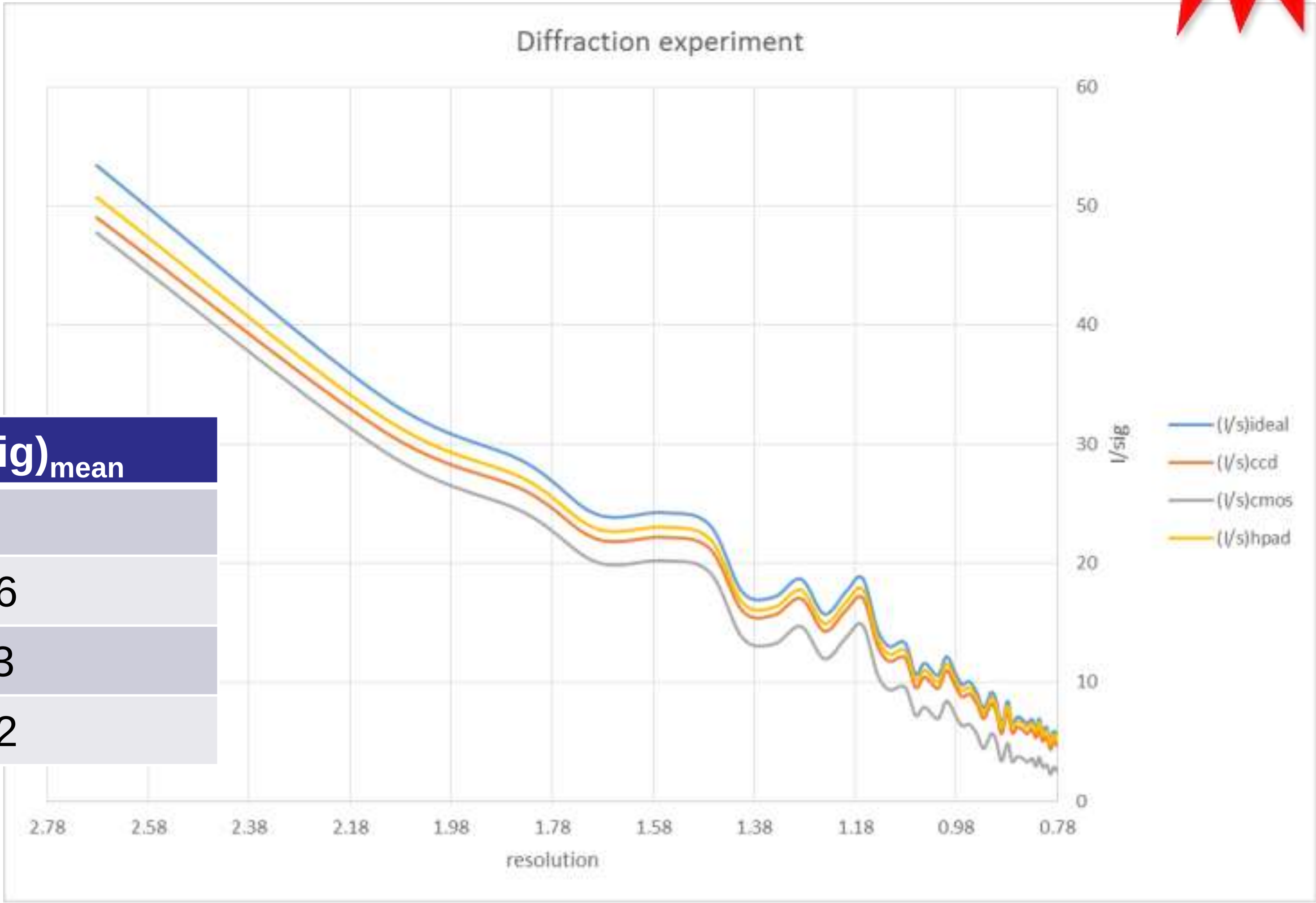




Importance of weak data

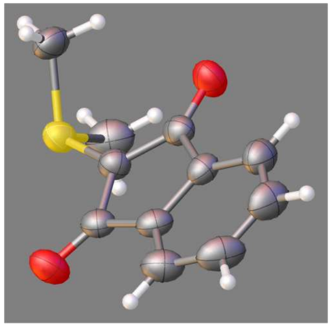
Comparison of tech

Cu



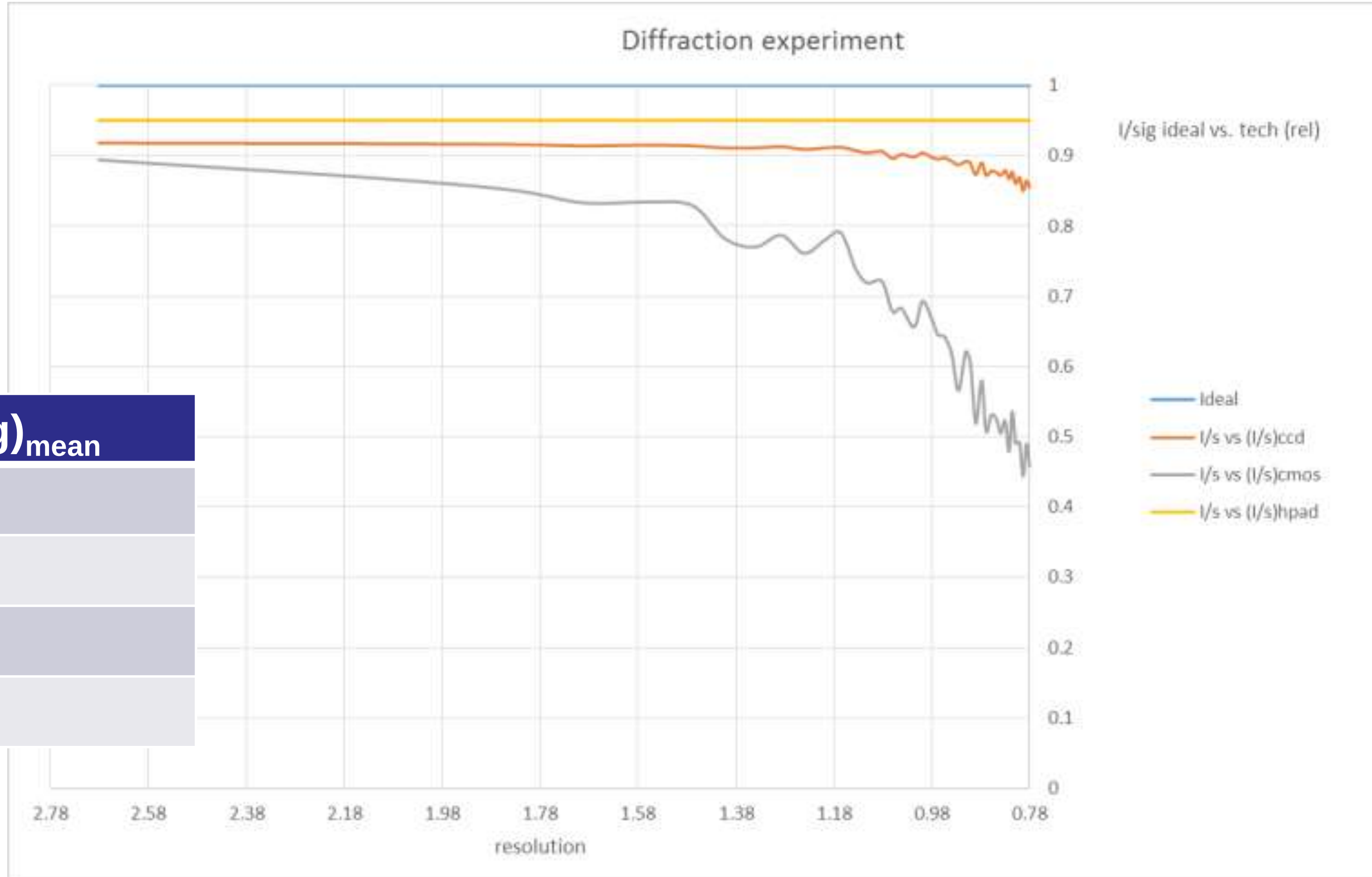
Tech	$(I/sig)_{mean}$
Ideal	15
CCD	13.6
CMOS	11.3
HPAD	14.2

Rigaku oxford diffraction



Importance of weak data

Comparison of tech



Tech	$(I/sig)_{mean}$
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CCD	13.6
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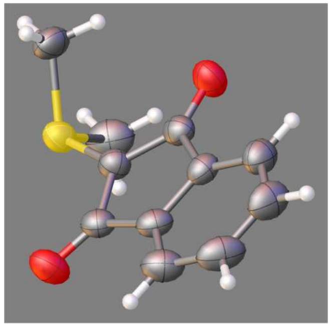
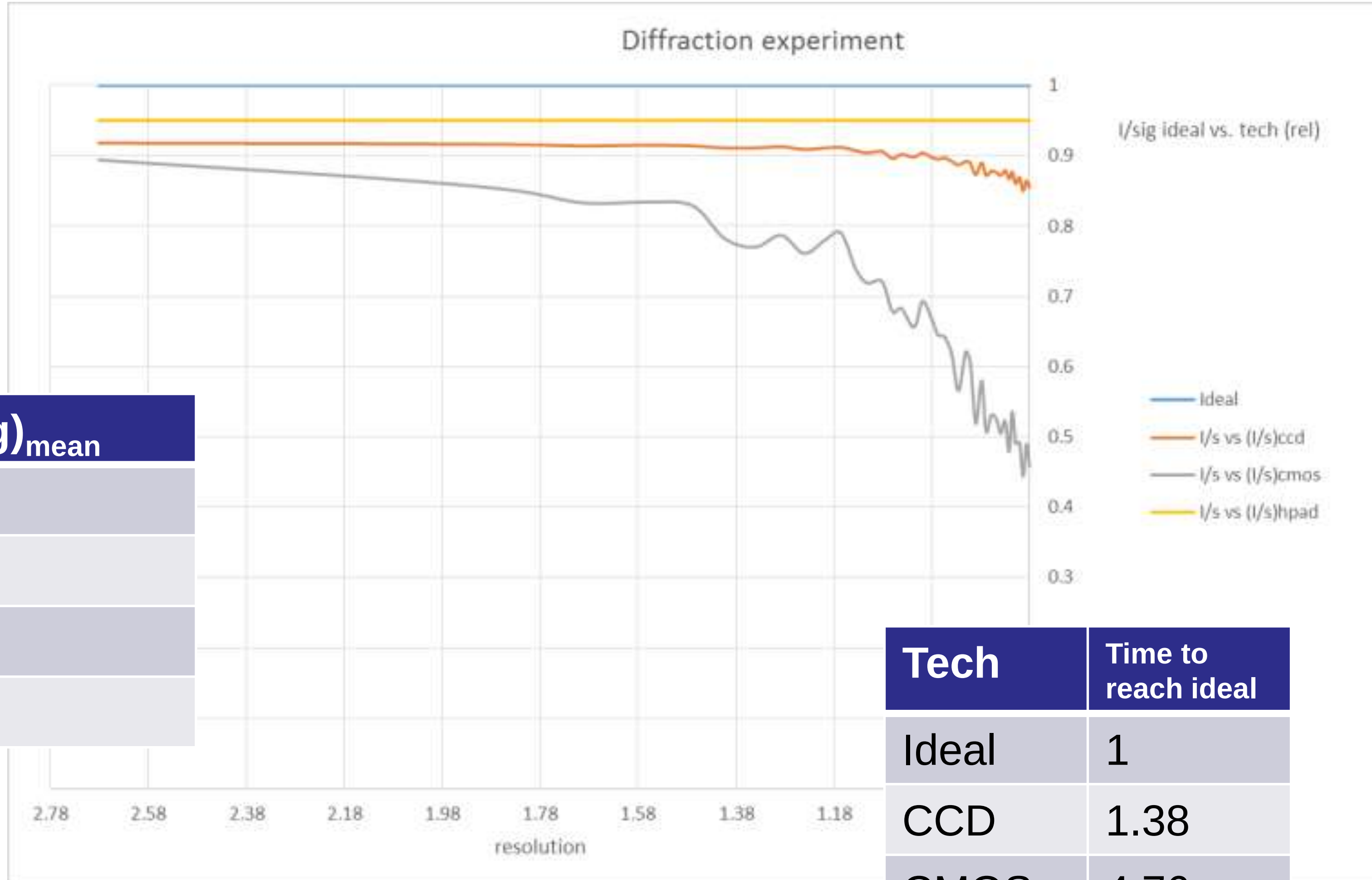
Rigaku oxford diffraction



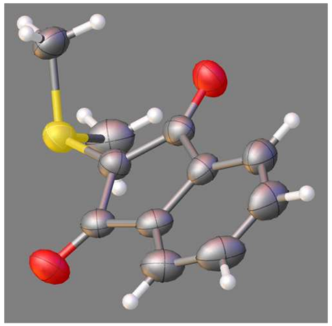
Importance of weak data

Comparison of tech



Tech	$(I/sig)_{mean}$
Ideal	15
CCD	13.6
CMOS	11.3
HPAD	14.2

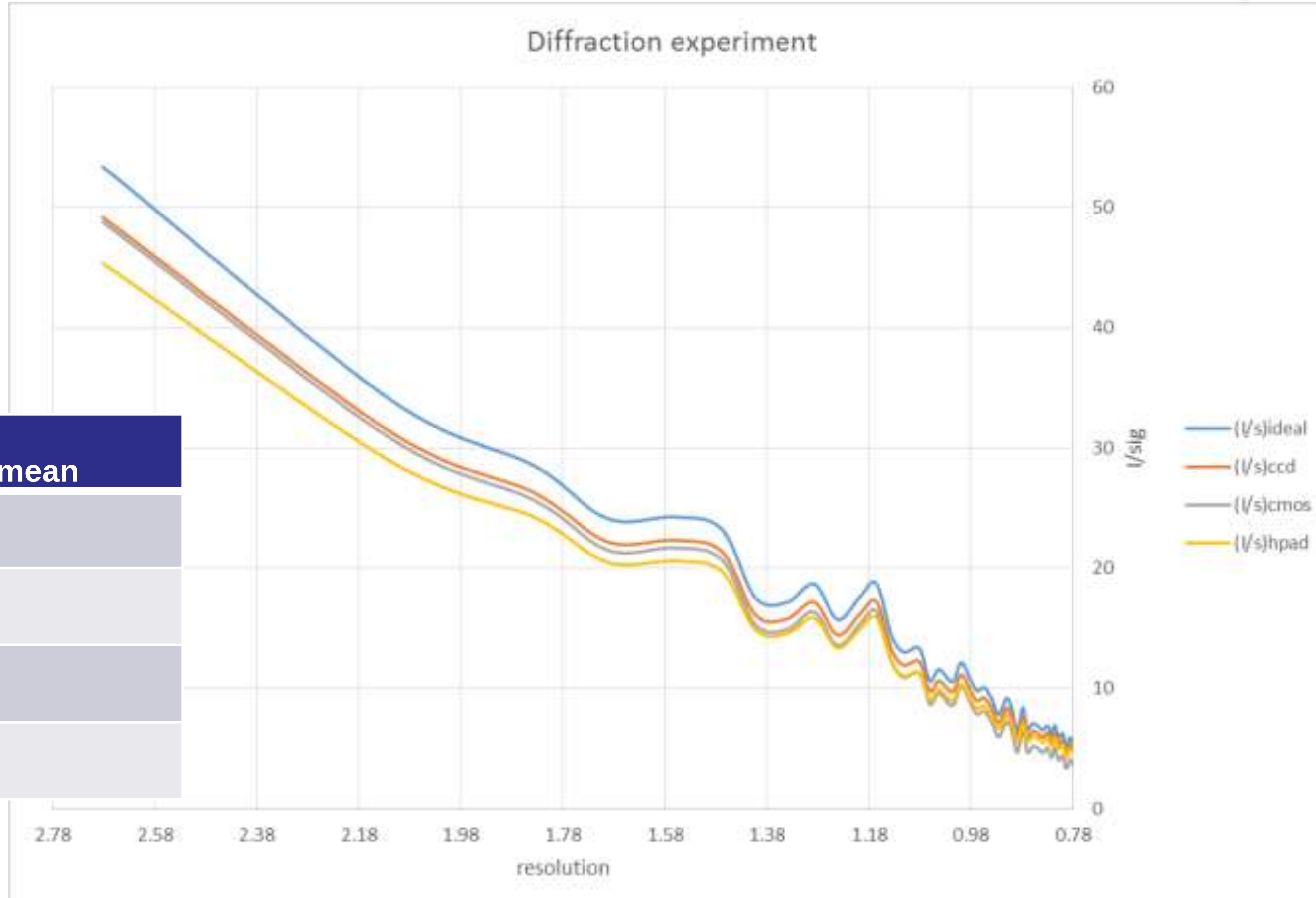
Tech	Time to reach ideal
Ideal	1
CCD	1.38
CMOS	4.76
HPAD	1.11



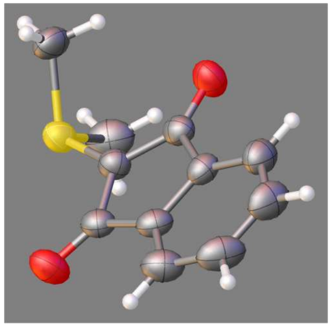
Importance of weak data

Comparison of tech

Mo



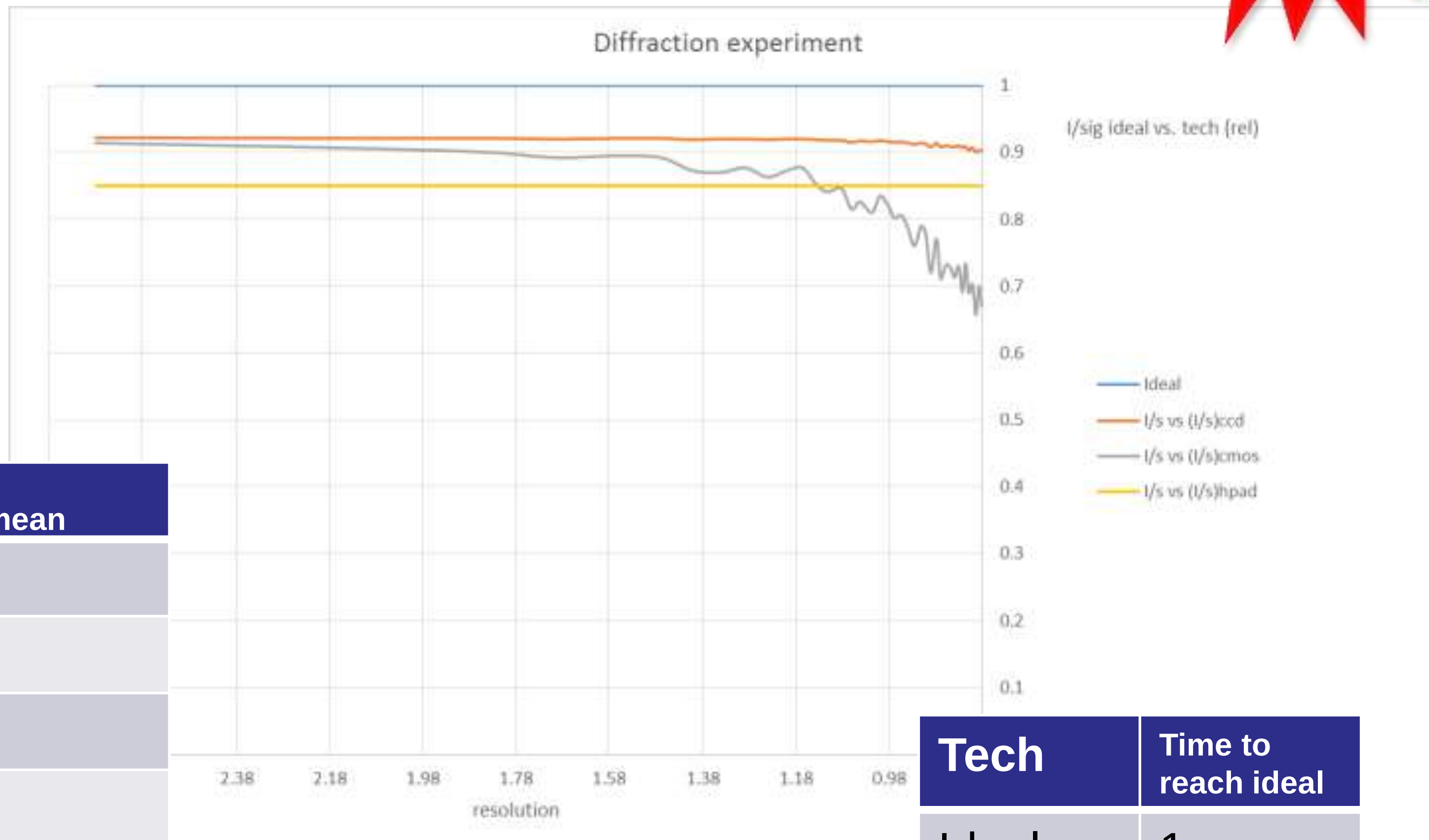
Tech	$(I/sig)_{mean}$
Ideal	15
CCD	13.8
CMOS	12.8
HPAD	12.7



Importance of weak data

Comparison of tech

Mo



Tech	$(I/sig)_{mean}$
Ideal	15
CCD	13.8
CMOS	12.8
HPAD	12.7

Tech	Time to reach ideal
Ideal	1
CCD	1.23
CMOS	2.22
HPAD	1.38

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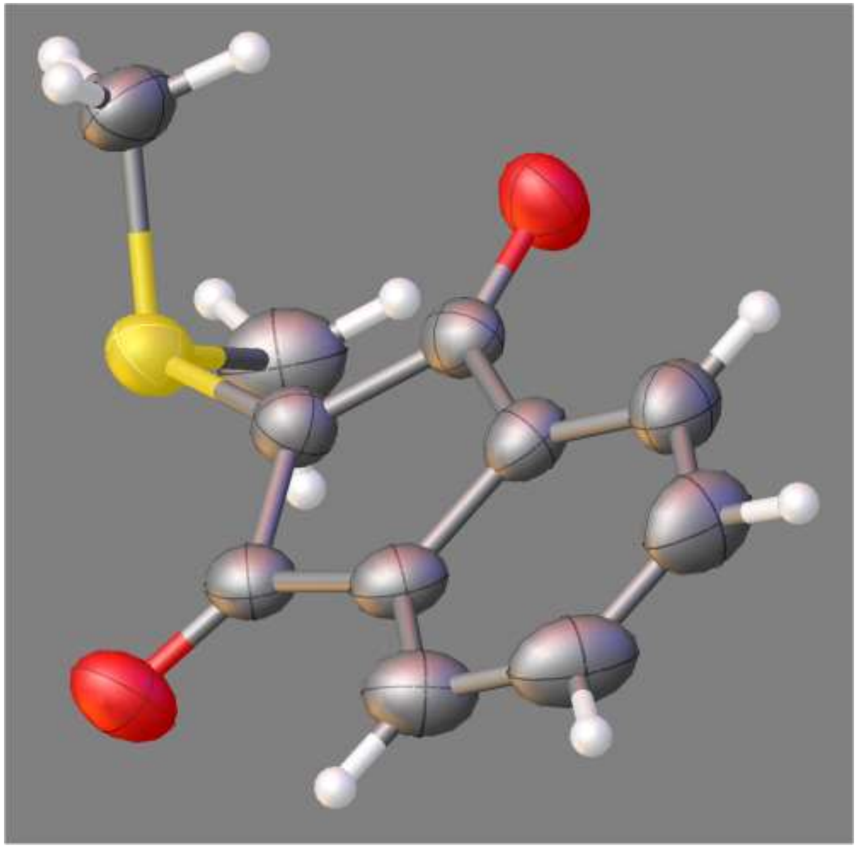
Importance of weak data

Charge density



- Make a typical experiment
  - Mo radiation
  - Resolution 0.45Ang
  - Diffraction limit set to 0.5Ang -> $(I/sig)_{mean}$ = 2
  - To get this we pump I:
  - $(I/sig)_{mean}$ = 35 to 0.837Ang (IUCR)

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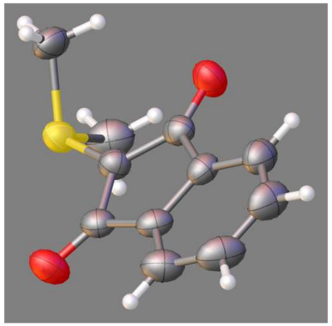


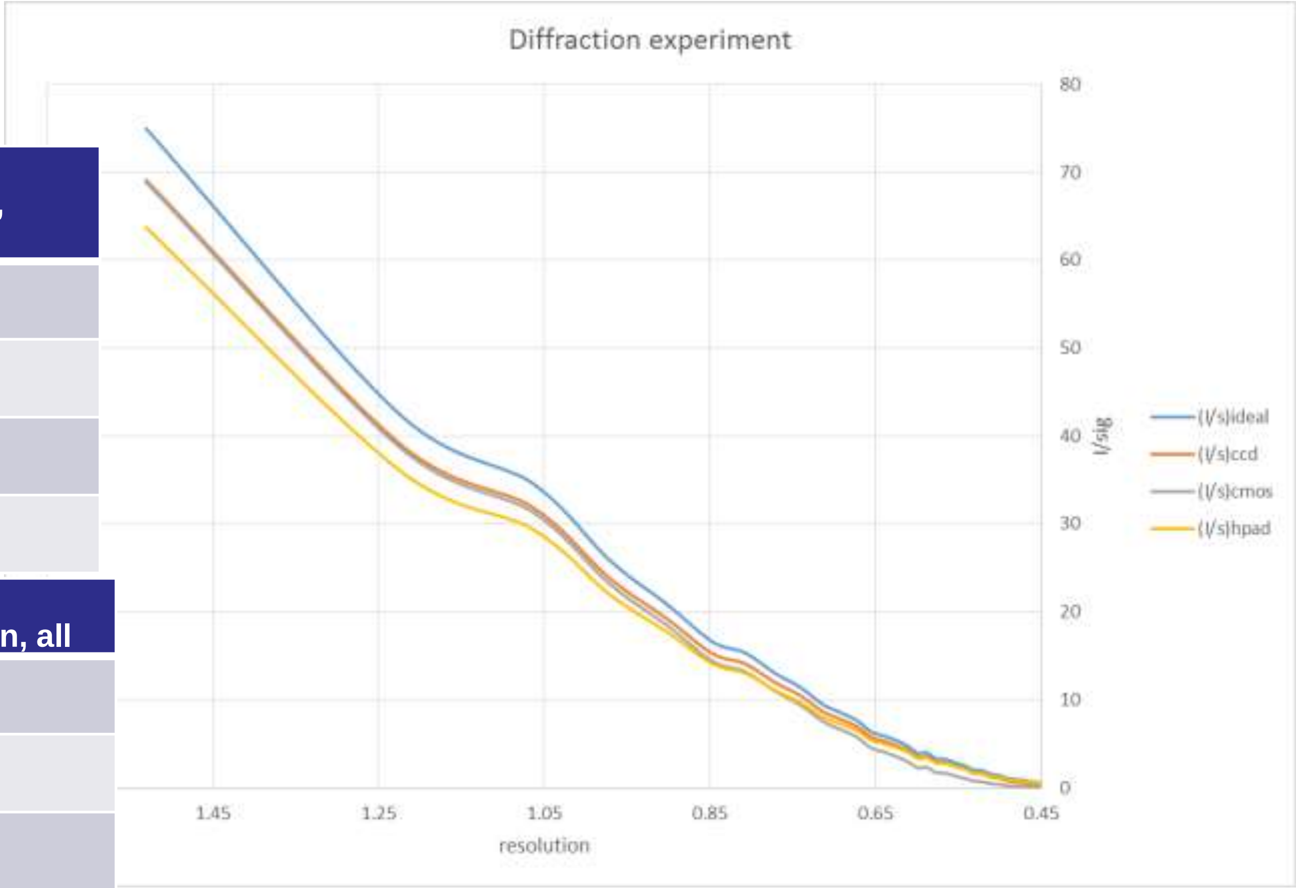
Importance of weak data

Comparison of tech

Mo

Tech	$(I/sig)_{mean, 0.837}$
Ideal	35.1
CCD	32.4
CMOS	31.8
HPAD	29.9

Tech	$(I/sig)_{mean, all}$
Ideal	8.6
CCD	7.8
CMOS	7.0
HPAD	7.3



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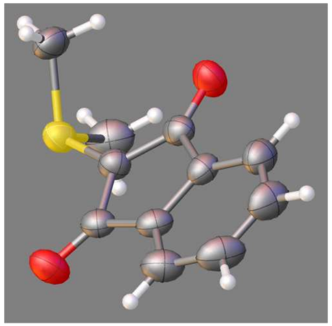

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Comparison of tech

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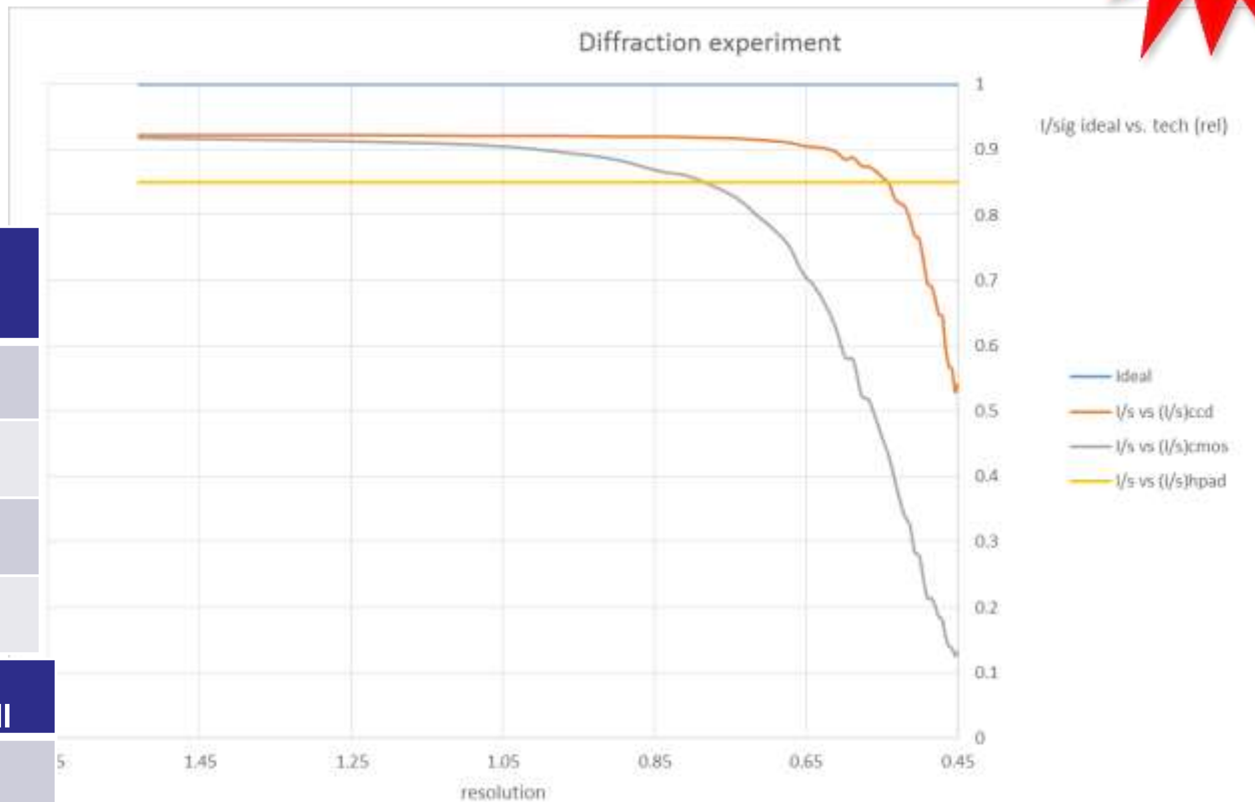

Tech	$(I/sig)_{mean, 0.837}$
Ideal	35.1
CCD	32.4
CMOS	31.8
HPAD	29.9

Tech	$(I/sig)_{mean, all}$
Ideal	8.6
CCD	7.8
CMOS	7.0
HPAD	7.3

Tech	Time to reach ideal
Ideal	1
CCD	3.42
CMOS	59.17
HPAD	1.38

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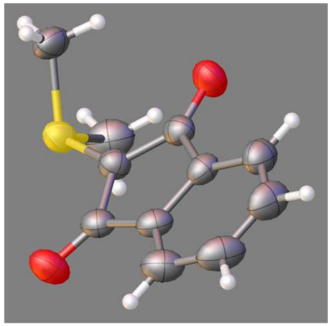

Importance of weak data

Conclusion

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Atlas S2 CCD vs. APS CMOS

Comparative Tests

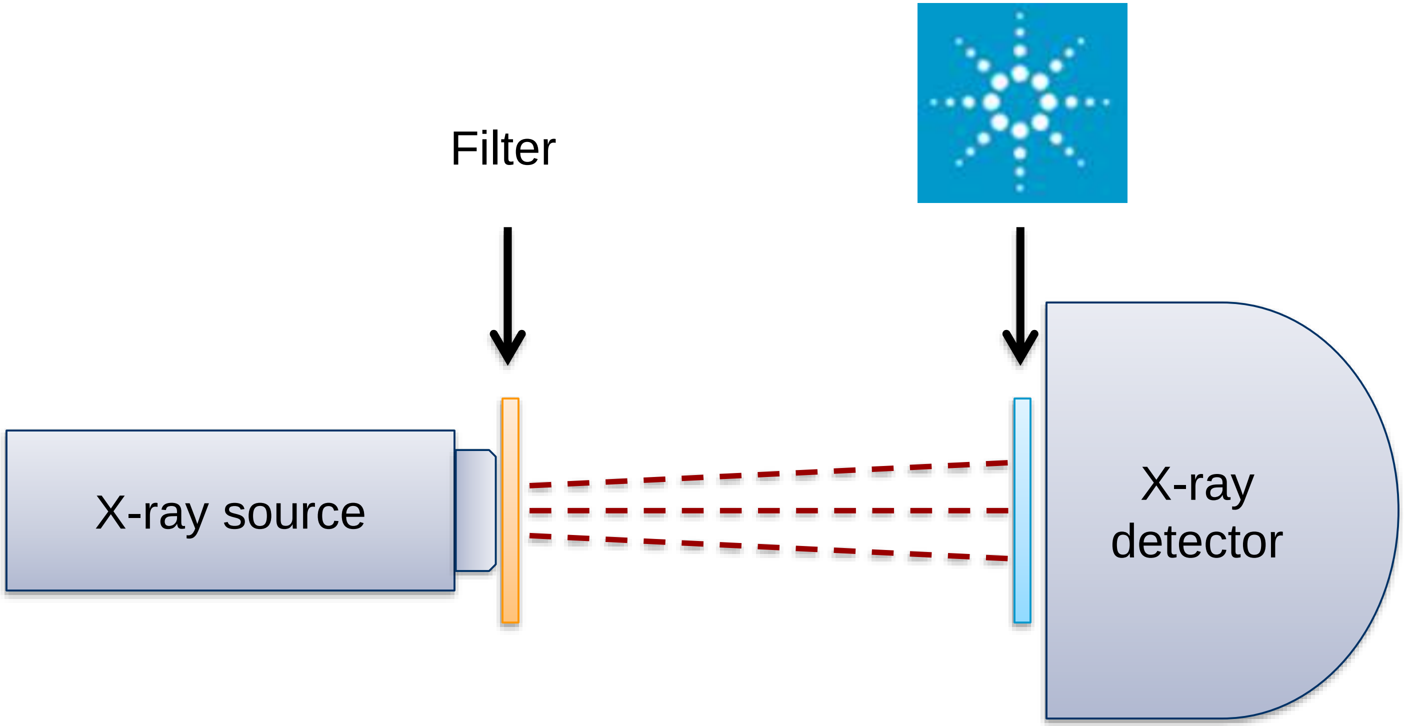
Oxford Diffraction R&D have designed, built and tested a CMOS detector of identical internal construction to a commercially available model

Feature	Atlas S2 CCD	APS CMOS (Oxford Diffraction R&D)
Active area, Taper	**100x100mm, taper 2:1**	**100x100mm, taper 1:1**
Gain [e^-/MoKα]	**180**	**261**
Sensor	**Truesense* Imaging CCD**	**Teledyne Dalsa RadEye 100 CMOS**
Noise [MoKα-photons]	**~0.05**	**~0.5**

*Formerly Kodak

CCD vs. APS CMOS

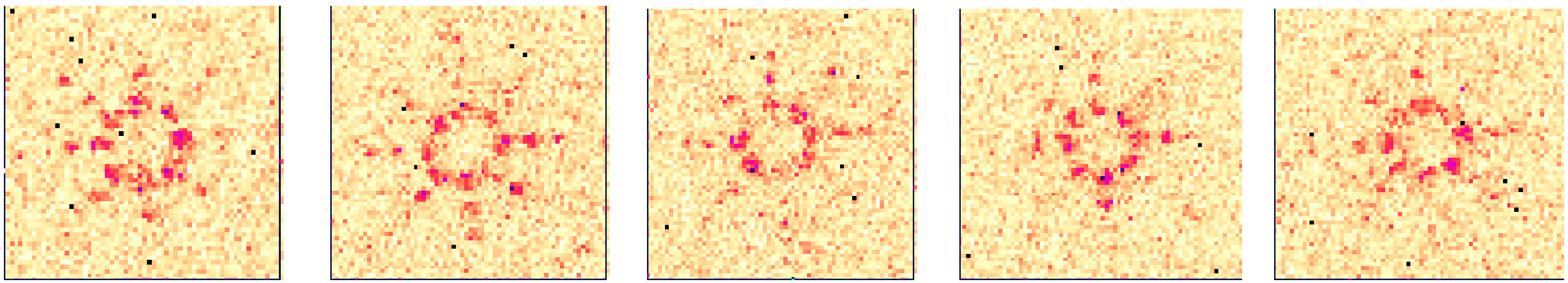
Comparative Detectivity Measurements



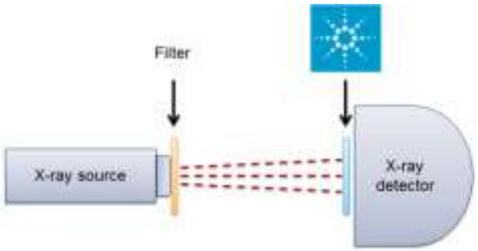
CCD vs. APS CMOS

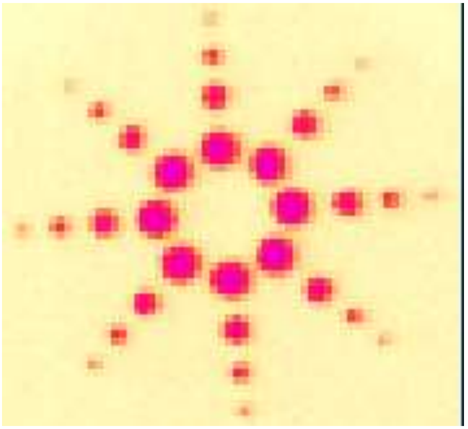
Comparative Detectivity Measurements

- The filter has been chosen in such a way as to observe single photon events



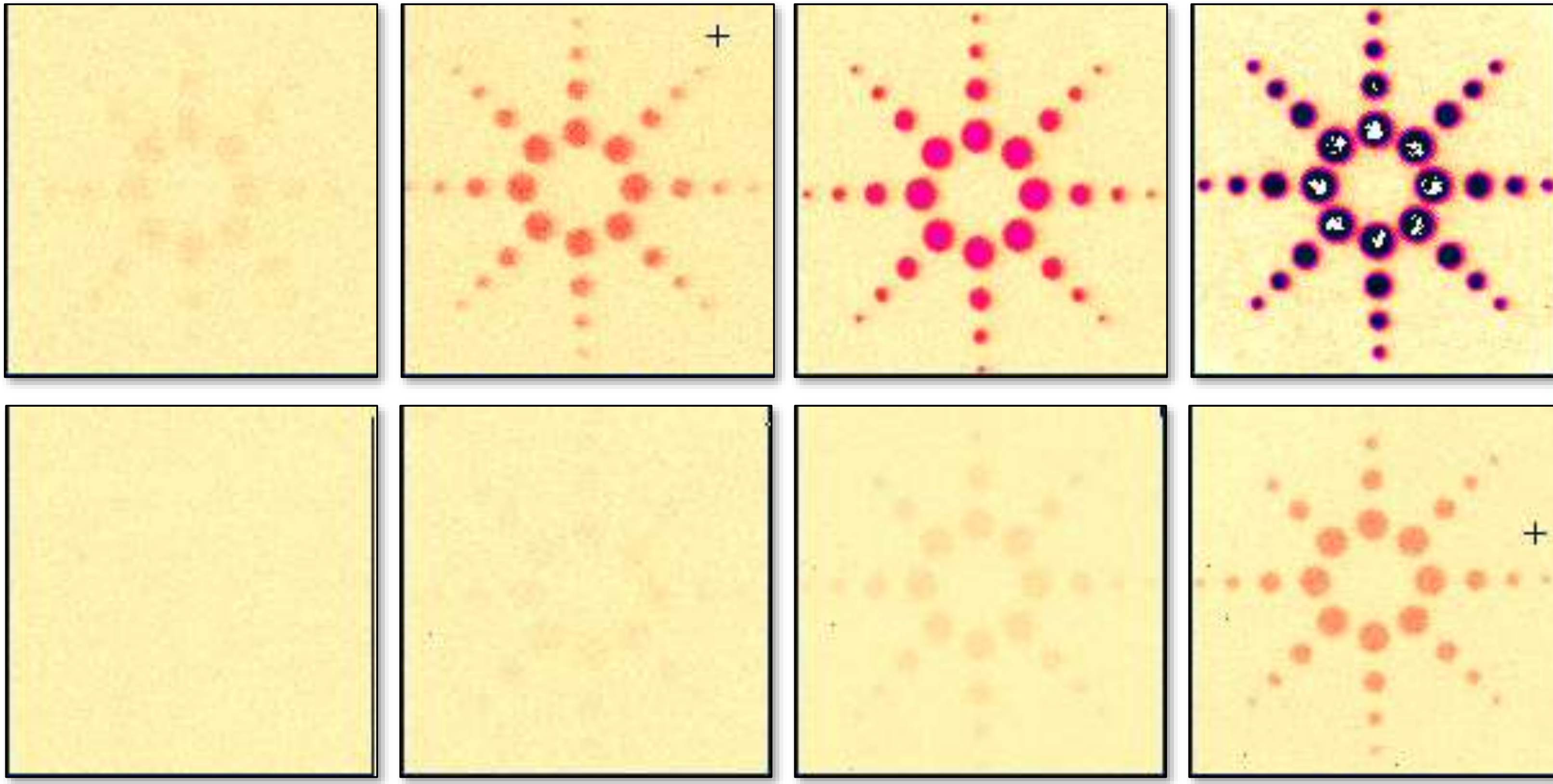
- In order to visualize signal-to-noise differences 100 images are averaged and scaled so that the noise level is the same for all modes of operation





CCD vs. APS CMOS

Comparative Detectivity Measurements



Atlas CCD

APS CMOS (RadEye100 Chip – Oxford Diffraction R&D)

Exposure time [s]: 0.1 | 0.5 | 1 | 5

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HW – Detector technology: Key metrics

- Detectivity
- Dynamic range
- Speed
- Size
- Price

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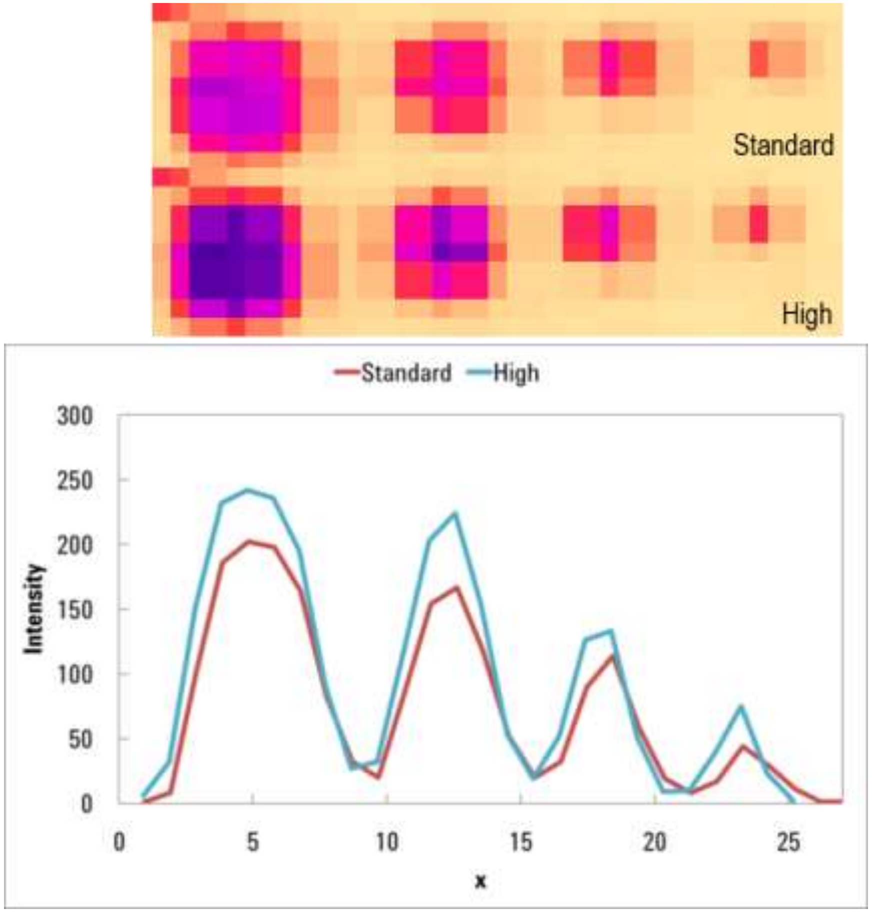
S2 CCD Detectors:
INTELLIGENT MEASUREMENT SYSTEM

Smart Sensitivity Control (SSC)

- **Self-optimizing** detector amplification based on strength of observed data (similar to ISO settings in digital photography)
- Standard, Medium and High SSC modes
- Maximises dynamic range for strong data
- Improvement in signal-to-noise for weak data
- A **unique** feature of Rigaku Oxford Diffractions CCD X-ray detectors



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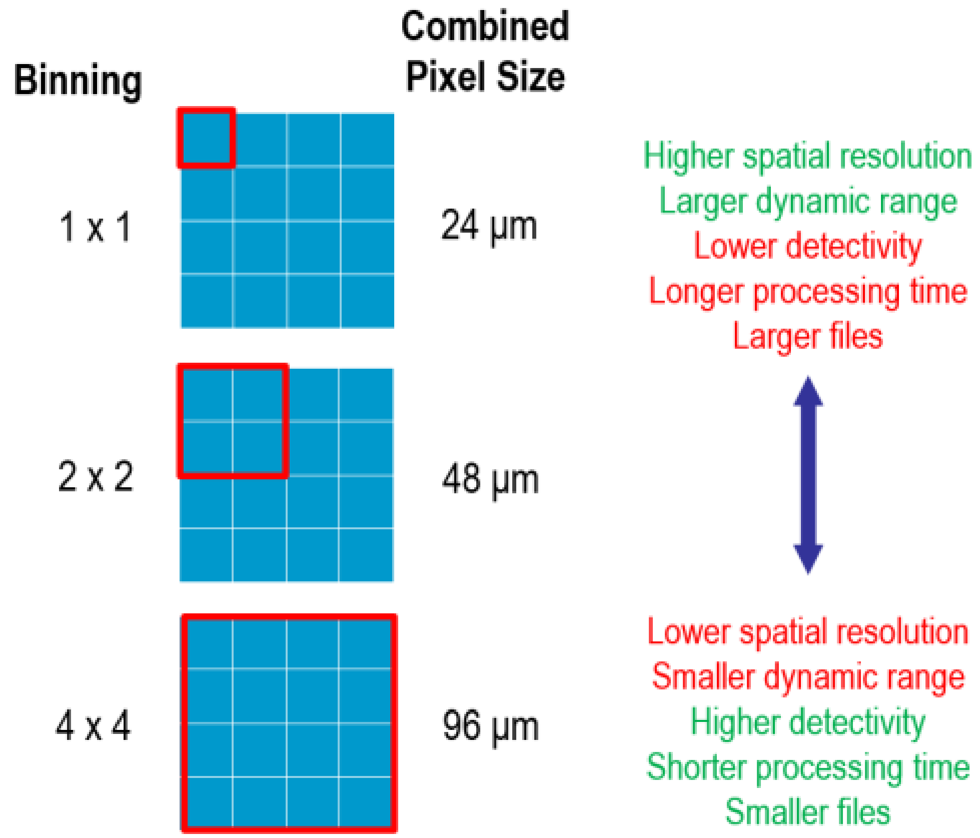
S2 CCD Detectors:
INTELLIGENT MEASUREMENT SYSTEM

Instant-switching hardware binning:

- **Adjustable** pixel sizes for variable resolution
- Flexibility in dynamic range
- Fast re-measurement of overflowed reflections
- Theta-dependent binning
- Automatic software switches binning modes **instantly**



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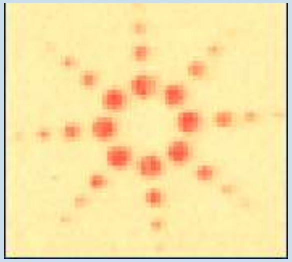
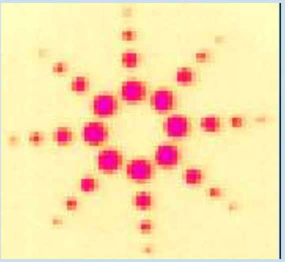
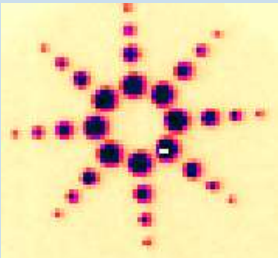
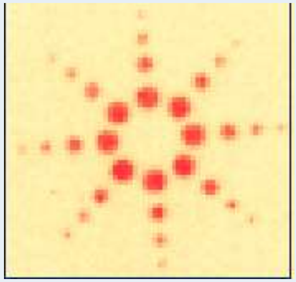
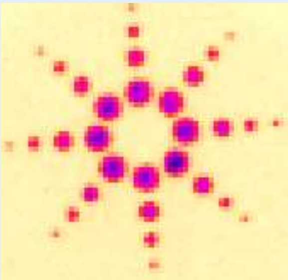
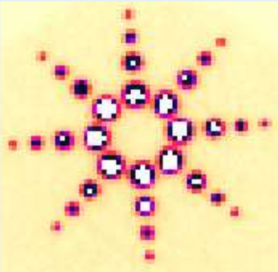
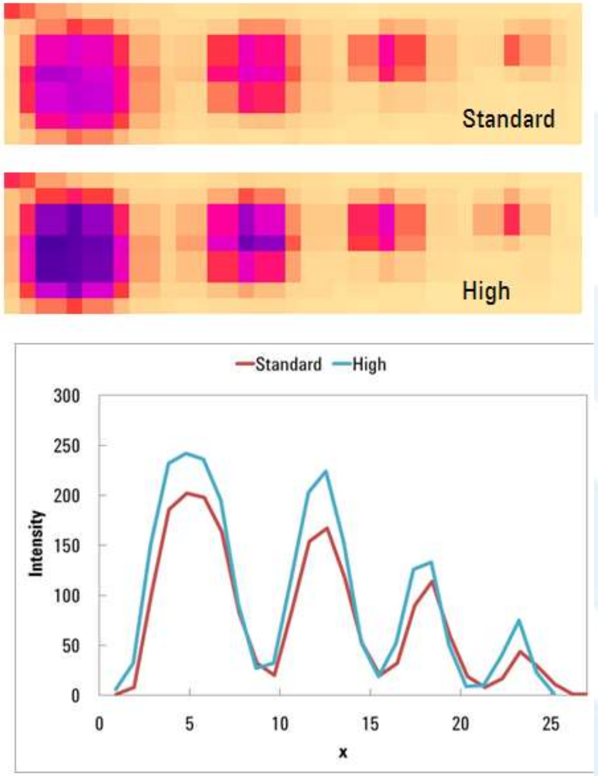
S2 CCD Detectors:
INTELLIGENT MEASUREMENT SYSTEM

Exposure time	0.5s	1s	2s
Standard SSC mode			
High SSC mode			



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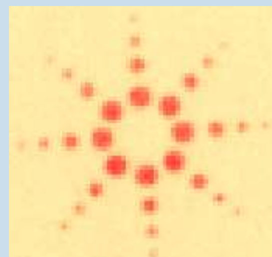
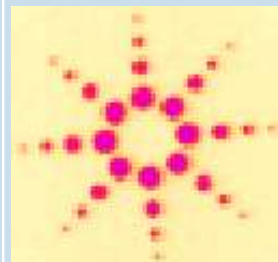
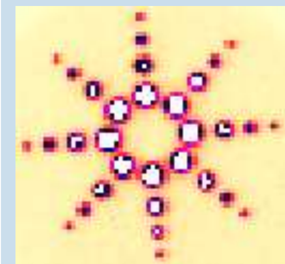
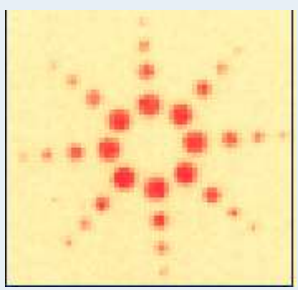
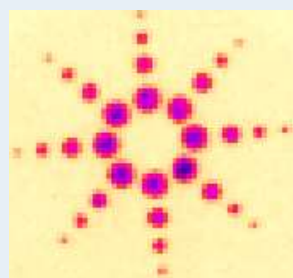
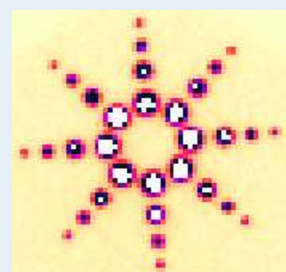
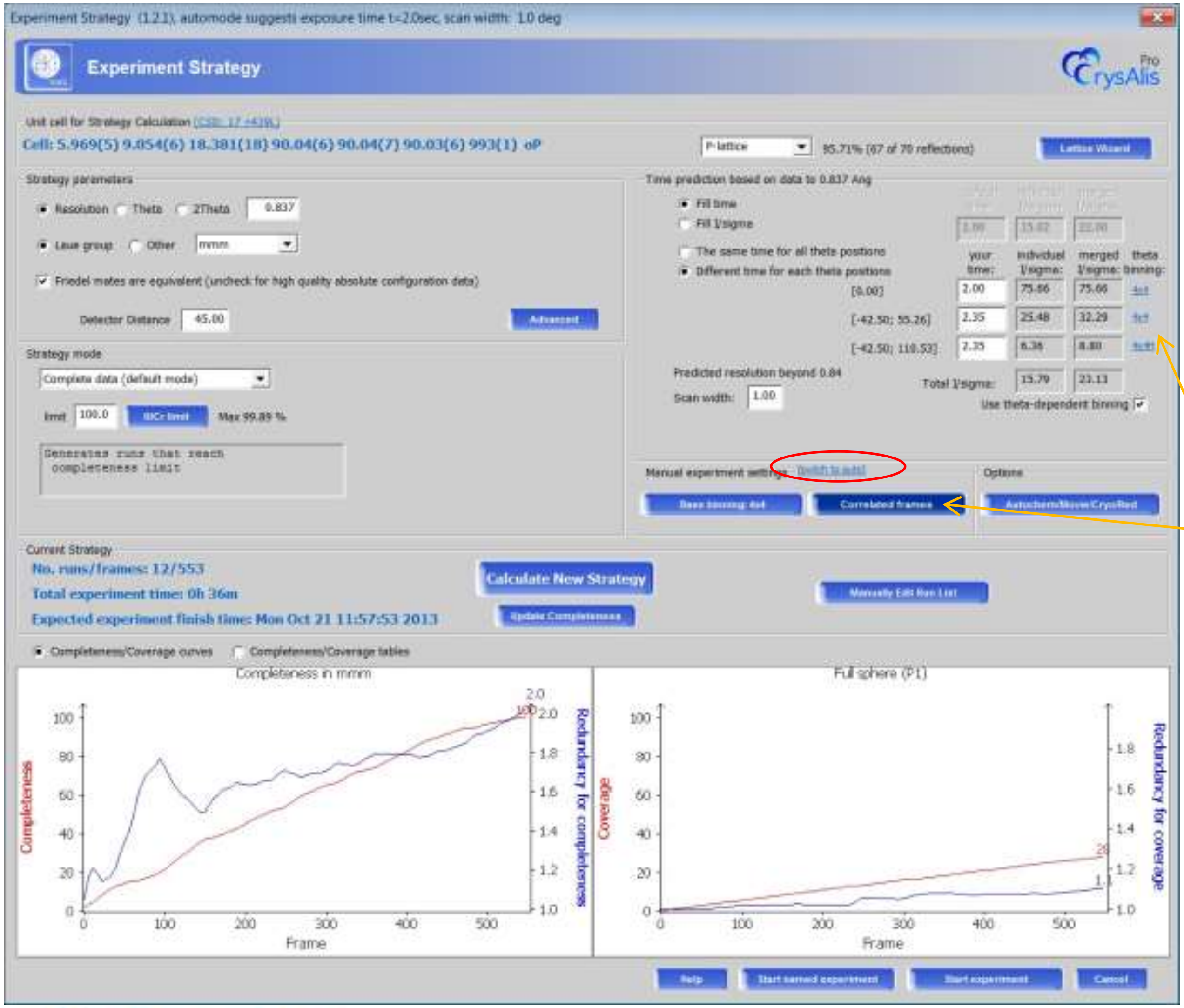
S2 CCD Detectors:
INTELLIGENT MEASUREMENT SYSTEM

Exposure time	0.5s	1s	2s	5s
2x2				
4x4				

S2 CCD Detectors:

INTELLIGENT MEASUREMENT SYSTEM: IMS



If the user modifies one of the suggested settings, 'Auto suggest' link appears, which allows him to re-enable automatic suggestions

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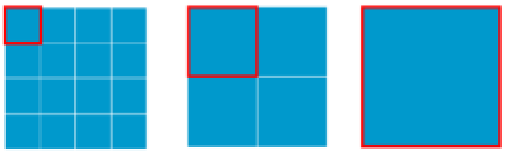
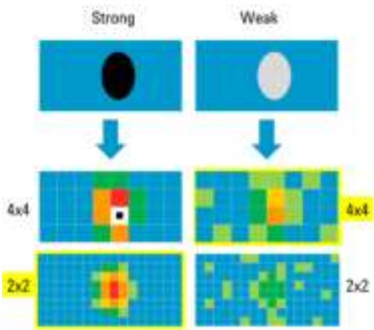
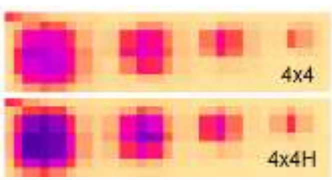
Strategy

Intelligent Measurement System – IMS for CCD



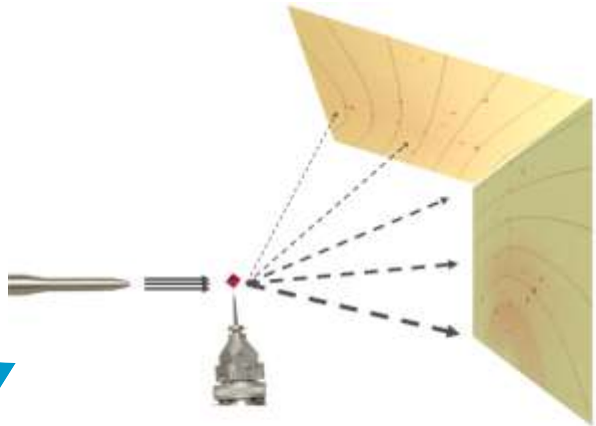
Base binning





Smart sensitivity control

IMS



Theta dependent binning

Gonio/scan settings correlation

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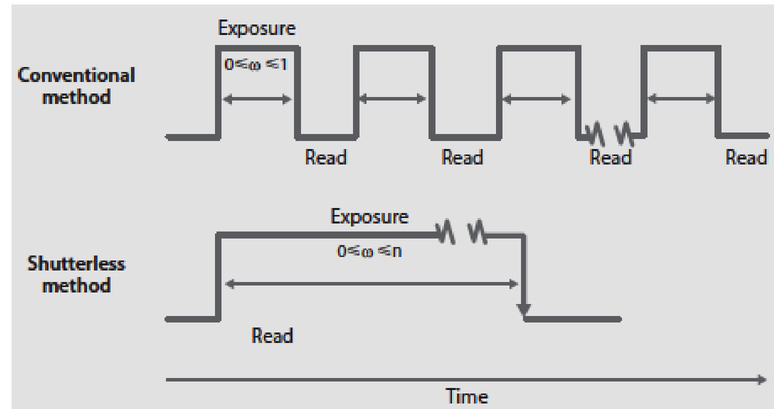
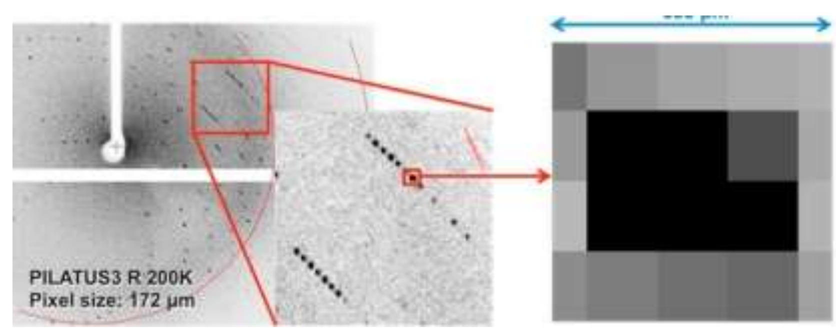
HPC – Hybrid Pixel Counters:

- HPC detectors deliver excellent data quality due to high dynamic range and superb signal-to-noise
  - No rescans required to correct for overloads or to measure strong data
- Signal threshold reduces noise from fluorescence
- Shutterless data collection
  - Simplifies measurement setup
  - Improves data quality
  - Can dramatically shorten wall time
- Top-hat point spread function means better spatial resolution for reflections



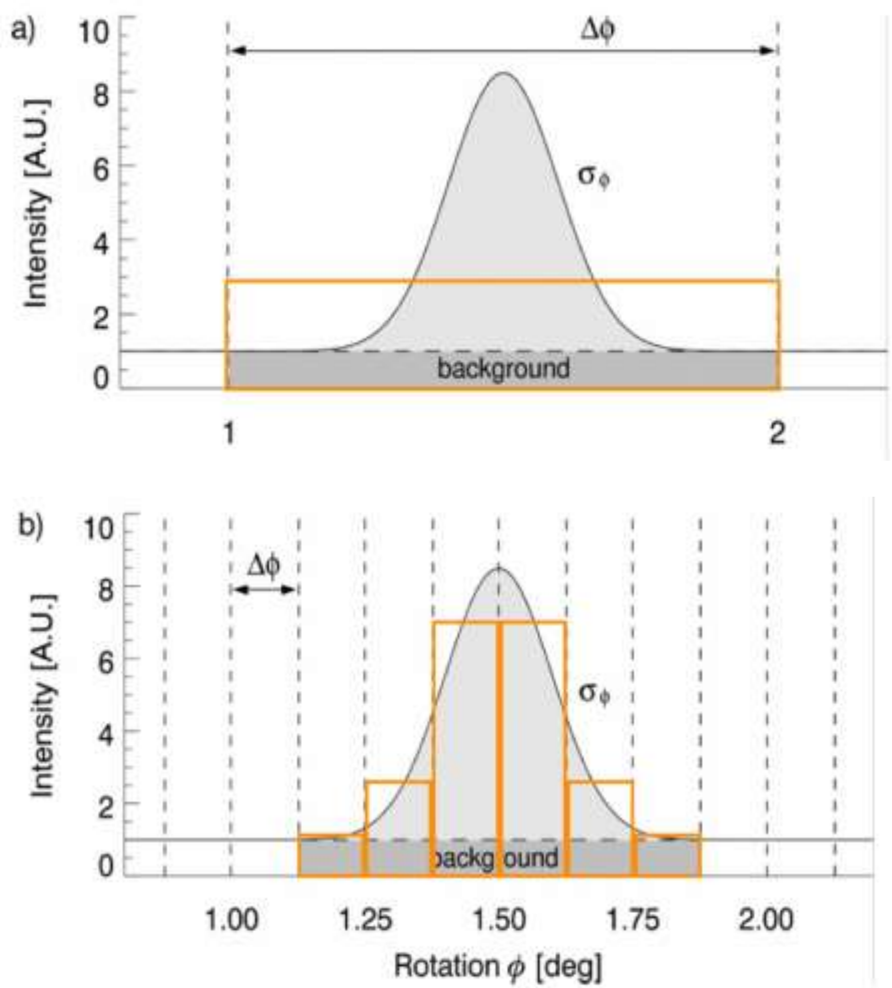
Shutterless data collection



Single pixel point spread function

Fine slicing and count rate correction

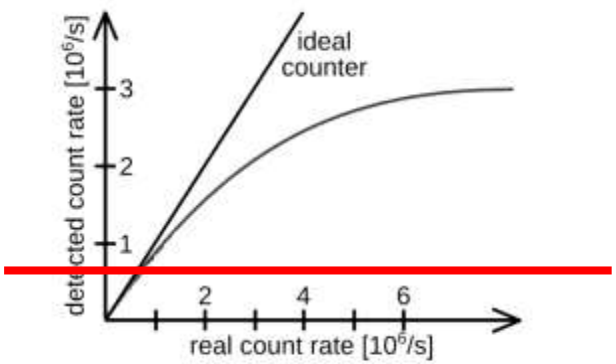
Wide slicing



Fine slicing

HPADs are rate meters!
To integrate they require rate correction!
Simple rate correction requires near constant signal

Rate = counts/time



Mueller et al., Acta Cryst. (2012). D68, 42-56:
Optimal fine phi-slicing for single-photon-counting pixel detectors

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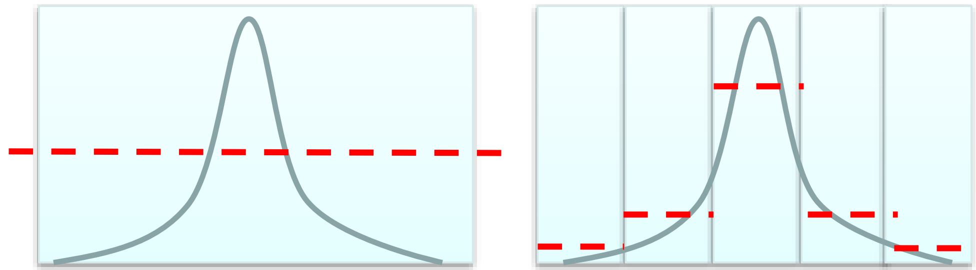
HPC – IMS: Feature

New 39

Feature:

- Strong reflections may be affected by coincidence-loss (dead time correction): rates > 400k/pix.s
- Fine-slicing may be required for more accurate count-rate correction



- Excessive fine slicing may yield photon loss due to (even) short dead or readout time
- In shutterless mode no re-measurement possible
- Pixels exceeding count-rate or absolute counter limit will be treated as overflows

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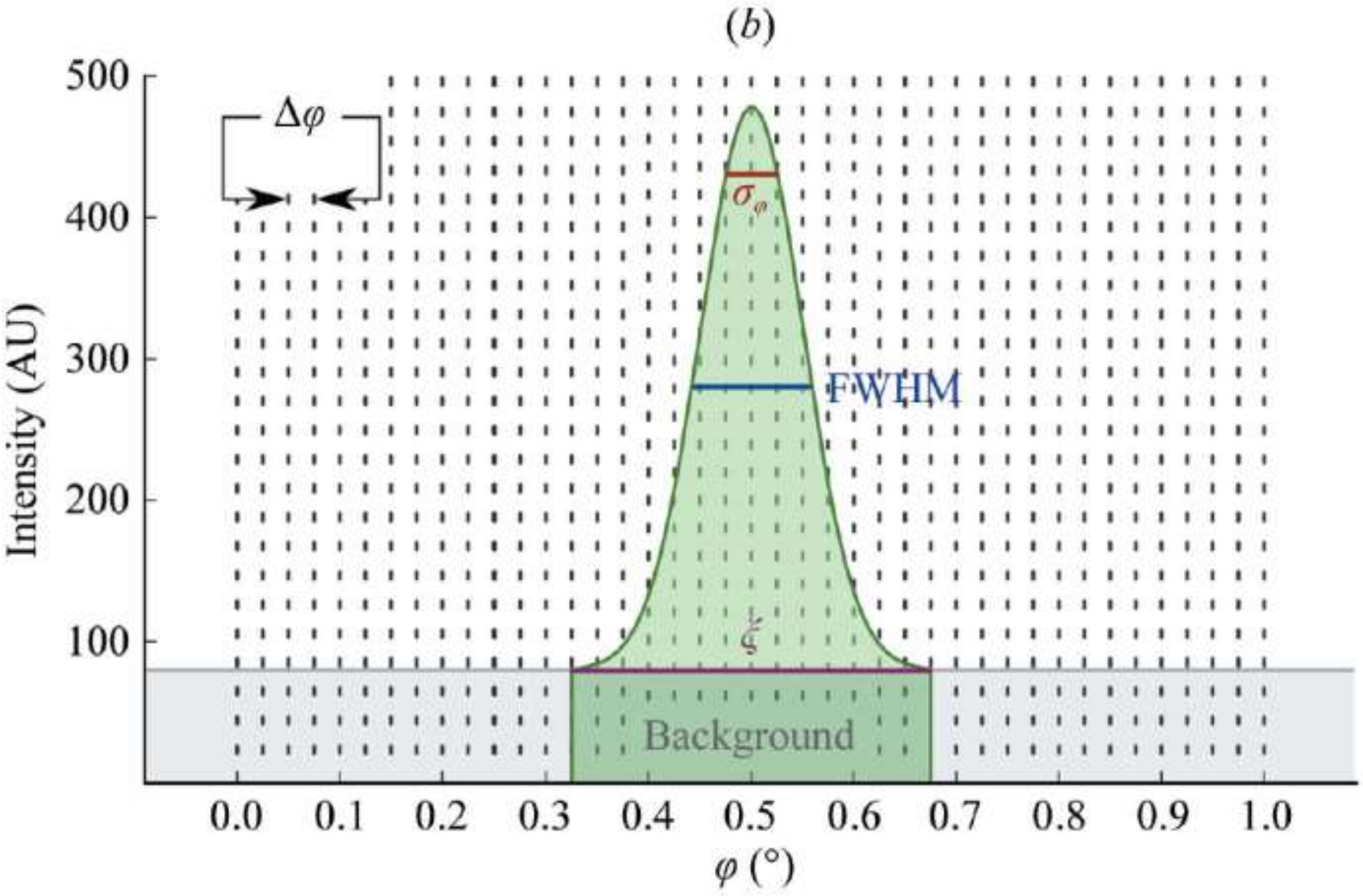
Data quality and count rates

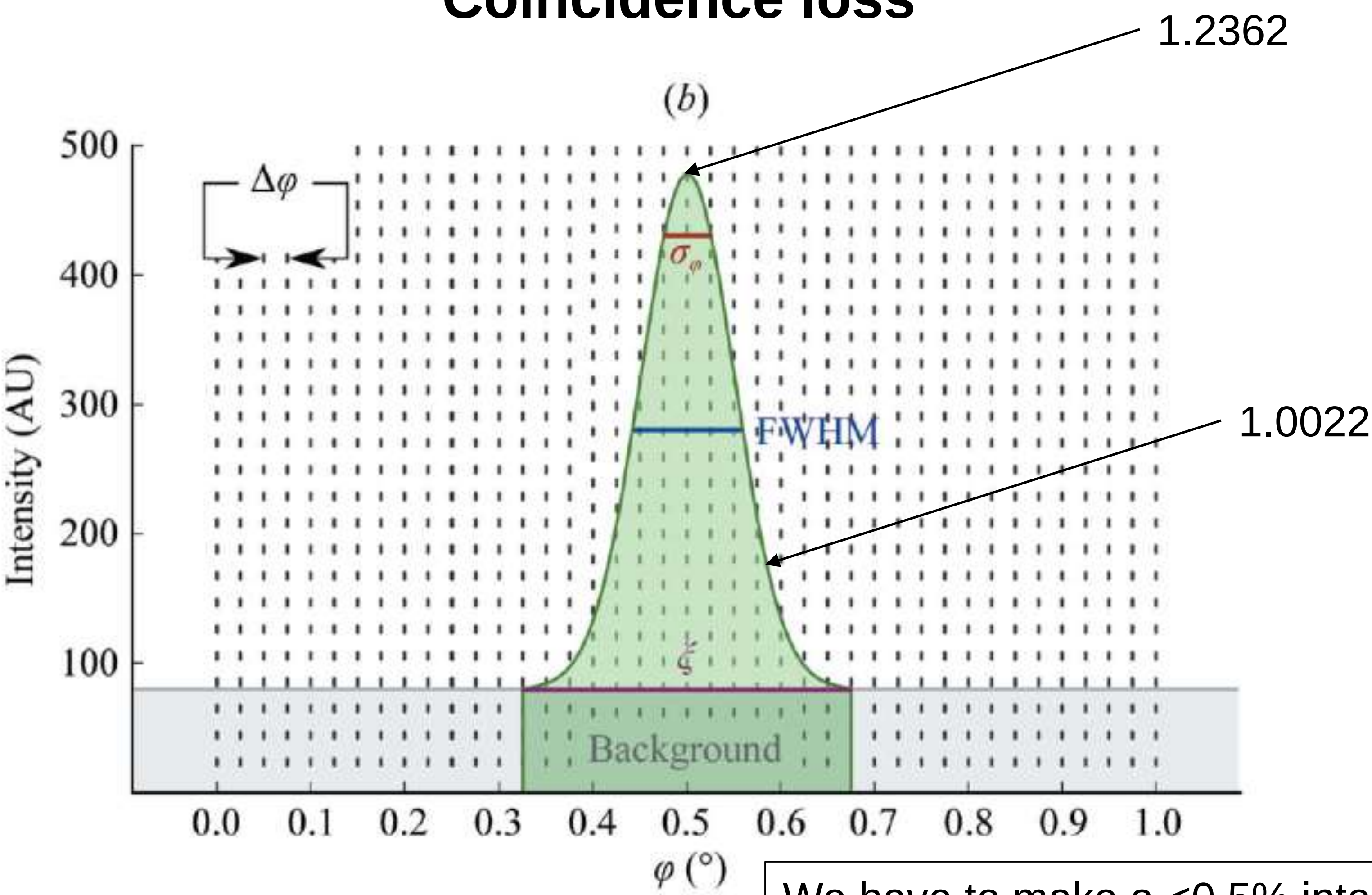
Coincidence loss



Mueller et al., Acta Cryst. (2012). D68, 42–56:
Optimal fine phi-slicing for single-photon-counting pixel detectors

Data quality and count rates

Coincidence loss

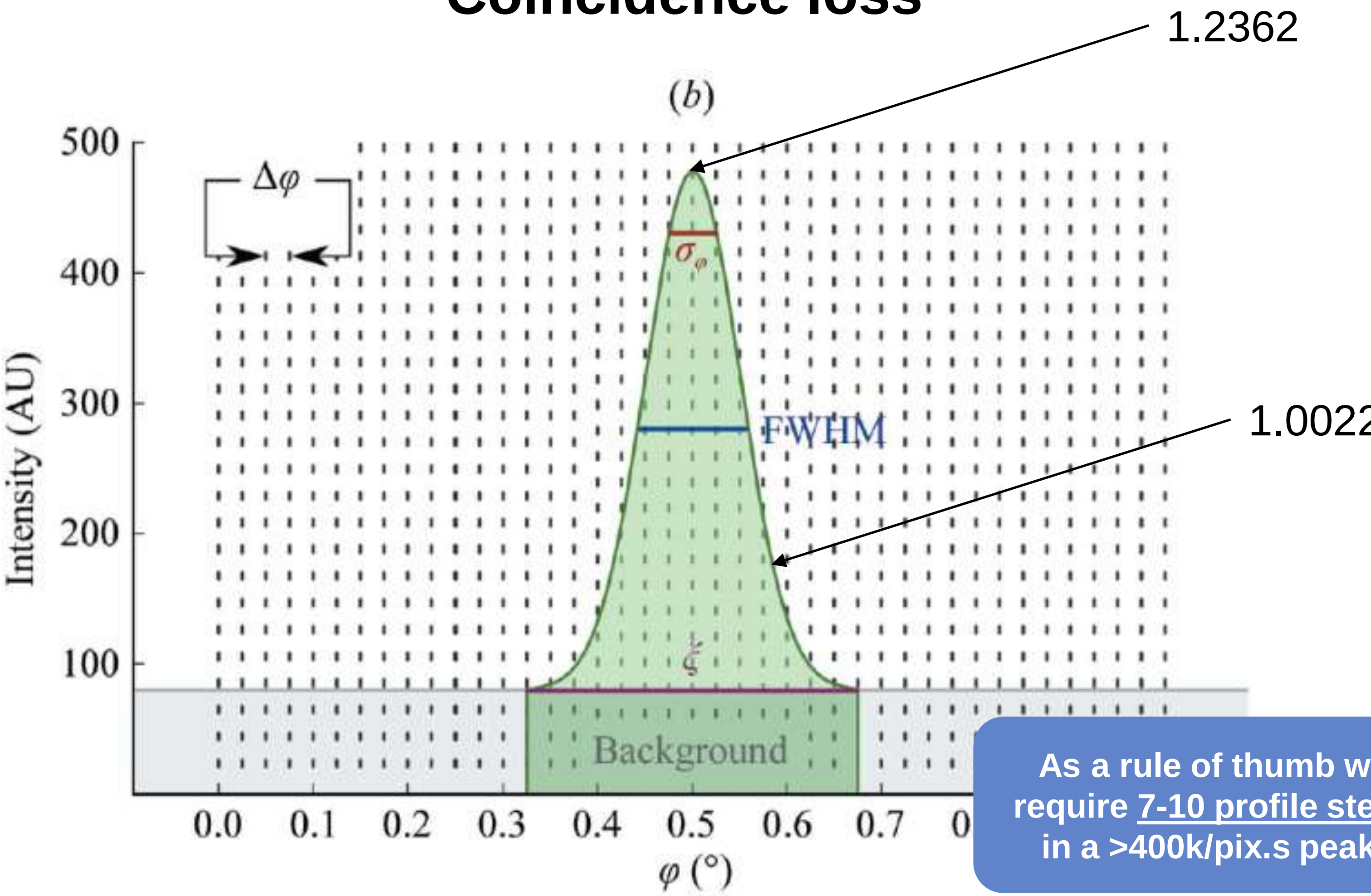


We have to make a <0.5% integral!

Mueller et al., Acta Cryst. (2012). D68, 42–56:
Optimal fine phi-slicing for single-photon-counting pixel detectors

Data quality and count rates

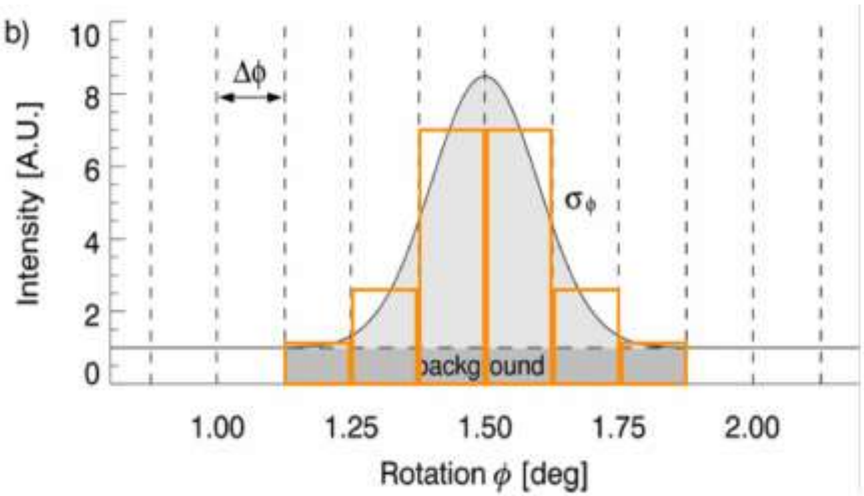
Coincidence loss



As a rule of thumb we require <u>7-10 profile steps</u> in a >400k/pix.s peak!

Mueller et al., Acta Cryst. (2012). D68, 42–56:
Optimal fine phi-slicing for single-photon-counting pixel detectors

Fine slicing and count rate correction

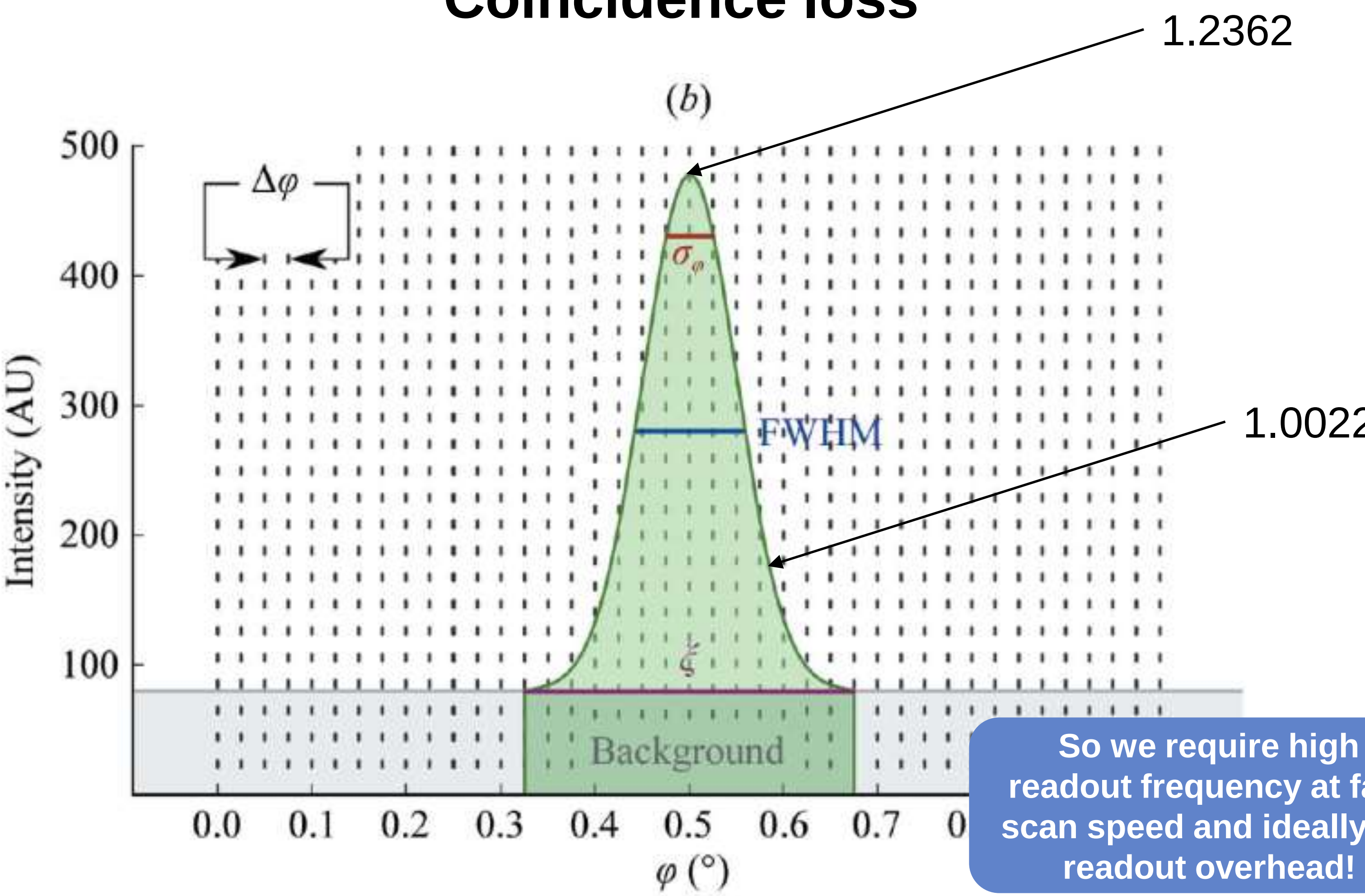


Rate = counts/time

Important feature:
No matter what scan speed we use the local angular rates stay!

Data quality and count rates

Coincidence loss

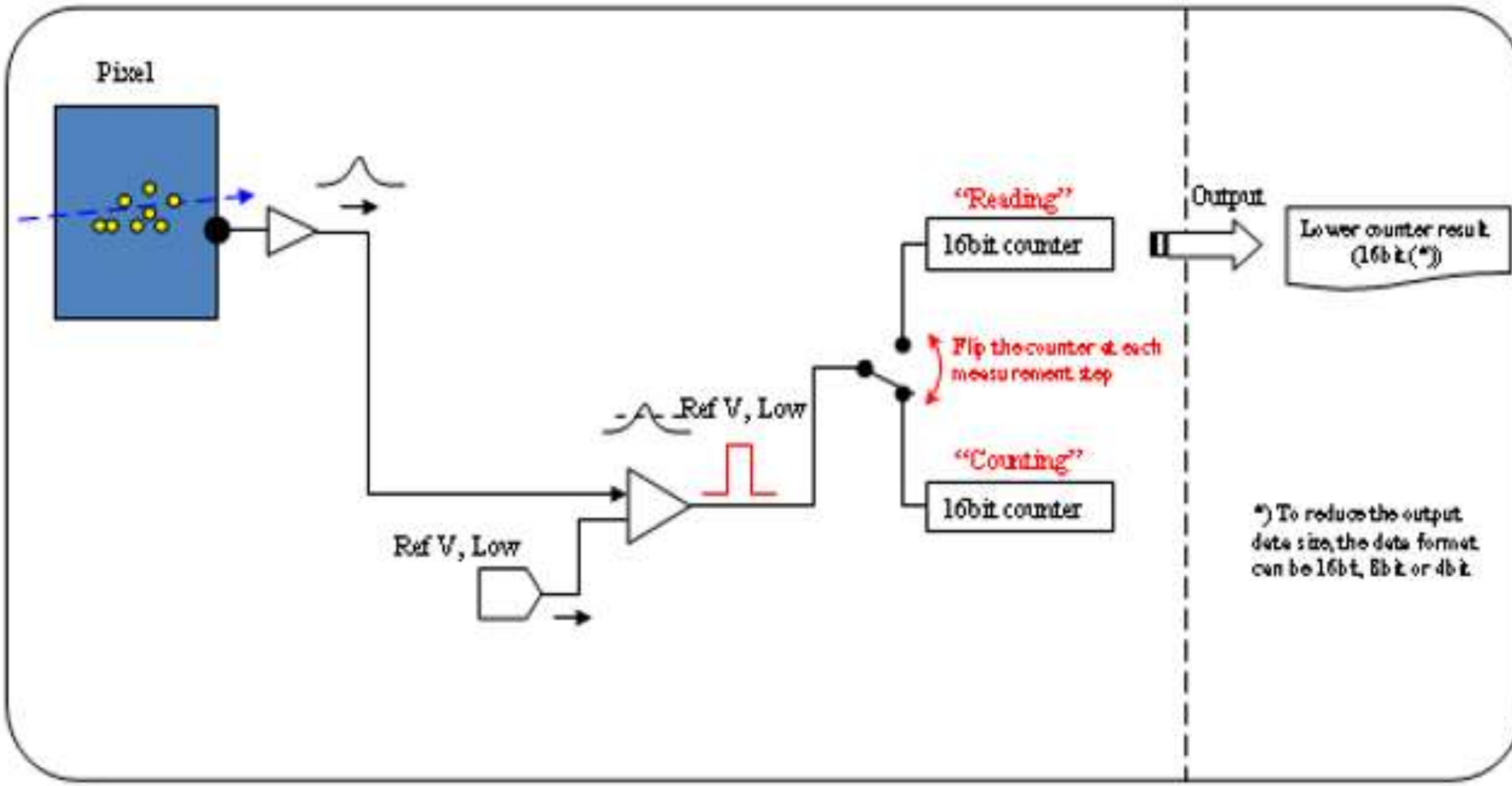


So we require high readout frequency at fast scan speed and ideally no readout overhead!

Mueller et al., Acta Cryst. (2012). D68, 42–56: Optimal fine phi-slicing for single-photon-counting pixel detectors

New Hybrid Photon Counting Detector

HyPix6000HE – (near) Zero Dead-Time Mode+100Hz



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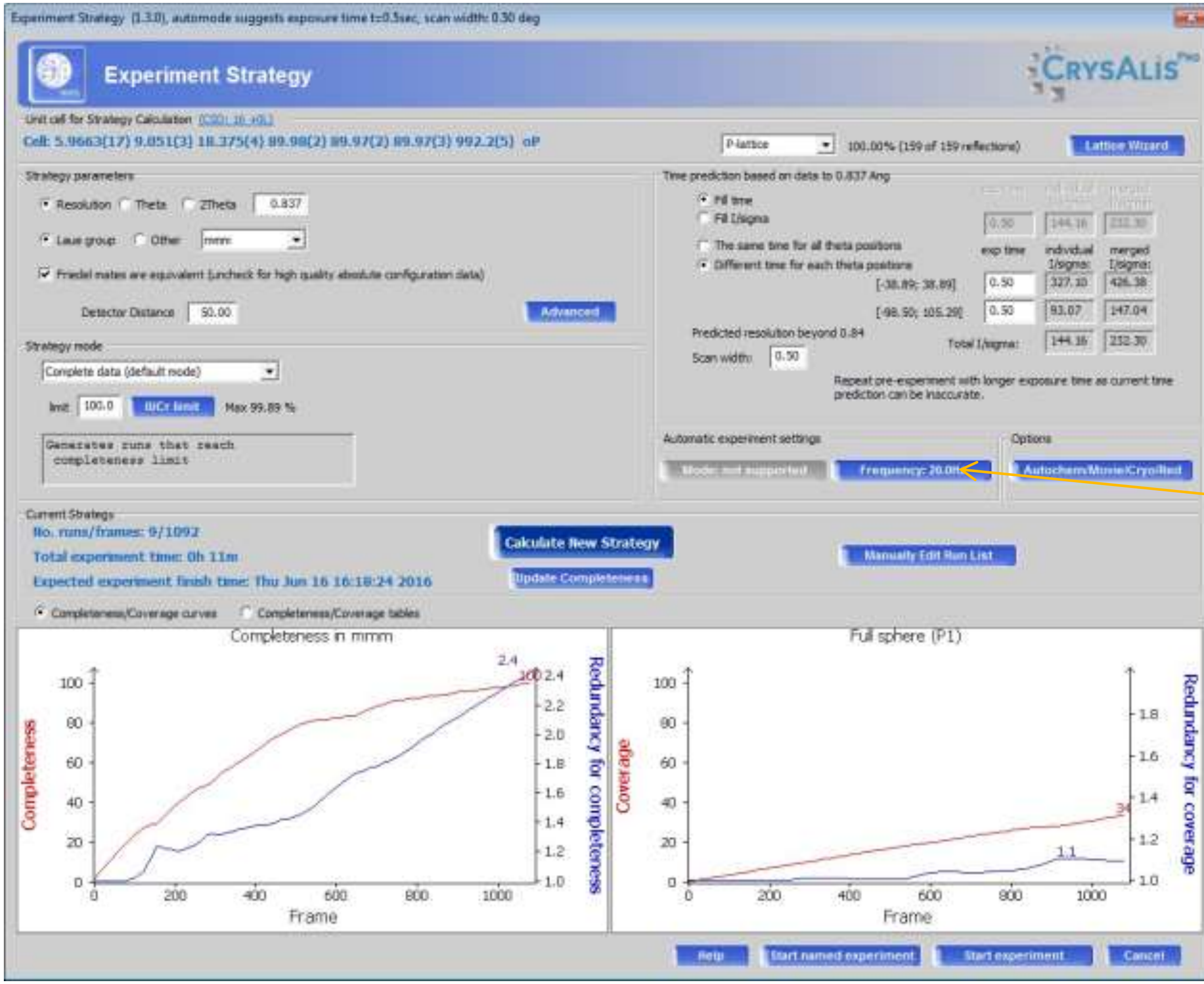
HPC – Hybrid Pixel Counters: IMS

New 39



The optimal data collection frequency is suggested from the pre-experiment evaluation and the user selected exposure time

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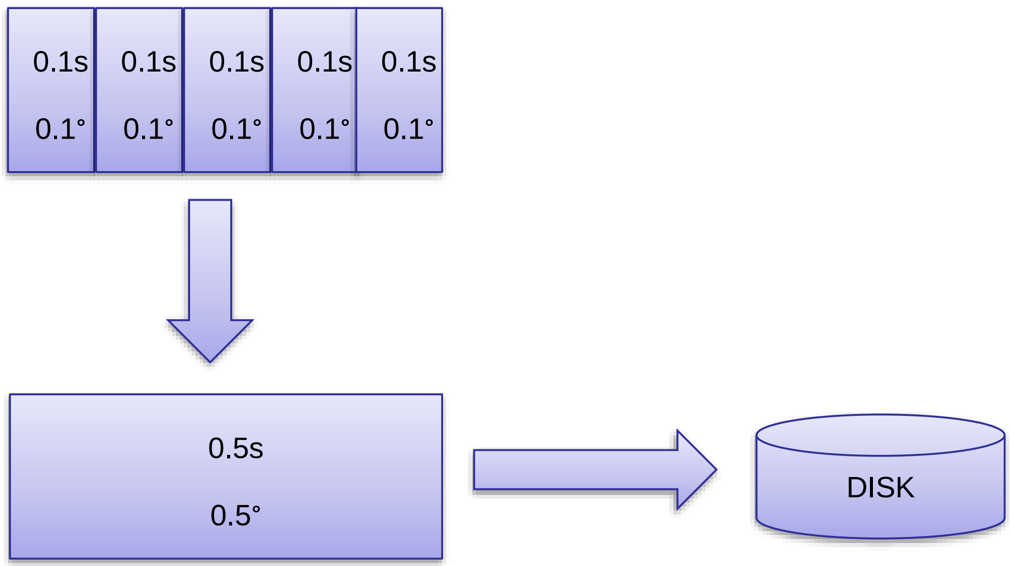
HPC – IMS

- Detector may operate at higher frequency than CrysAlisPro frame rate
- Accumulation of detector frames (high freq) into final frames (lower frequency) is done in memory at acquisition time



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Strategy

Intelligent Measurement System – IMS for HPC

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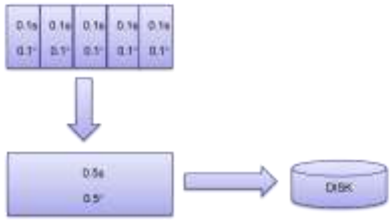

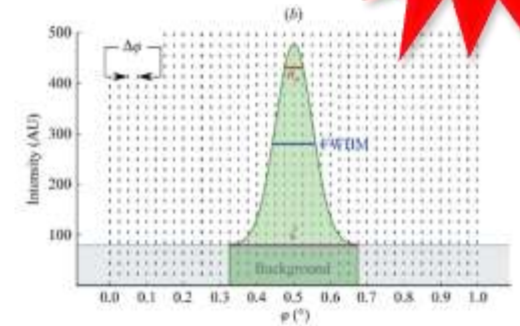

Base scan width

Rate limits
Counter limits

IMS
HPAD

Generator setting

HPAD op mode

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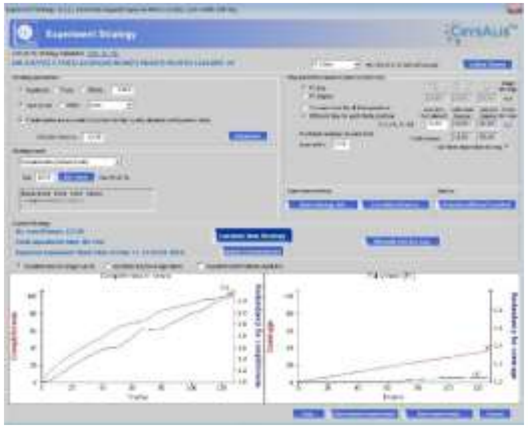
Advances from where?

HW – instrumentation

$$\frac{I}{\sigma}$$



Procedure

Data reduction
Corrections

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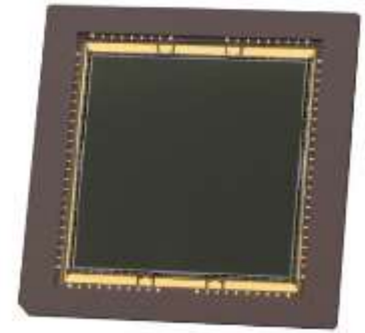
HW – Sources

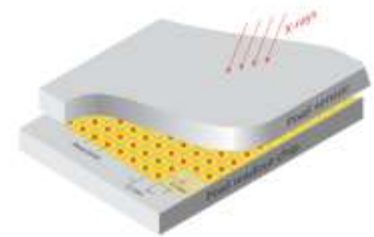
Same sample 0.3mm, normal tubes (2kW, 0.5mm collimator), micro-focus (50W), 007HF (1200W)

Source type	Integral intensity relative Enhance Mo
Enhance Mo	1
Enhance Cu	5
Ultra Cu	40
Nova Cu 2nd gen	240
PhotonJet S Cu	480
PhotonJet R Cu	Up to 3000

HW – Fullwell/Dynamic

Detector generation	Full well X_{ph}	Relative
KM4CCD, Sapphire 2x2	10’000	1
Ruby 2x2	2’500	0.25
Atlas 2x2	3’000	0.3
Atlas – S2 4x4	48’000	4.8
Pilatus 200K	2^{20}=1’000’000	100
HyPix 6000HE	2^{32}=4’000’000’000	40000





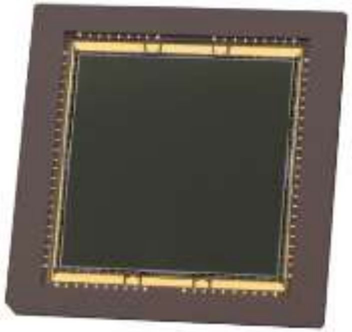
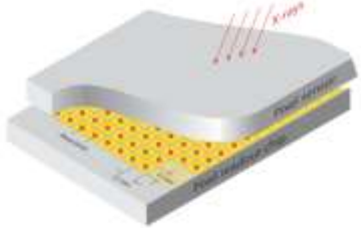
HW – Detector Speed

Detector generation	FPS	Relative
KM4CCD, Sapphire 512^2	0.1	1
Ruby 512^2	0.21	2
Atlas 512^2	0.7	7
Atlas – S2 512^2	1.4	14
Pilatus 200K	20 shutterless 86% duty cycle	200
HyPix 6000HE	100 shutterless ~100% duty cycle	100







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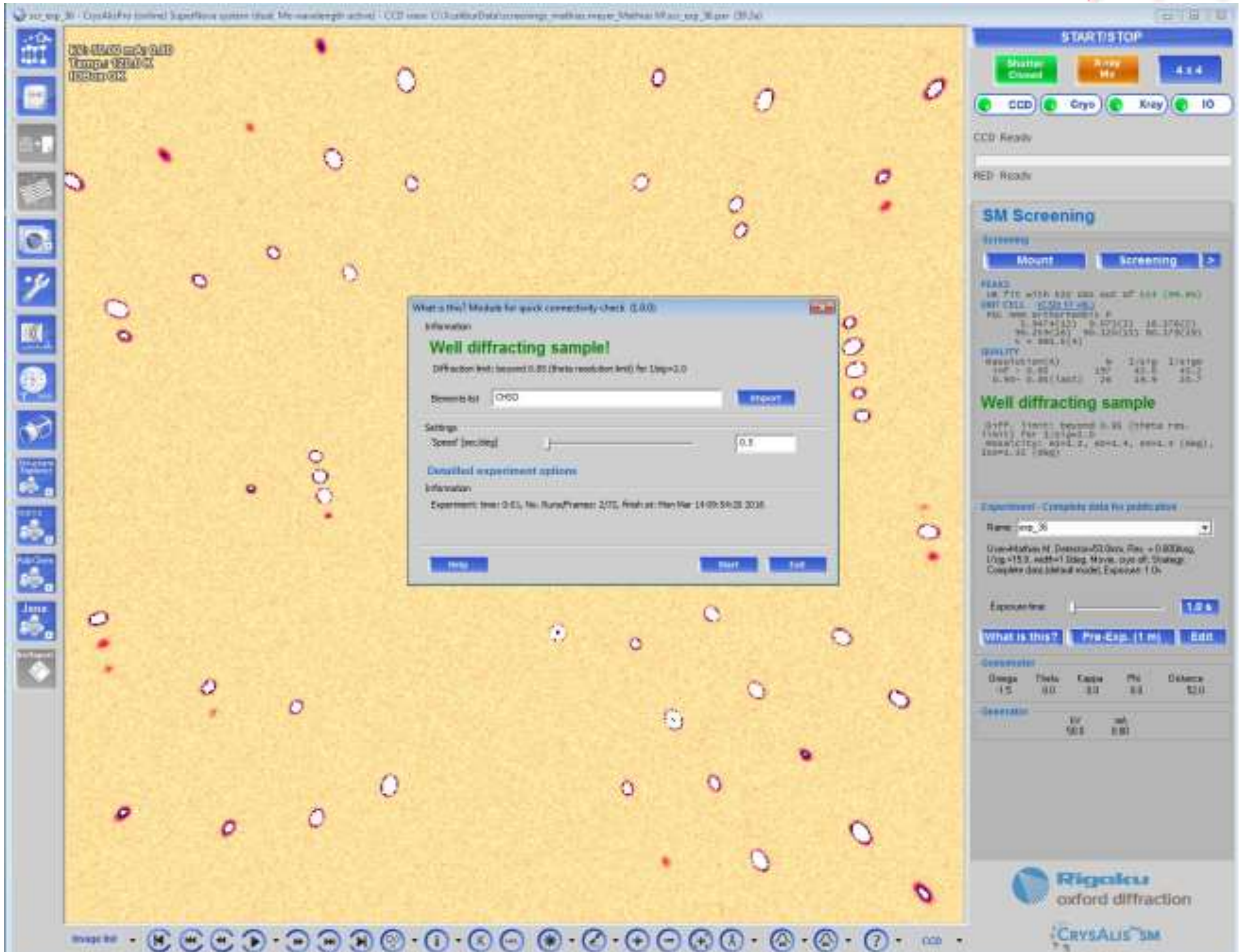
HW – Detector Size

Detector relative size	Unique speed	Observation speed
Eos 1	**1**	**1**
Atlas 2.4	**1.3-1.6**	**1.6-1.8**
Titan 3.7	**1.4-1.8**	**2.0-2.2**

‘What is this?’ tool

New 38

- Available after screening
- Only requires compound elements
- Uses AutoChem2.1/3.0
- Uses up to 5deg/s (CCD) or 10deg/s (HPAD) scan speed!



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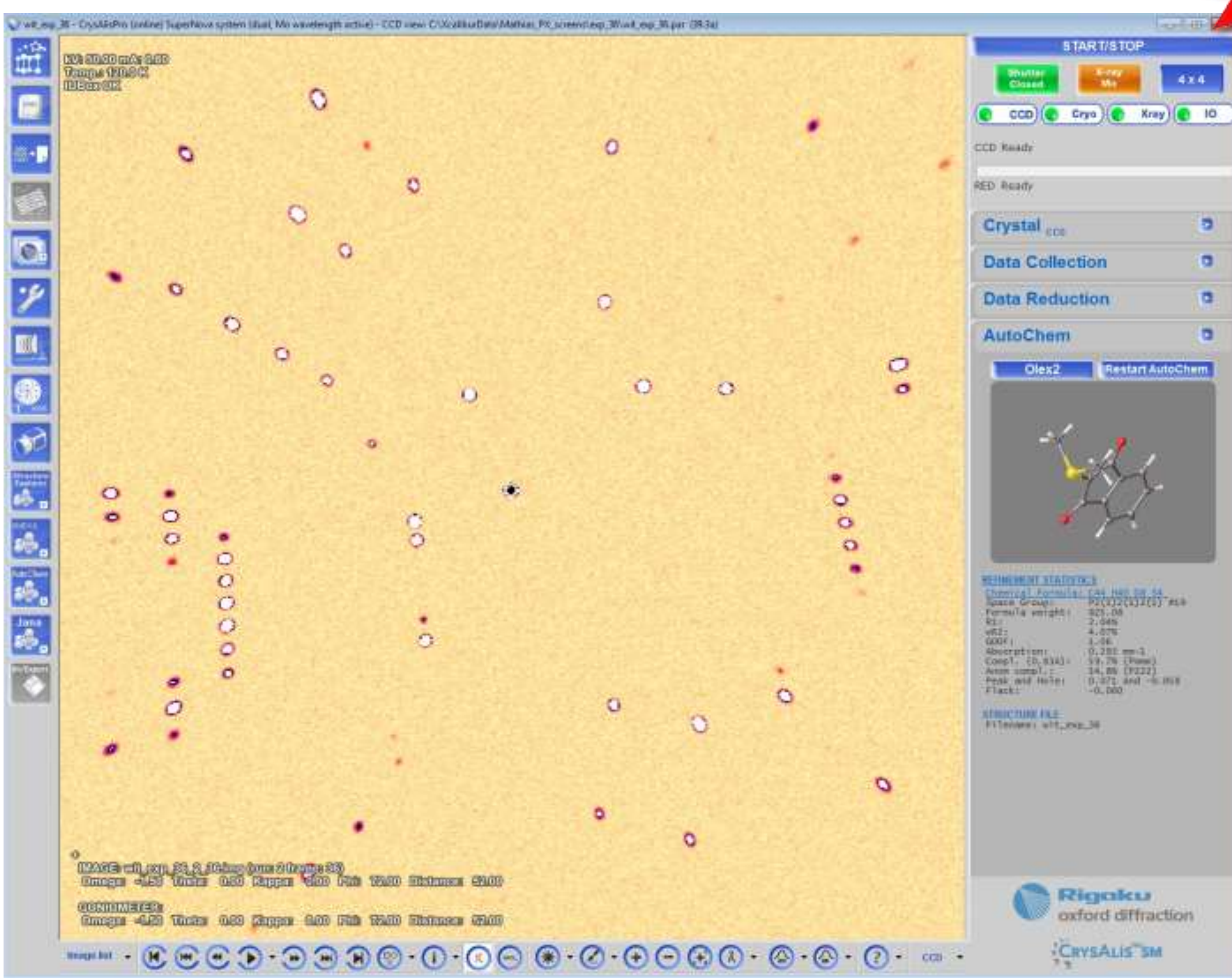
What is this?' tool: 70s later…

New 38



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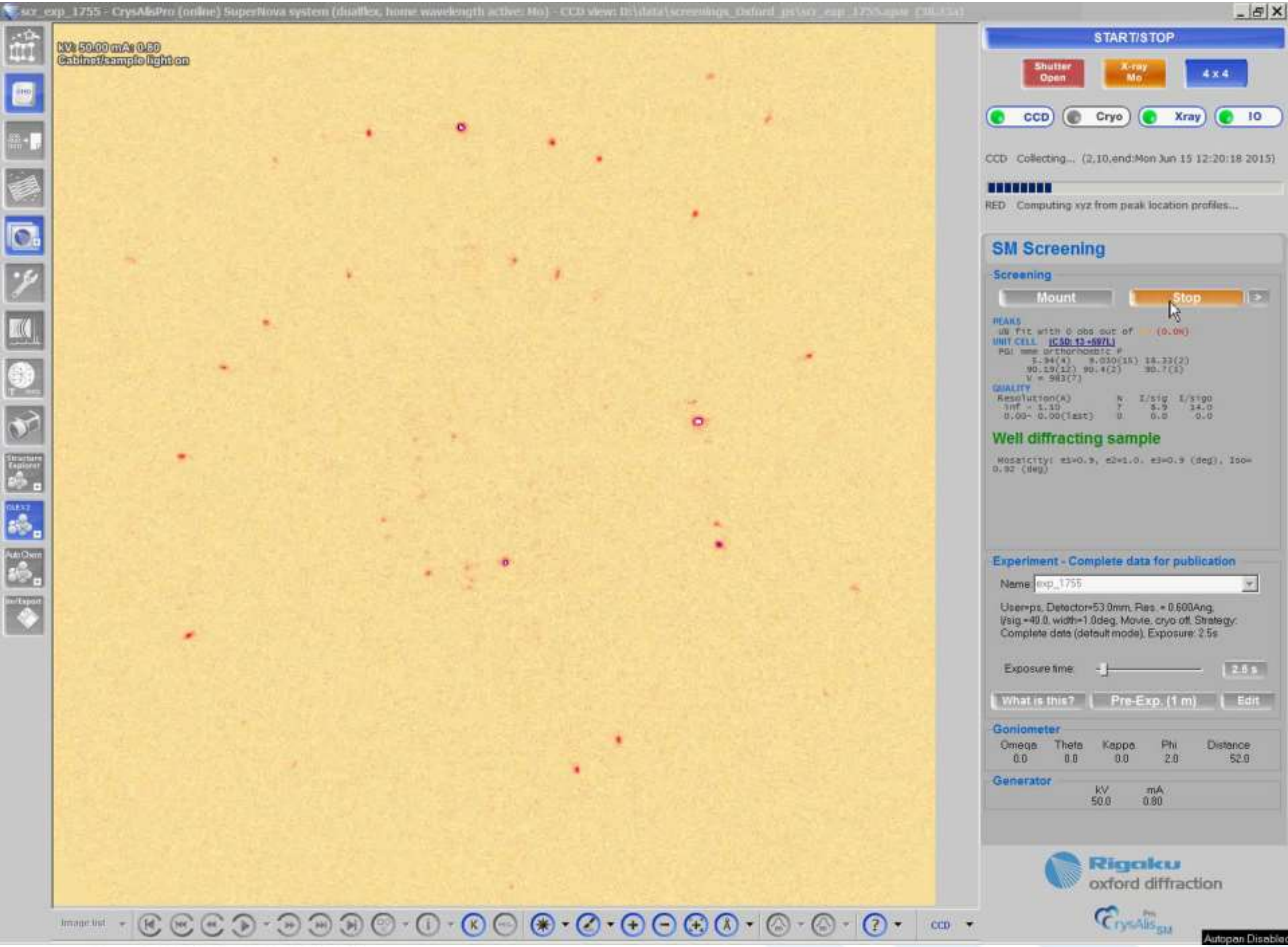
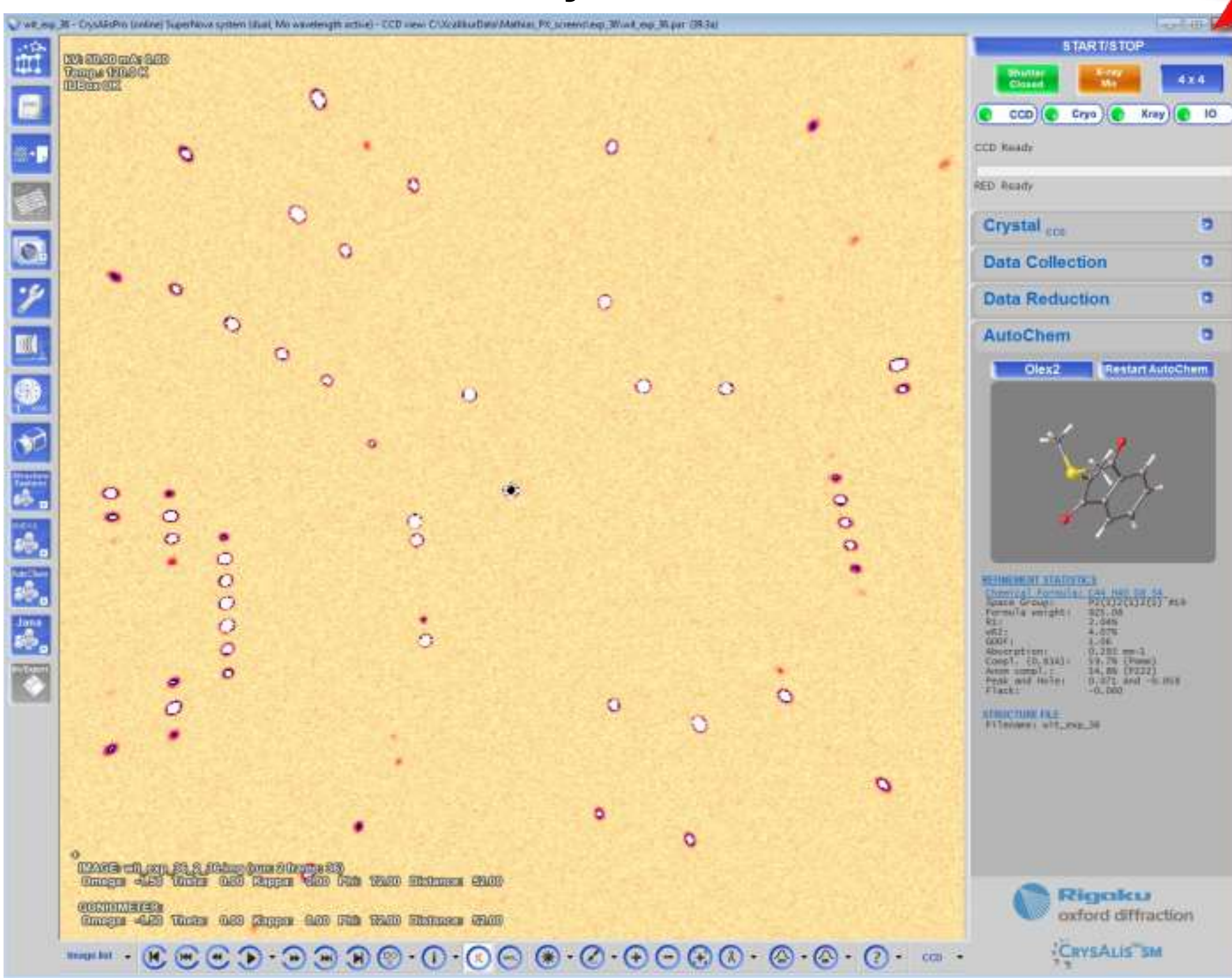


kV: 50.00 mA: 0.80
Cabinet/sample light on
START/STOP
Shutter Open
X-ray Mo
4 x 4
CCD
Cryo
Xray
IO
CCD Collecting... (2,10,end:Mon Jun 15 12:20:18 2015)
RED Computing xyz from peak location profiles...
SM Screening
Screening
Mount
Stop
PEAKS
UNIT CELL
QUALITY
Well diffracting sample
Experiment - Complete data for publication
Name exp_1755
User=ps, Detector=53.0mm, Res. = 0.600Ang,
I/sig.=40.0, width=1.0deg, Movie, cryo off, Strategy:
Complete data (default mode), Exposure: 2.5s
Exposure time
2.5 s
What is this?
Pre-Exp. (1 m)
Edit
Goniometer
Omega Theta Kappa Phi Distance
0.0 0.0 0.0 2.0 52.0
Generator
kV mA
50.0 0.80
Rigaku
oxford diffraction
CrysAlis Pro SM
Image list
CCD
Autopan Disabled

What is this?' tool:

Connectivity solved! <70s

New 38



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XtaLAB Synergy: PhotonJet R, HyPix6000HE

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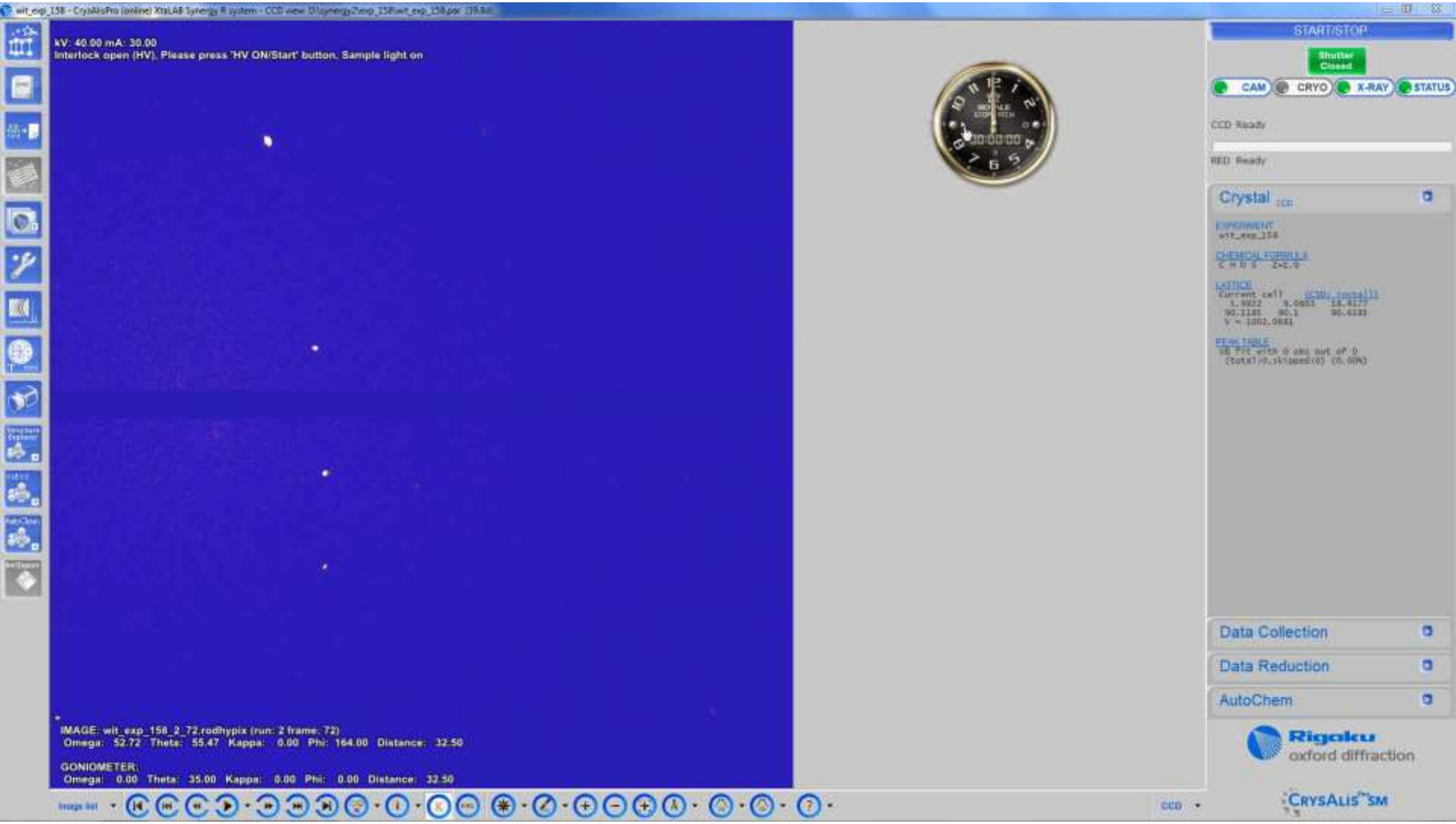
XtaLAB Synergy and HyPix6000HE:

100µm pixel, 100Hz operation, 10deg/s scans



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Synergy: XtaLAB Synergy

New
39

A combination of leading edge components and user-inspired software tied together through a highly parallelized architecture to produce fast, precise data in an intelligent fashion.

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Synergy: XtaLAB Synergy

New 39

NEW PhotonJet sources – our 3rd generation microfocus X-ray sources

NEW goniometer – with motor speeds which have been **doubled**

Closer sample to detector distance

The widest range of available detectors to suit. CCD or HPC? Your choice.

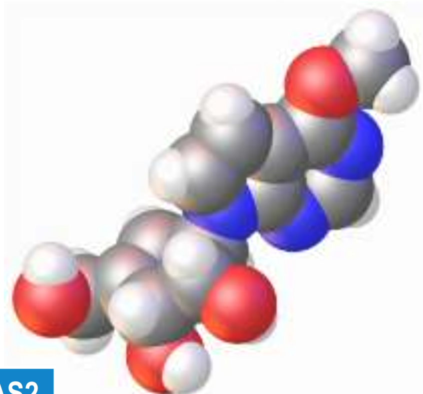
Unique telescoping 2θ arm provides total flexibility for your diffraction experiment.

Enhanced kappa goniometer design with symmetrical 2θ positioning

Rigaku oxford diffraction

Synergy: XtaLAB Synergy

New 39



These results highlight the benefits of the new, faster goniometer, the closer detector distance and increase in source flux of the microfocus source with the Atlas S2 detector.

Experiment parameters	SuperNova AS2	XtaLAB Synergy AS2 very fast	XtaLAB Synergy AS2 using extra data
Crystal to detector distance (mm)	50	35.5	35.5
Completeness to 0.84 Å	99.2	98.6	99.8
Redundancy	2.7	2.1	2.7
Relative goniometer speed	x1	x2	x2
I/sigma to 0.84Å	26	39	59
Experiment time	12 min 48 sec	7 min 38 sec	11 min 17 sec
Rint	0.036	0.021	0.016
R1 (%)	3.97	3.00	2.54

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Ylid data collection IUCR in minimum time

For comparison: SN Atlas: 52mm = 12mins



	Atlas – speed 2deg/s			Pilatus 200k – 5deg/s			HyPix 6000HE – 10deg/s		
Distance [mm]	Time [min]	Runs	Frames	Time [min]	Runs	Frames	Time [min]	Runs	Frames
35	**5**	8	338	**3**	13	495	**2**	9	483
45	**5**	7	350	**3**	13	539	**2**	12	586
55	**7**	10	412	**4**	17	679	**3**	17	814

Thank you for listening!

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