

Detectors for single-crystal area detector diffractometers

Mathias Meyer

X-ray Group Software Manager

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

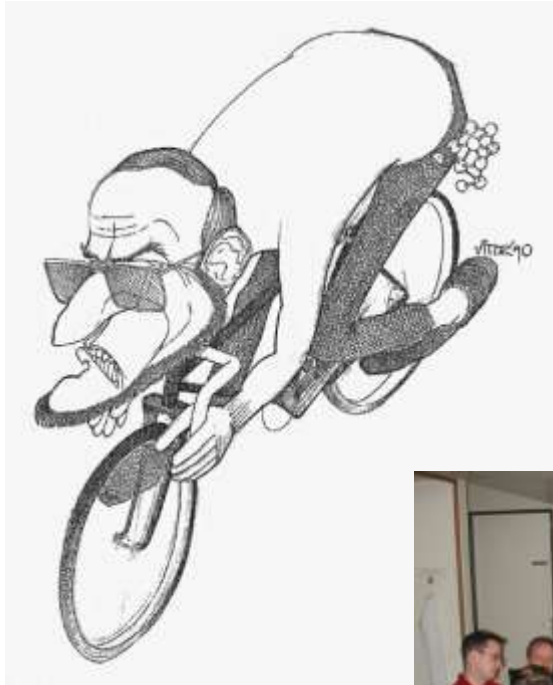
The joining of Rigaku and Oxford opens up a realm of possibilities, the merging of superior hardware, software and expertise will allow for an exciting future in single crystal diffraction. The merging of the two groups is represented in our new logo which takes elements from both.



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



Roots PhD at UNIL 1992...



Frustration 1993: incommensurates

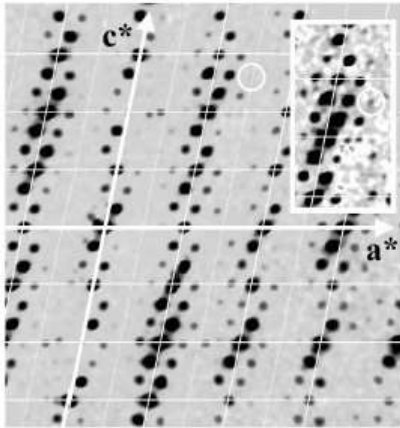
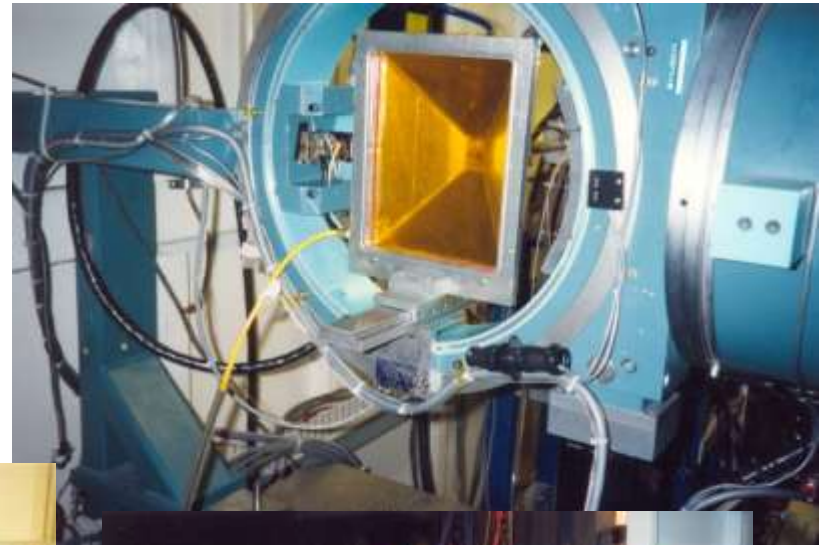
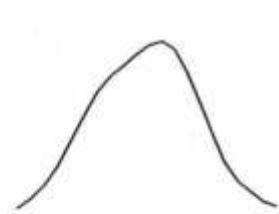


Figure 2
Reconstruction of the reciprocal plane $h2l$ in γ -sodium carbonate. The intersections of dotted and solid white lines correspond to main reflections systematically absent as a result of the C centering. The white circle shows the position of the fifth-order satellite 2215. This reflection is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

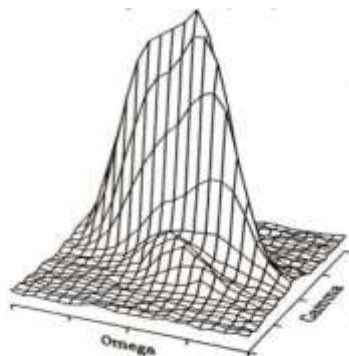
340 Michal Dusek *et al.* • Sodium carbonate revisited



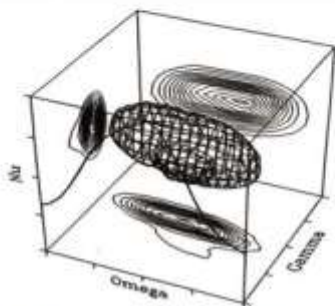
Efficiently measure incommensurate samples?



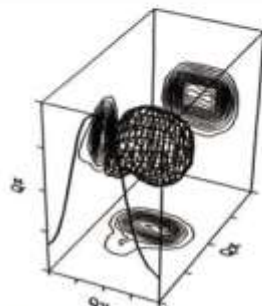
Projection onto the ω axis



Projection onto the ω, γ plane



3D display in angular coordinates

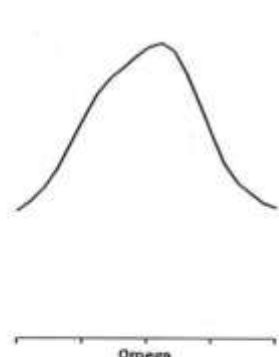


3D display in cartesian reciprocal-space coordinates

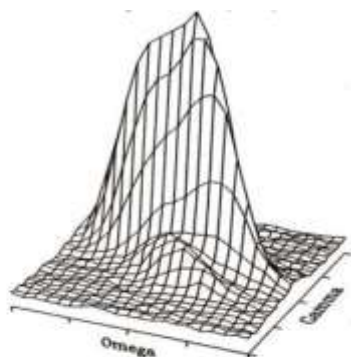


(McIntyre, Neutron News 2, 1992, 15)

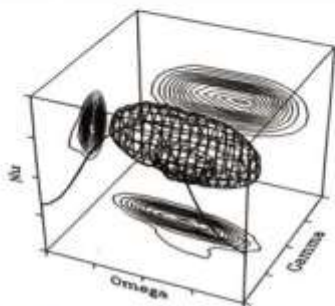
Efficiently measure incommensurate samples?



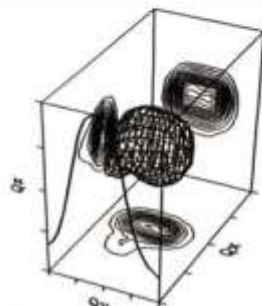
Projection onto the ω axis



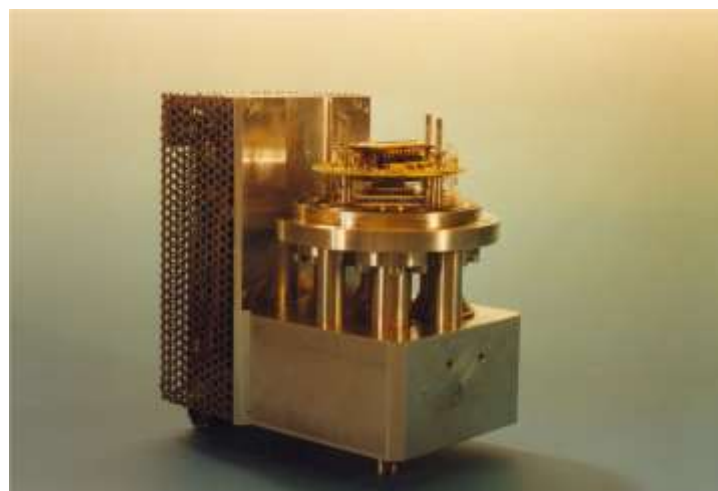
Projection onto the ω, γ plane



3D display in angular coordinates



3D display in cartesian reciprocal-space coordinates



(McIntyre, Neutron News 2, 1992, 15)

A 'Gedankenexperiment'

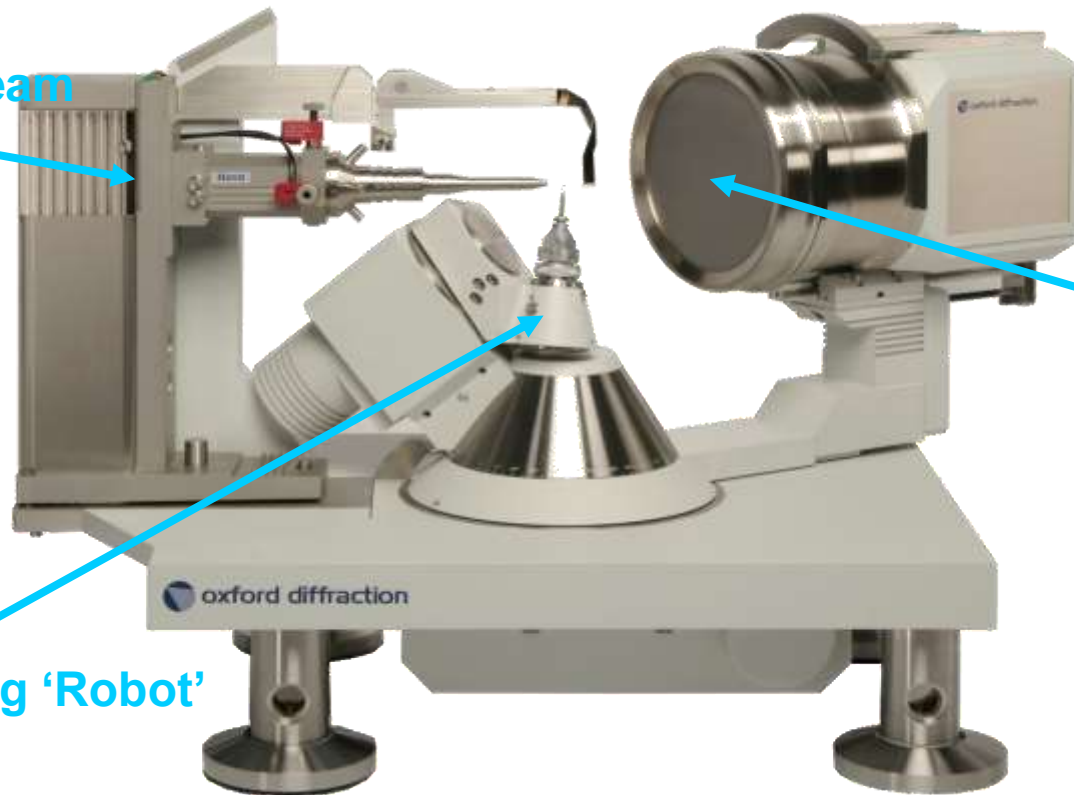
- We build an area detector diffractometer
- Have source
- Have goniometer
- Have an 'ideal detector'

A 'Gedankenexperiment'

X-ray Source
Provides X-ray Beam

Goniometer:
Crystal Positioning 'Robot'

X-ray Detector
-Specialist
digital camera
- 'photos' of
X-rays



The 'ideal' detector



The 'ideal' detector

- What should it do?
 - Experiments at Cu, Mo, Ag, synchrotron?
 - Samples 1 μ m 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

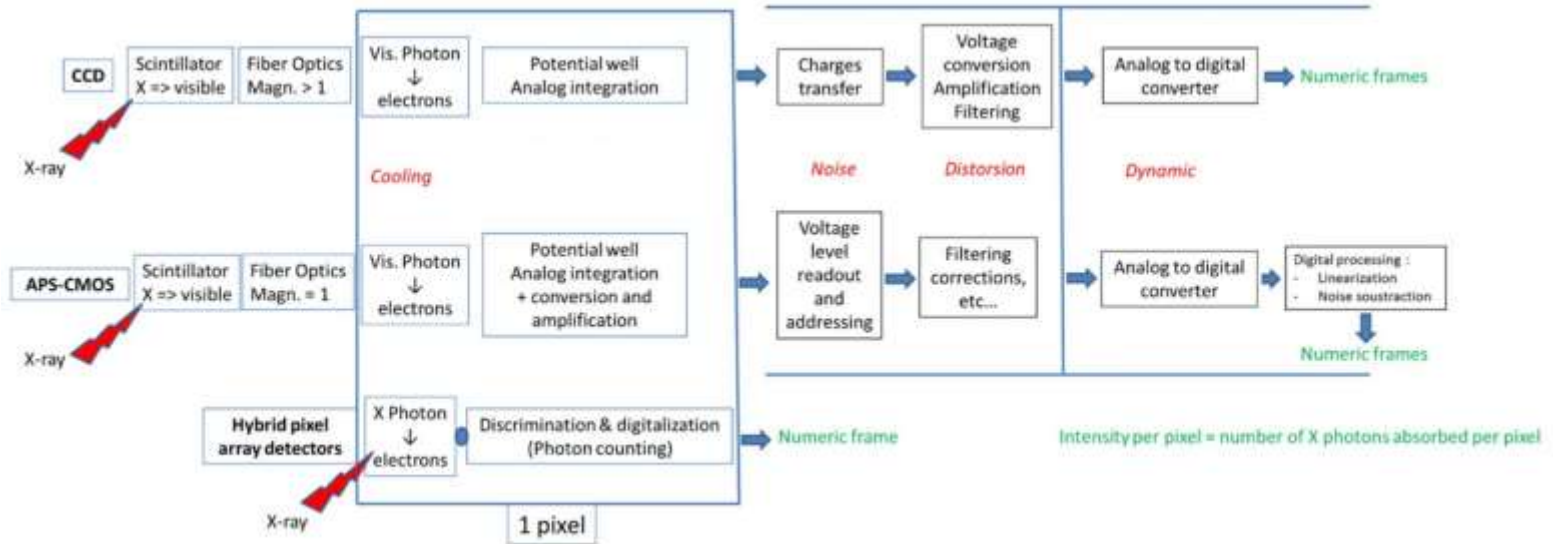
- ACA 2016 launch HyPix 6000
 - 100 microns resolution with top-hat PSF
 - 100Hz – shutterless with near 0 dead-time
 - 10 deg/sec top dc speed, very fast positioning
 - PhotonJet sources
 - WIT in 17s
 - Full mmm data set in ~2min
 - P1 = full sphere in <15min



Synergy R and HyPix6000HE: *100 μ m pixel, 100Hz operation, 10deg/s scans*



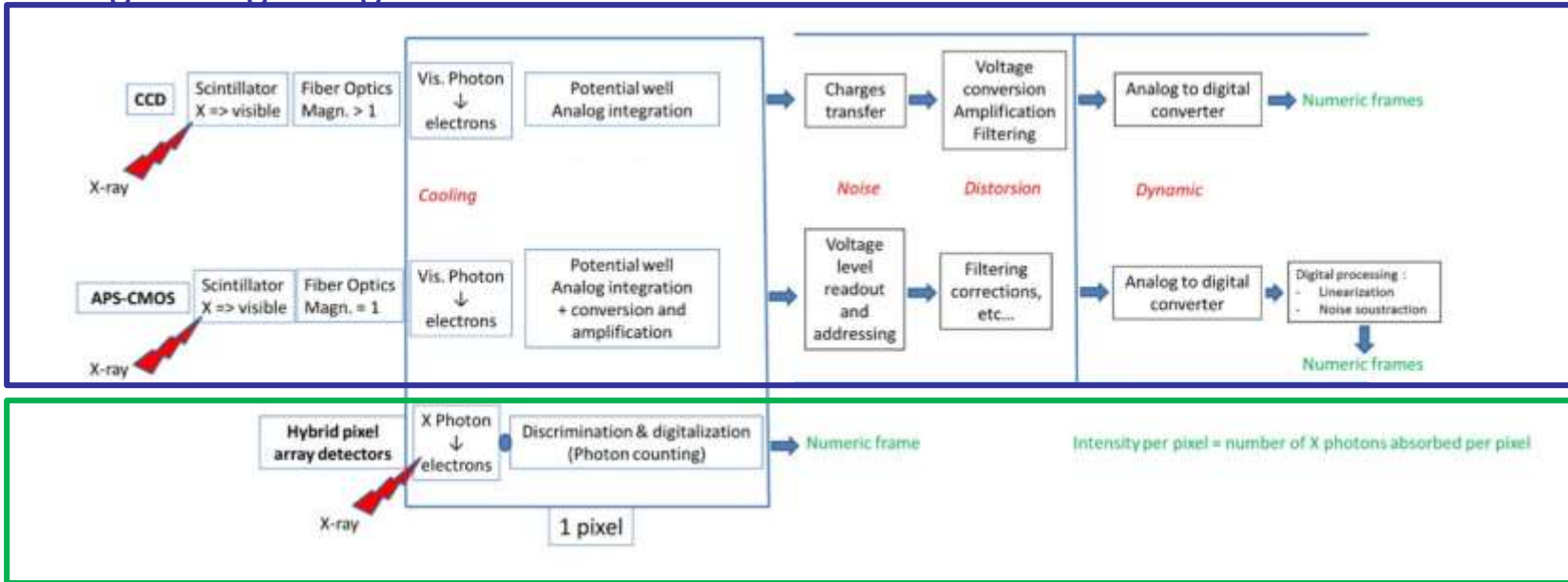
The 'real' detectors SX AD



*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

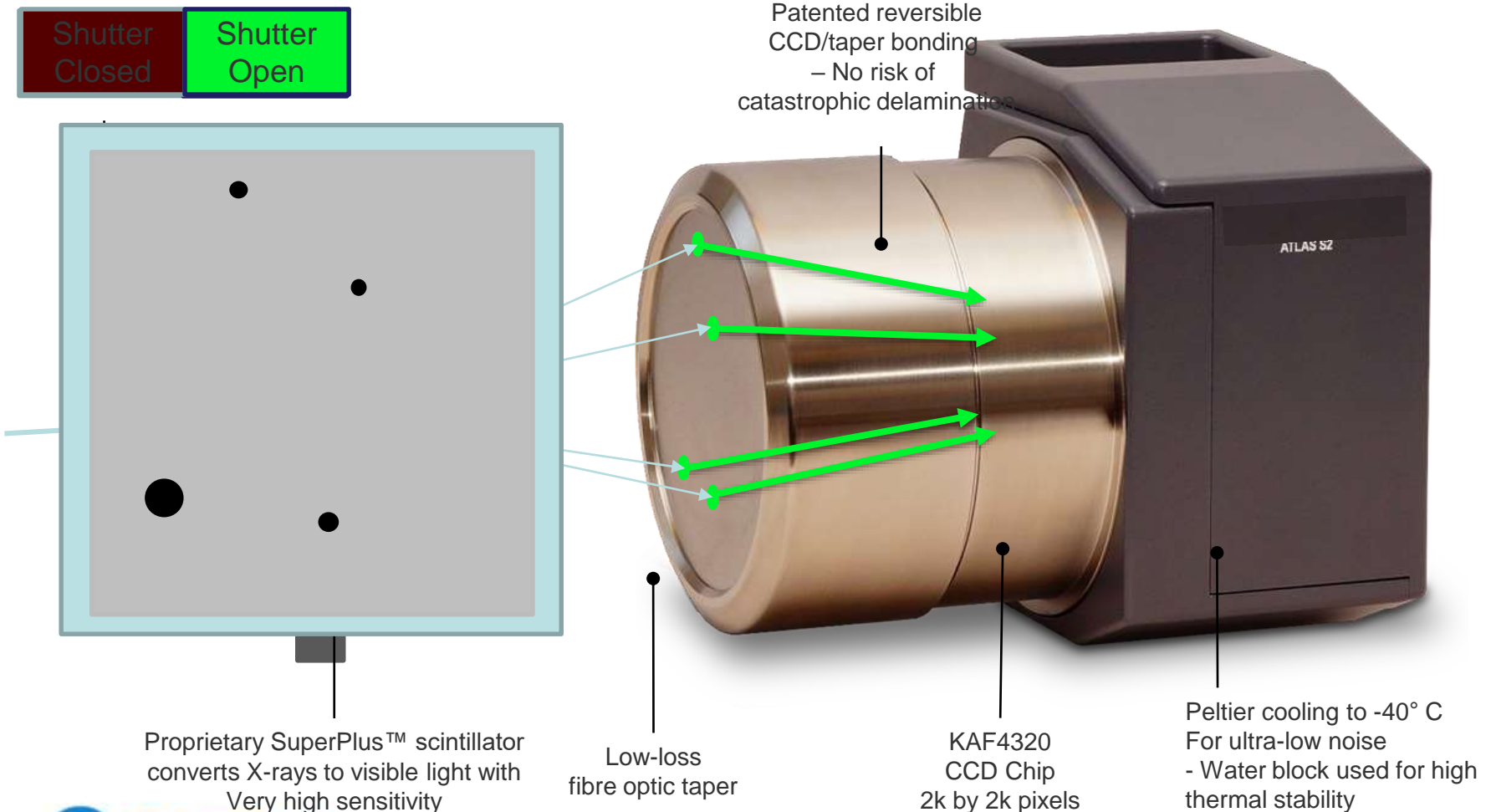
Charge integrating detectors



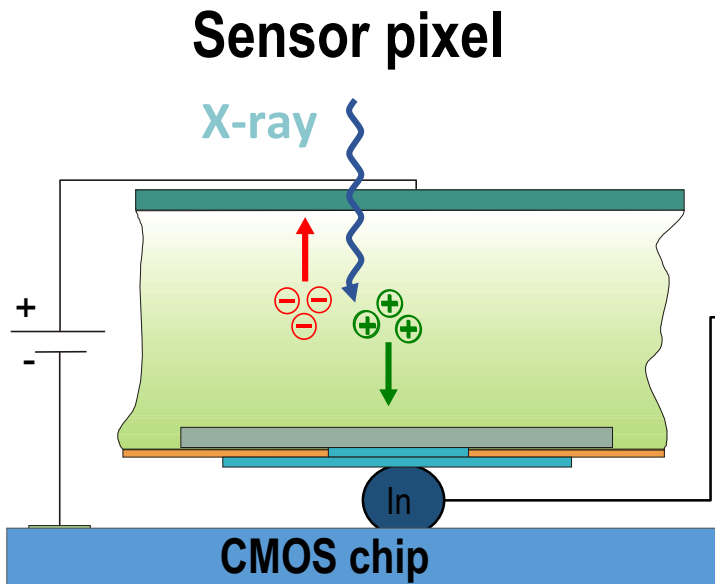
Event counters: HPAD, HPC – Hybrid pixel counters

*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

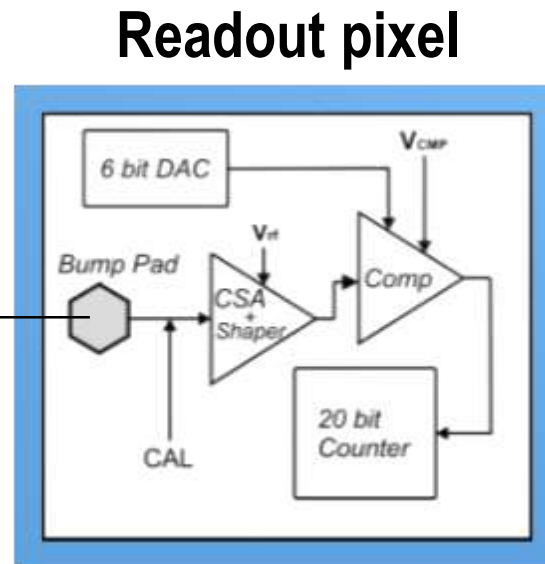


The closest thing to an ideal detector...



Direct Detection of X-rays in silicon sensor

→ Point Spread Function of 1 pixel



Single Photon-counting in CMOS

→ no readout noise & dark current

→ high dynamic range (20 bit)

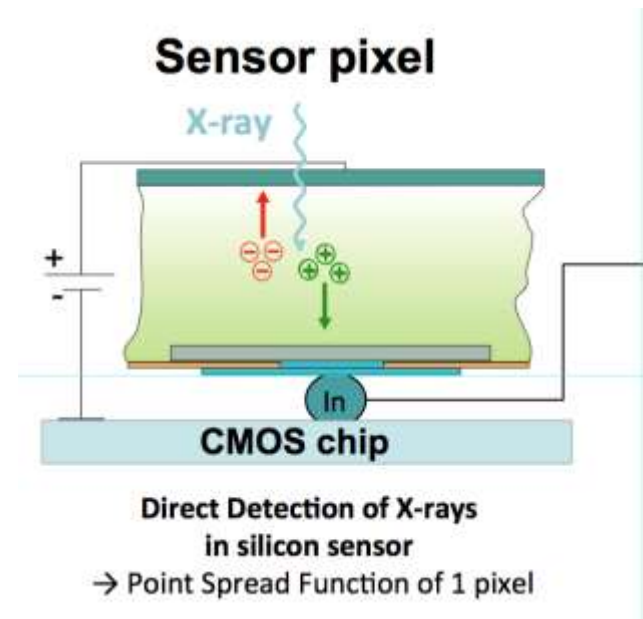
→ fast readout

CMOS: Complementary metal–oxide–semiconductor

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

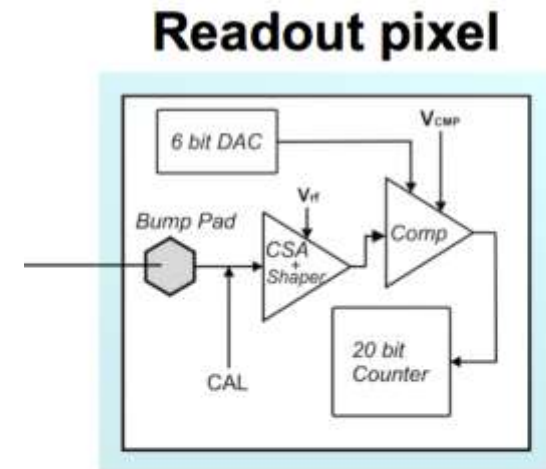
Key Features of HPC Detectors

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



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 rates: up to 2×10^6 photons per second *per pixel*
- Short readout time: 7 ms
- Frame rate up to 20 images per second



Single Photon-counting in CMOS

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

adapted from: Dectris

The 'real' detectors SX AD

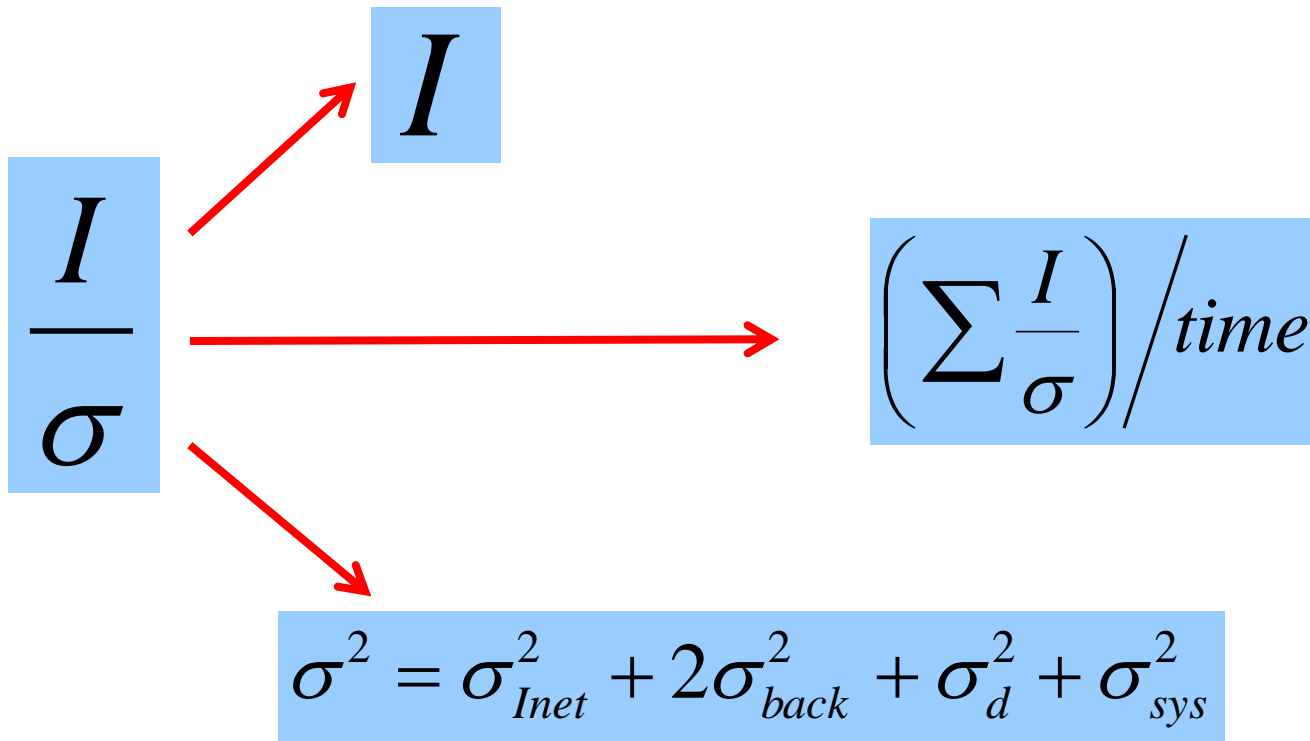
Integrative detectors ,CPAD'		Event counters ,HPAD' HPC – Hybrid pixel counters
Indirect detection via X-ray scintillator		Direct detection via photo-electric effect
Light conduction via taper/fiber glass		-
Light detection ,Integrating'		Charge detection ,Photon counting'
CCD	CMOS	-
FET->ADC->memory		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



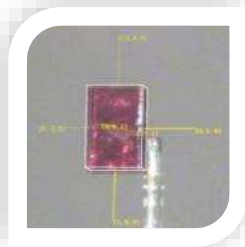
What is data quality?



The single crystal diffraction experiment



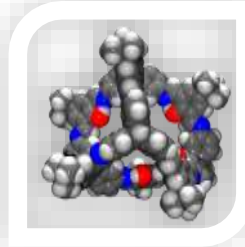
Inorganic Materials



Absorbing Samples



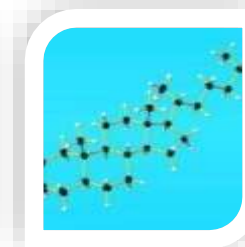
Mo vs. Cu



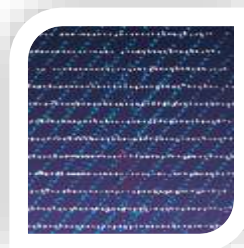
Benefits of Cu



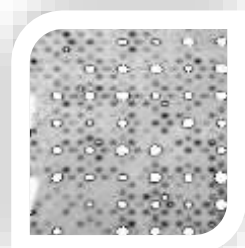
Small Crystals



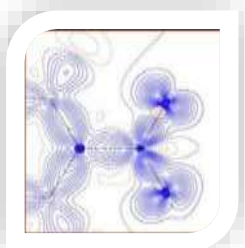
Absolute Configuration



Twinning



Incommensurates



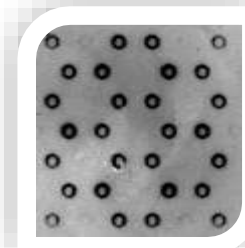
Charge Density



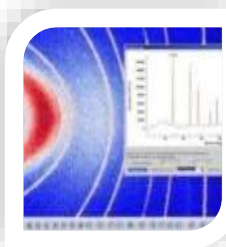
High Pressure



Large Crystals



Diffuse Scatter



Powder Diffraction



Photocrystallography



Novel Applications

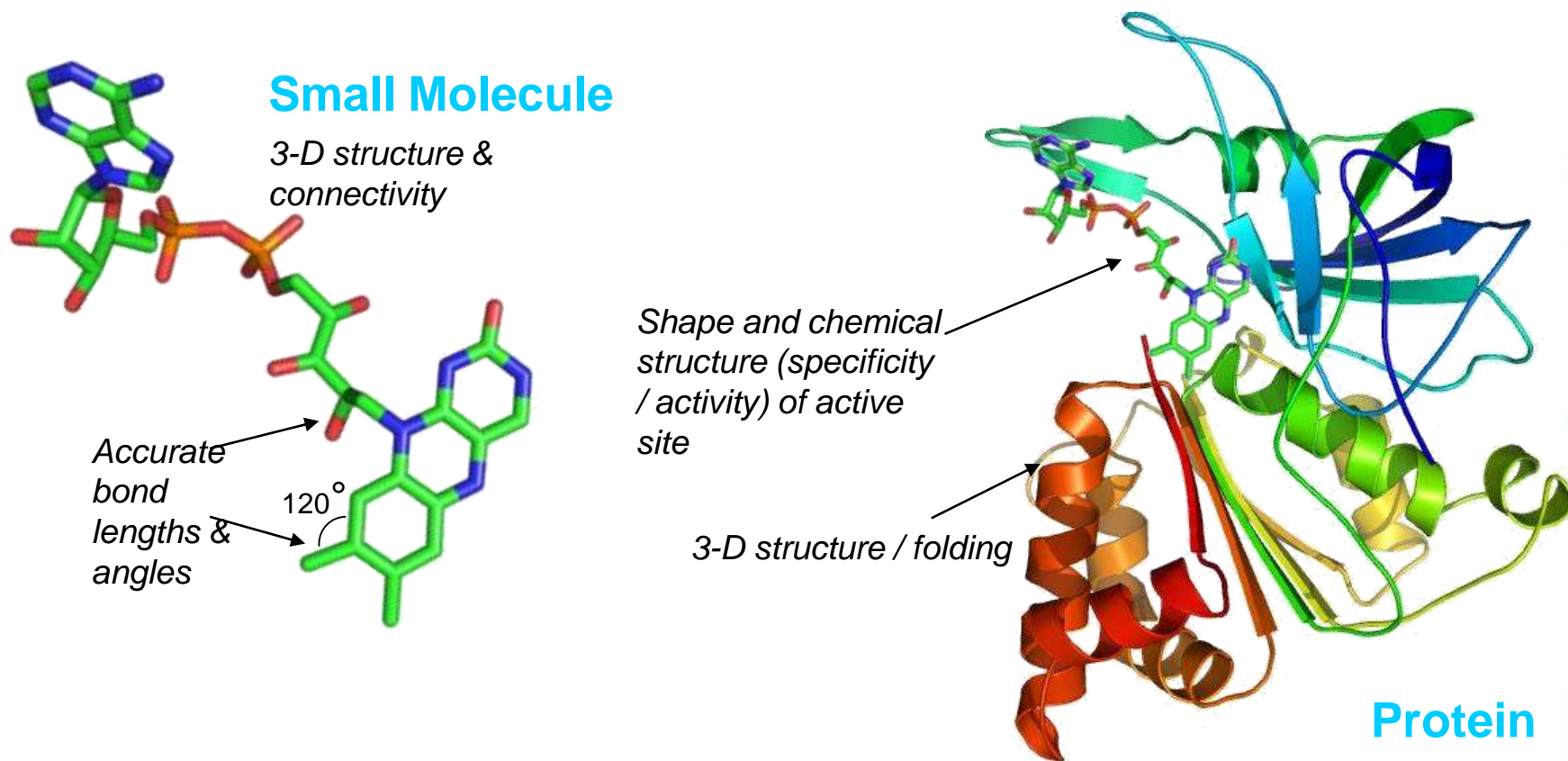


Multi-Temperature

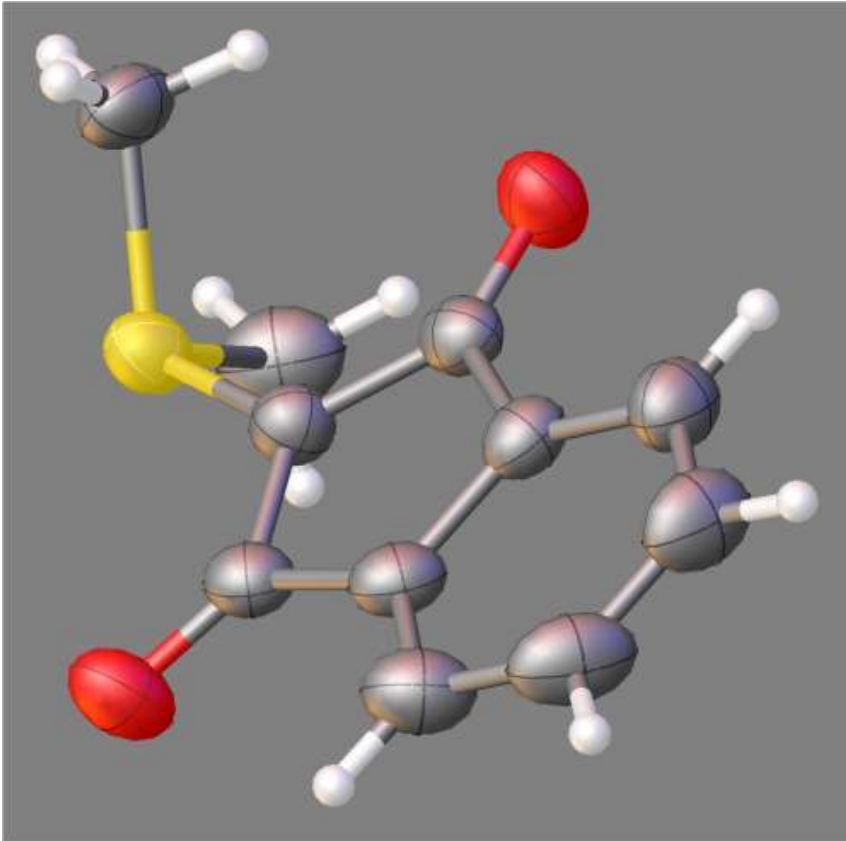


Quasi-Crystals

The single crystal diffraction experiment

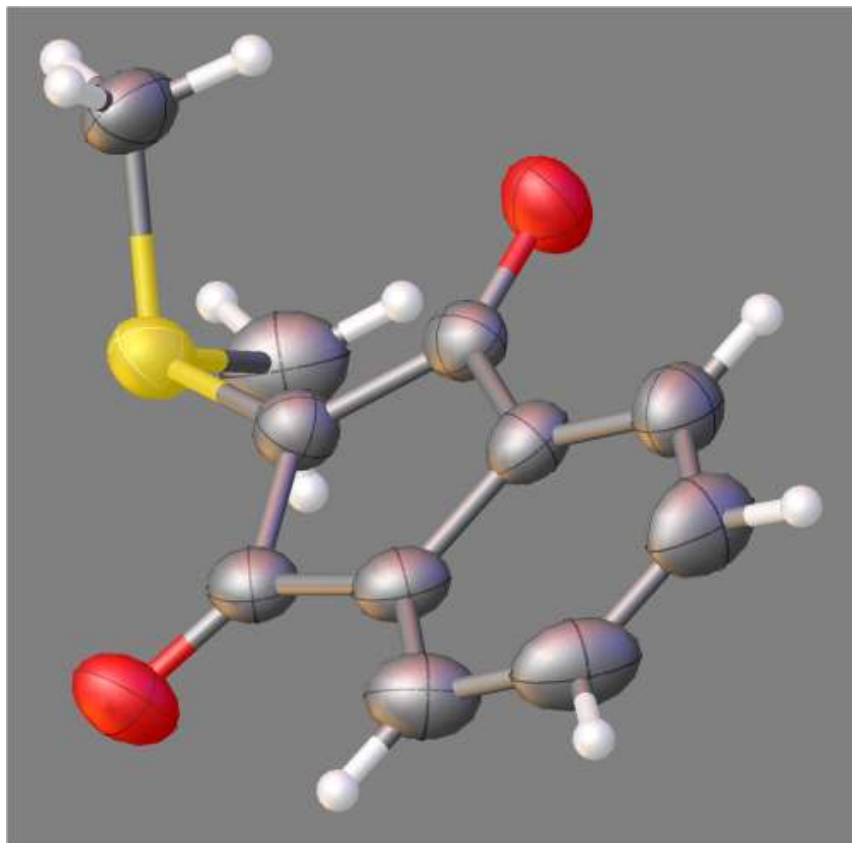


Importance of weak data

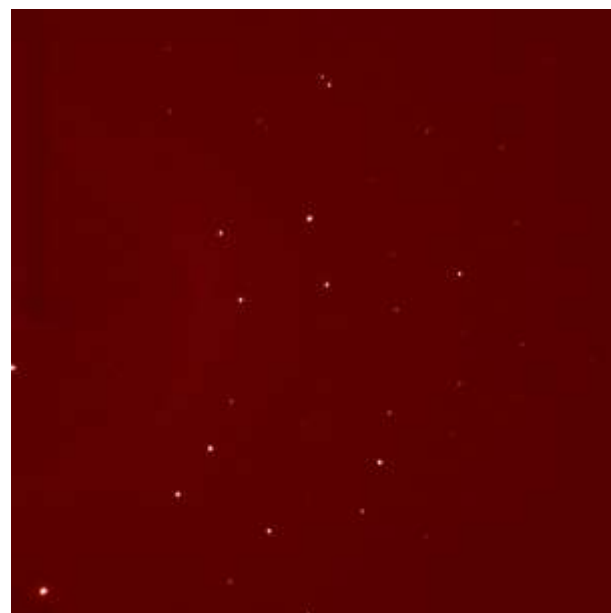


- Make a typical experiment
 - Cu radiation
 - Resolution 0.78Å
 - $(I/\sigma)_{\text{mean}} = 15$ to 0.837Å (IUCR)

Importance of weak data

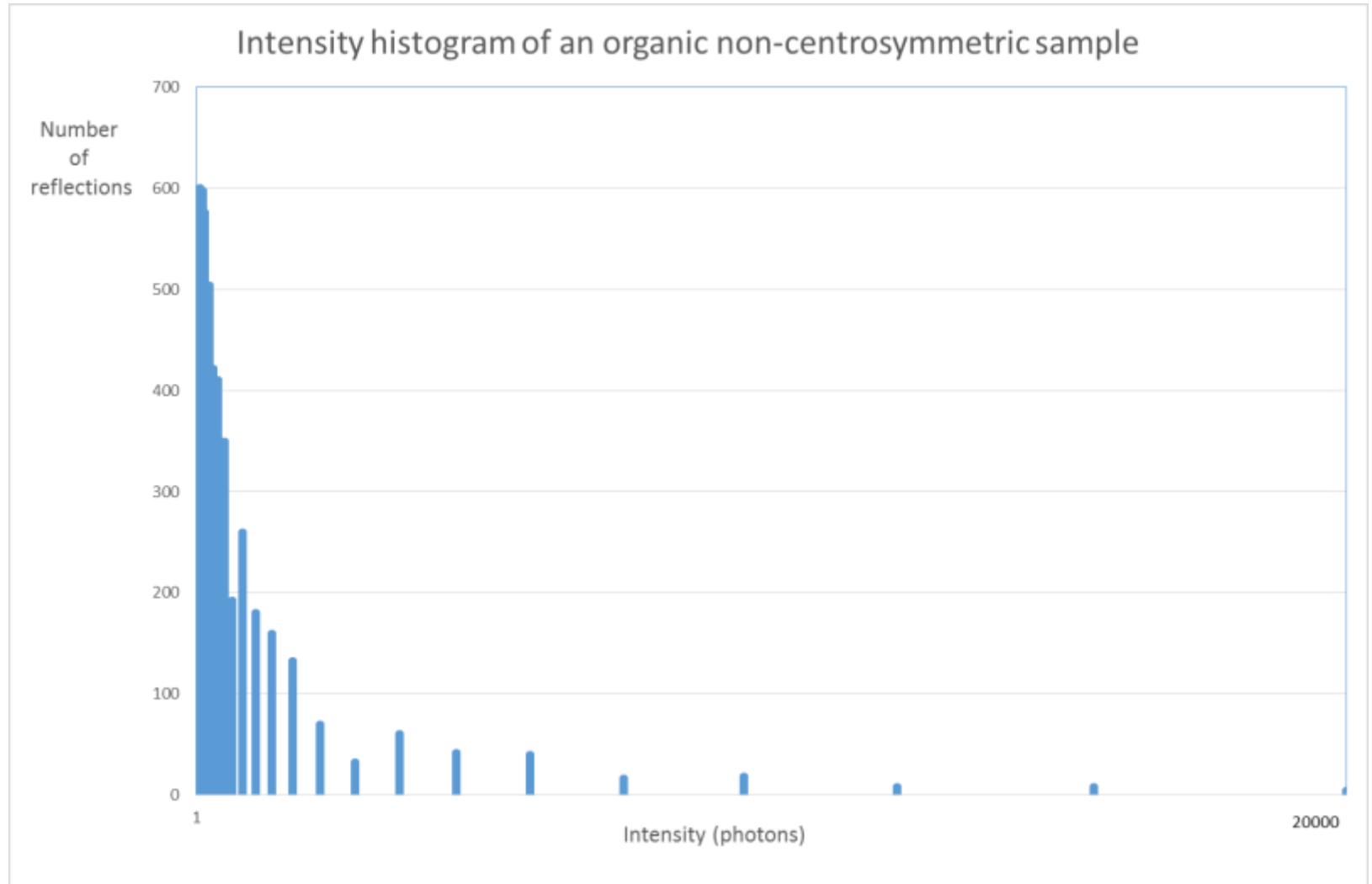


- Make a typical experiment
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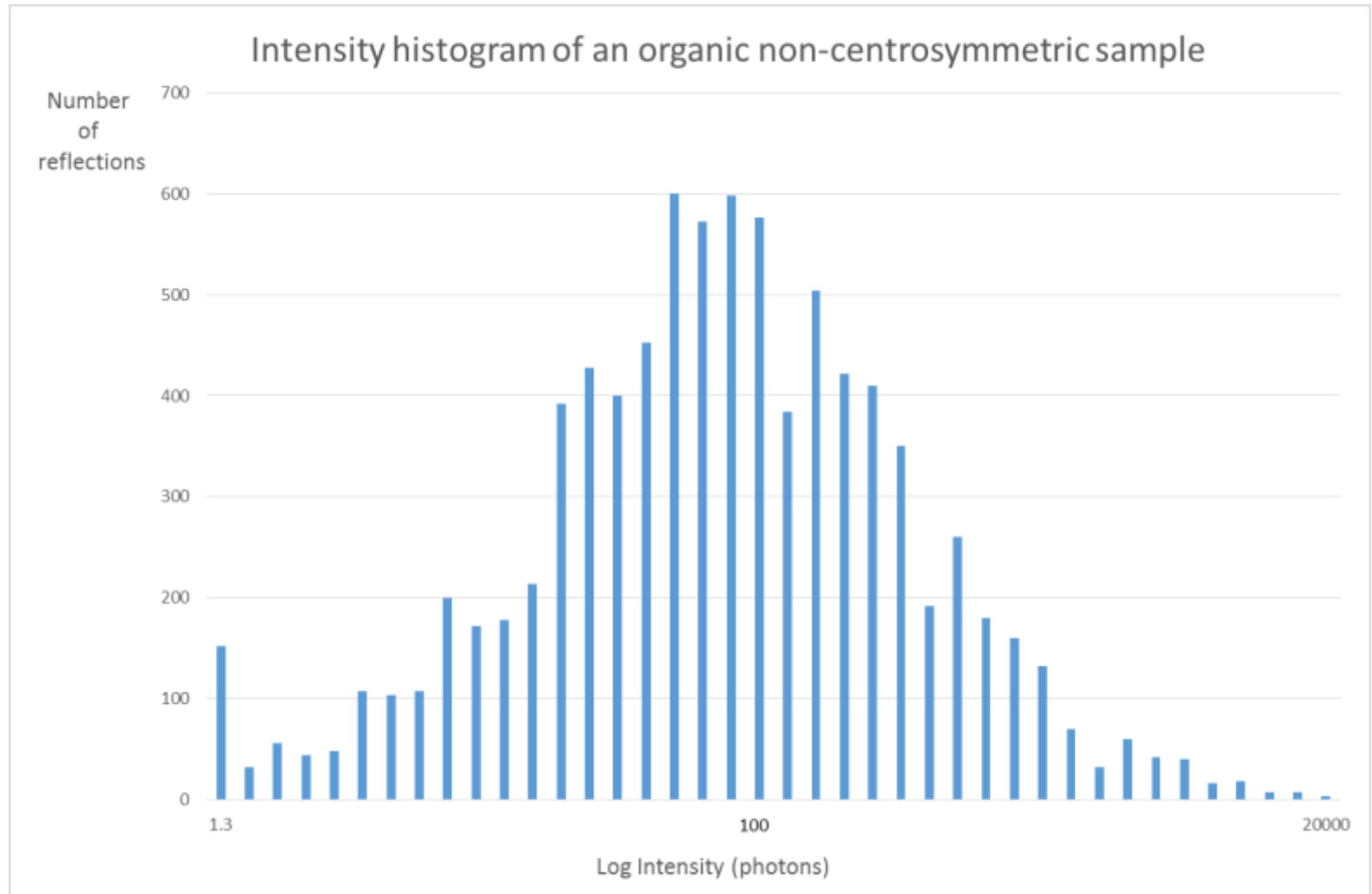
Importance of weak data

Histogram of data



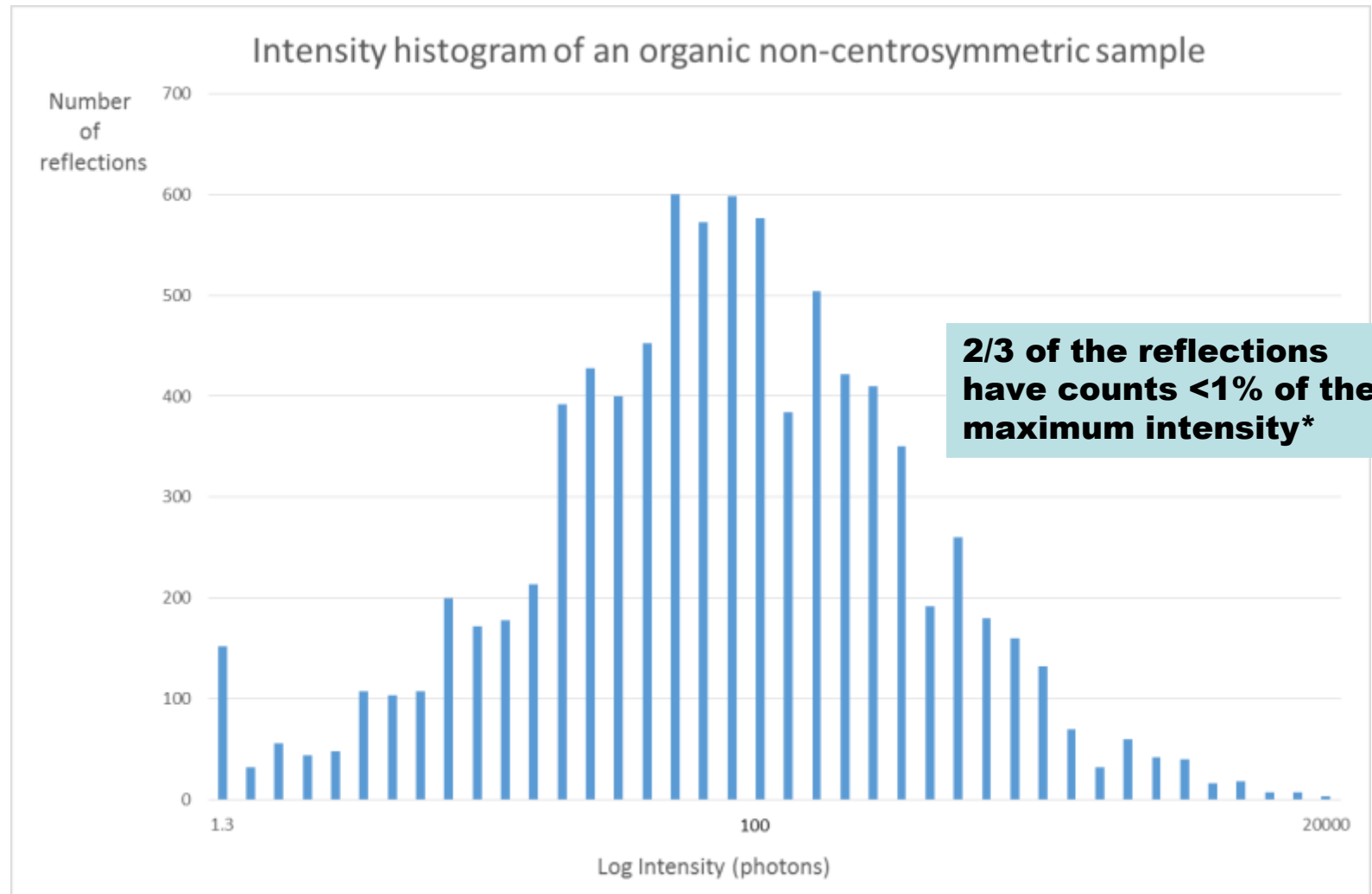
Importance of weak data

Histogram of data



Importance of weak data

Histogram of data



*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)

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

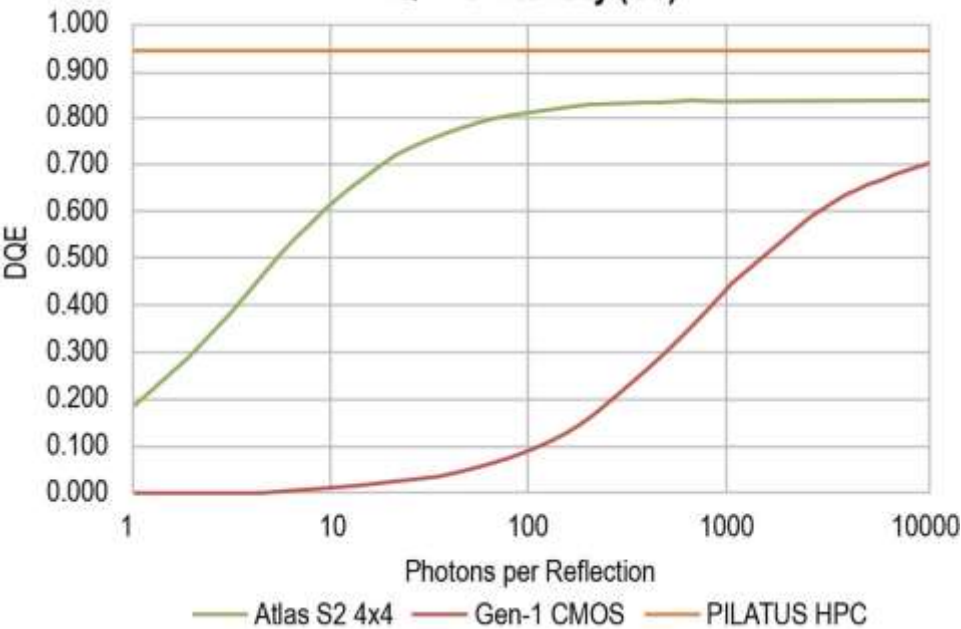
Diagram illustrating the components of the Detective Quantum Efficiency (DQE) equation:

- $T_w \eta_{ph}$: Window Scintillator
- $1 + \frac{1}{g}$: Gain
- $\frac{A(n_r^2 + i_d t)}{IT_w \eta_{ph} g^2}$: Noise: Read and dark

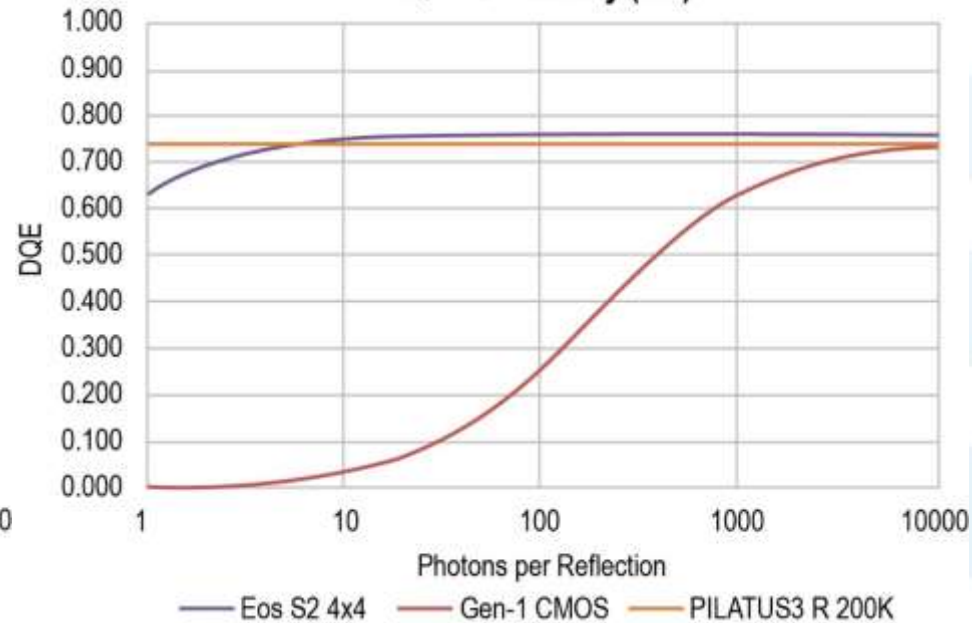
M. Stanton et al., J. Appl. Cryst. (1992). 25, 638-645

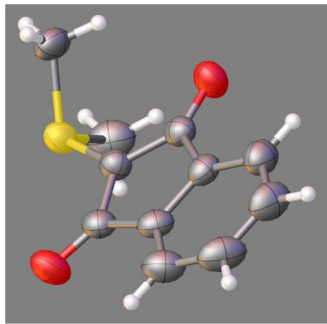
DQE: Cu and Mo

DQE vs Intensity (Cu)



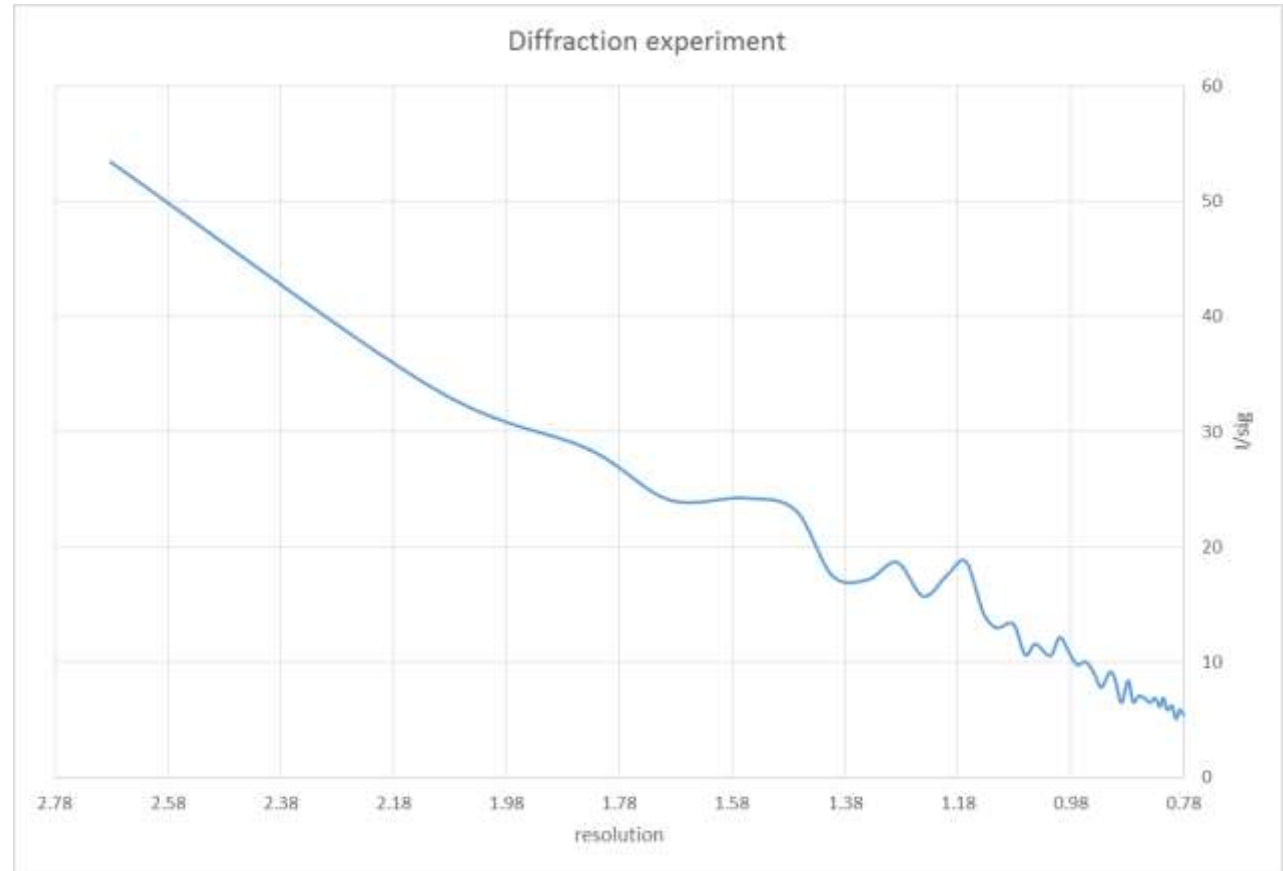
DQE vs Intensity (Mo)

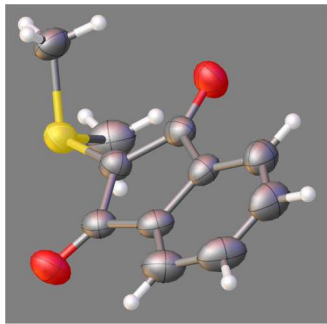




Importance of weak data

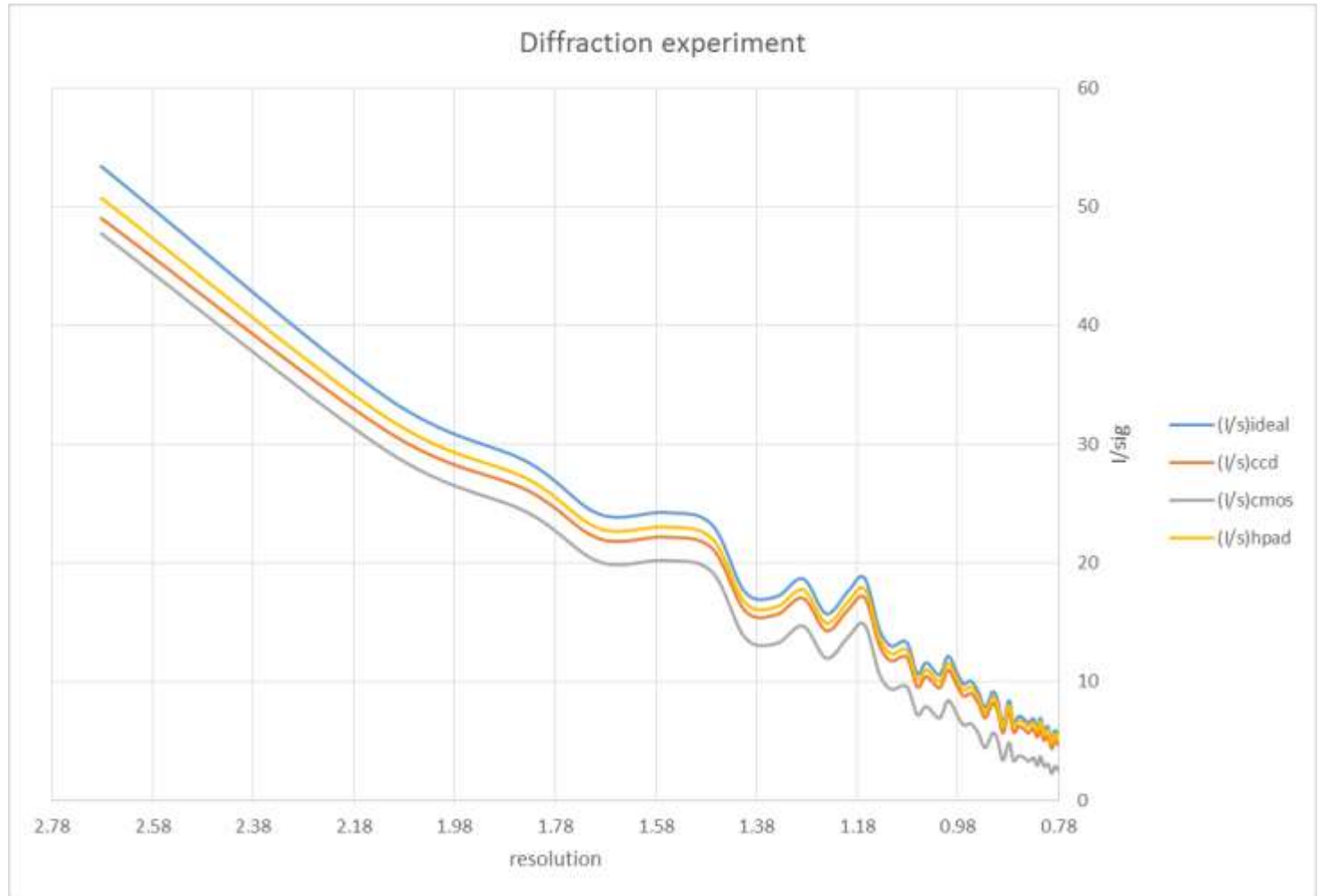
Comparison of tech

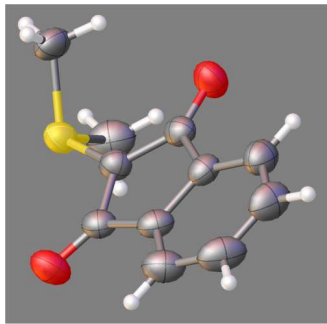




Importance of weak data

Comparison of tech



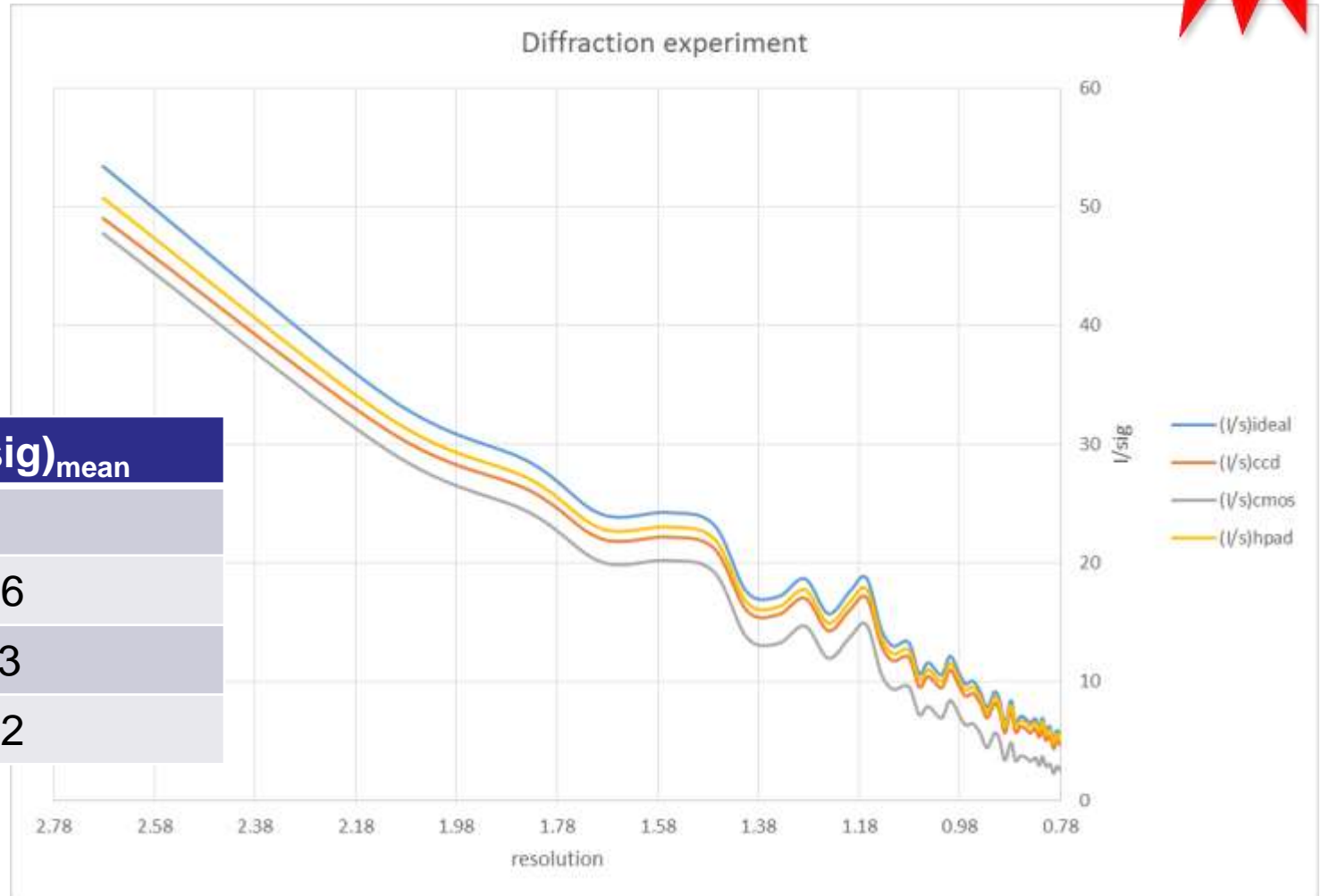


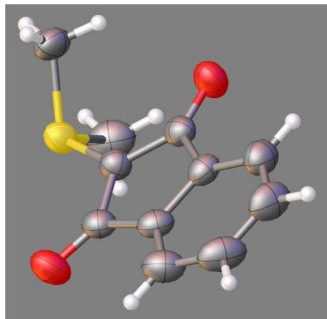
Importance of weak data

Comparison of tech



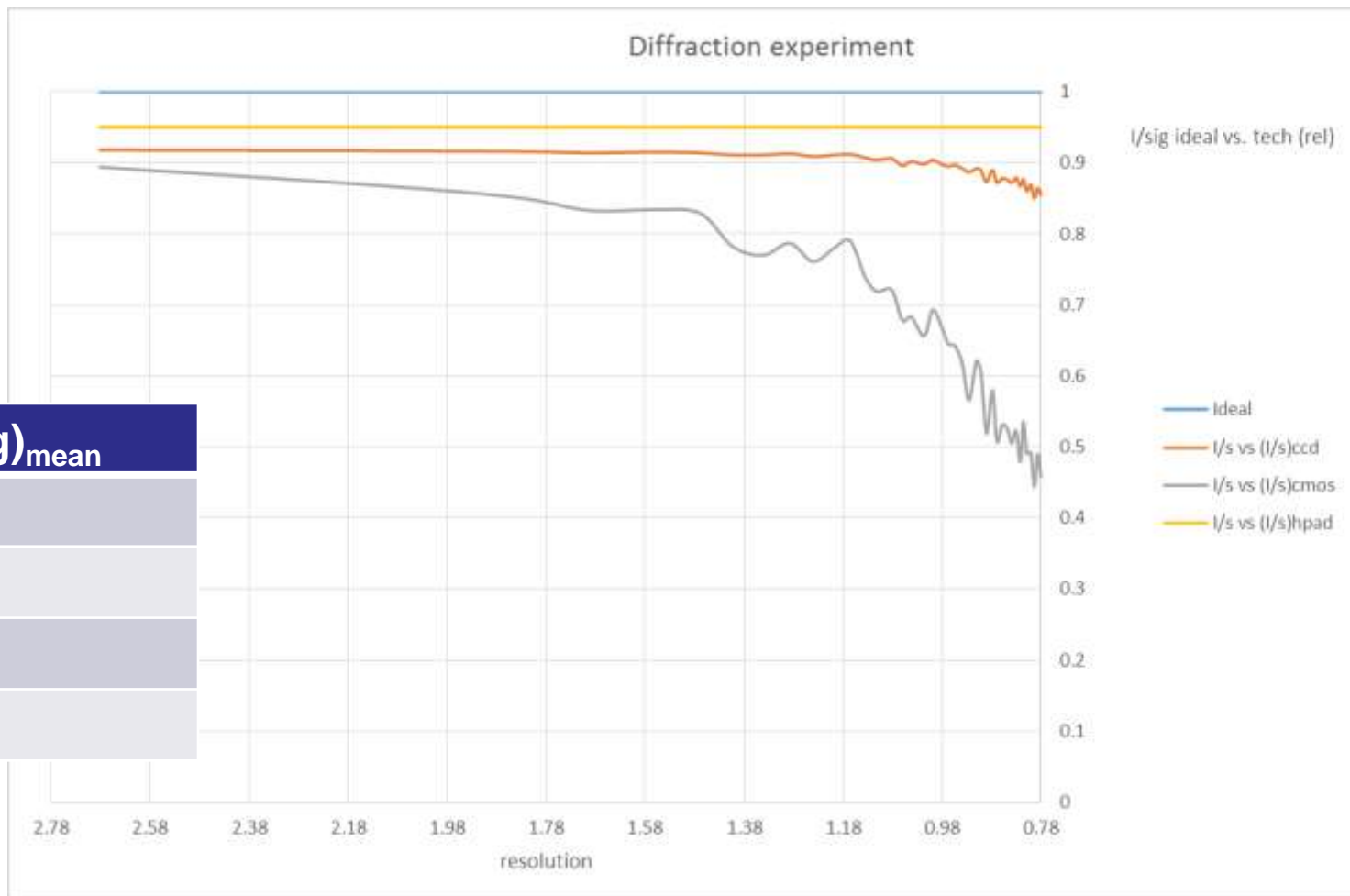
Tech	$(I/\sigma)_{\text{mean}}$
Ideal	15
CCD	13.6
CMOS	11.3
HPAD	14.2

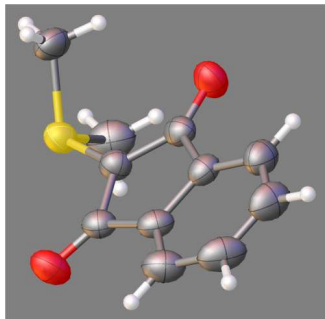




Importance of weak data

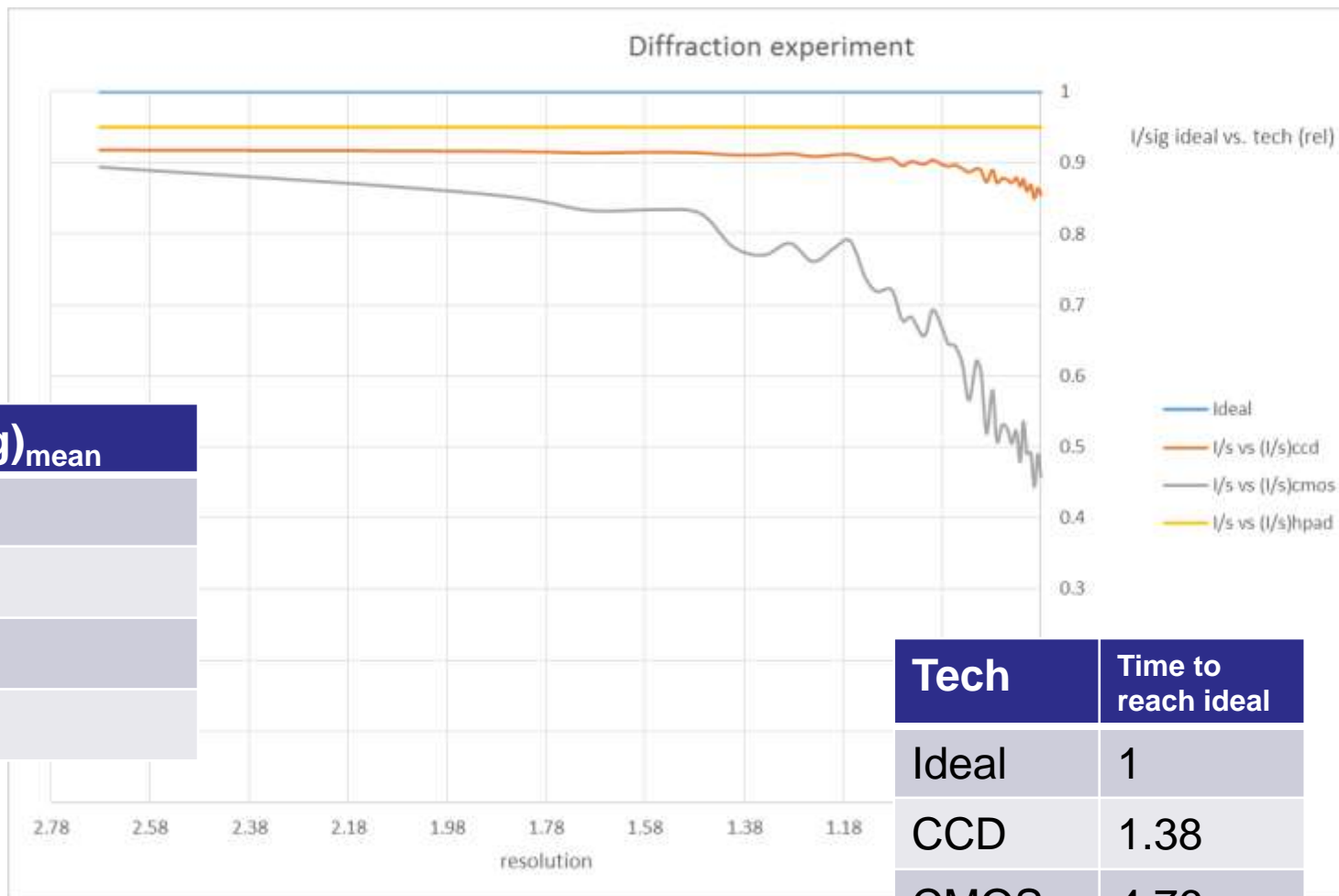
Comparison of tech





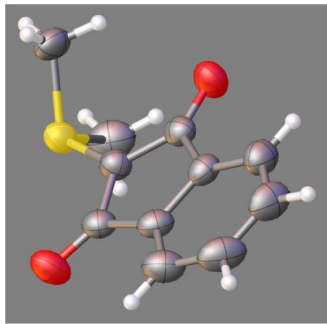
Importance of weak data

Comparison of tech



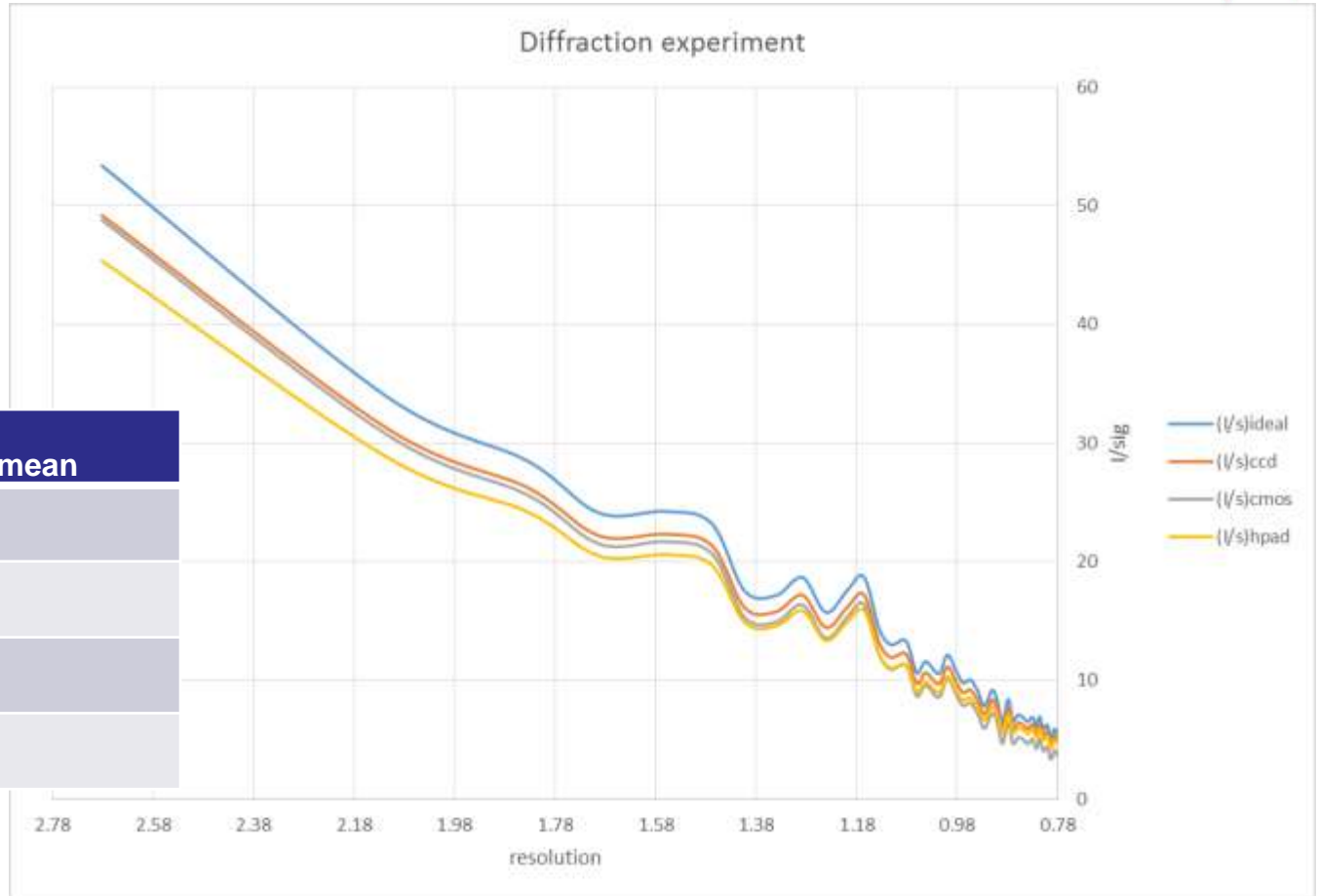
Tech	(I/sig) _{mean}
Ideal	15
CCD	13.6
CMOS	11.3
HPAD	14.2

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

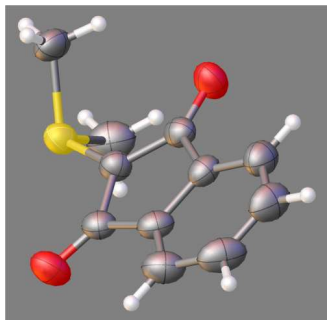


Importance of weak data

Comparison of tech

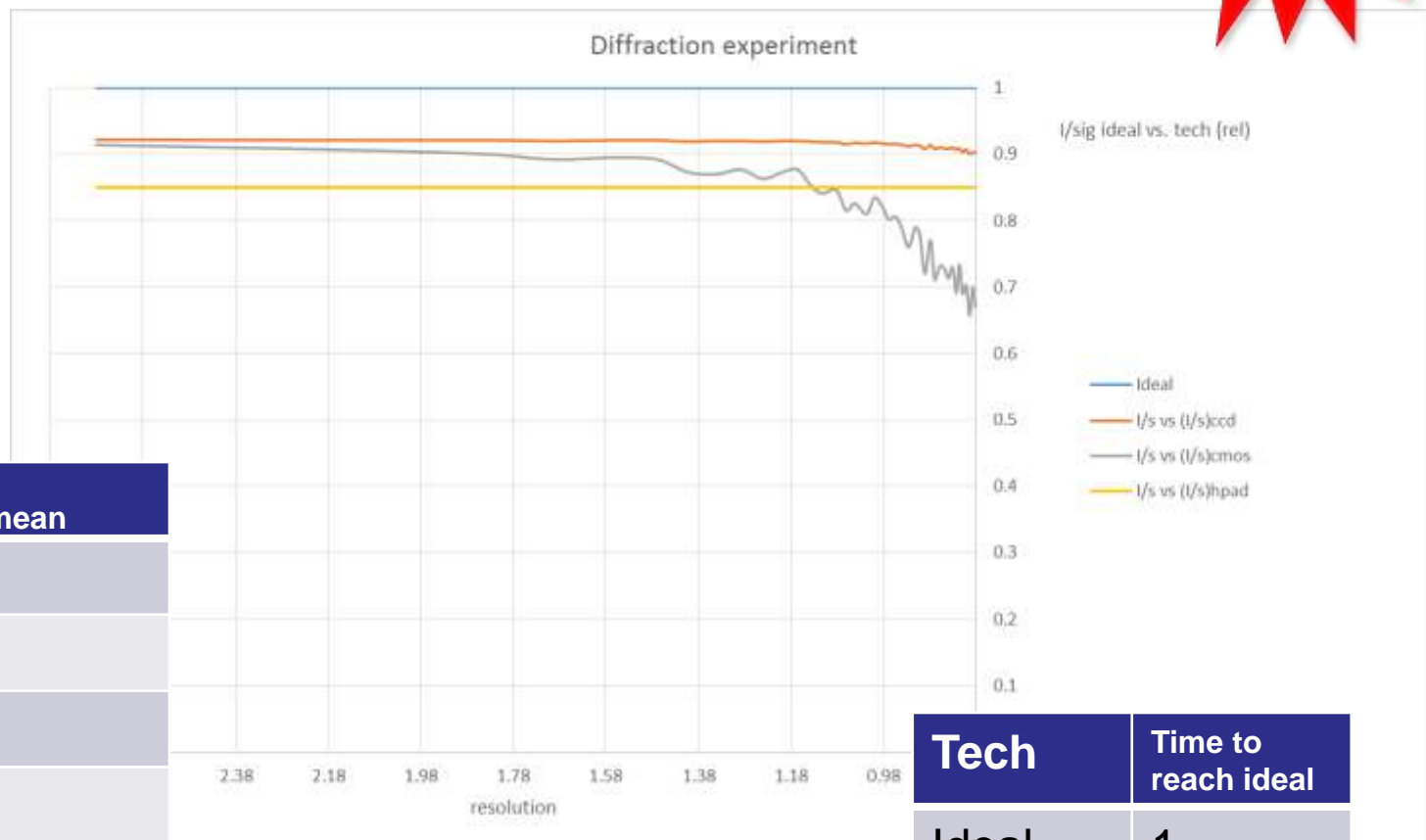


Tech	(I/sig) _{mean}
Ideal	15
CCD	13.8
CMOS	12.8
HPAD	12.7



Importance of weak data

Comparison of tech

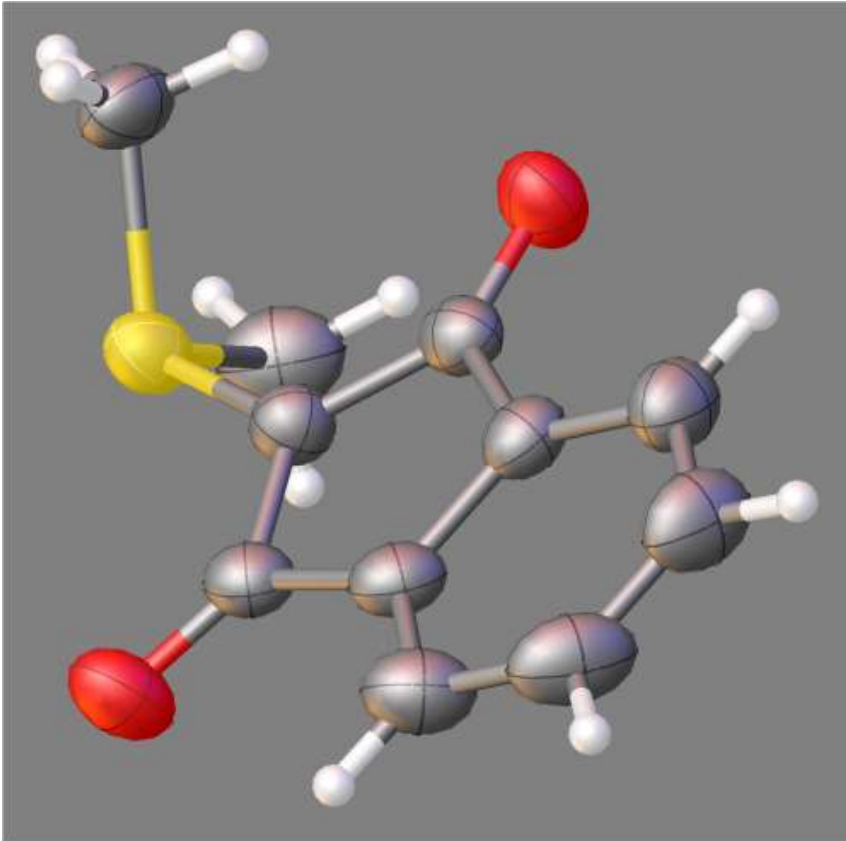


Tech	(I/sig) _{mean}
Ideal	15
CCD	13.8
CMOS	12.8
HPAD	12.7

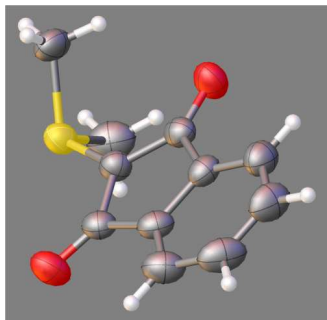
Tech	Time to reach ideal
Ideal	1
CCD	1.23
CMOS	2.22
HPAD	1.38

Importance of weak data

Charge density



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

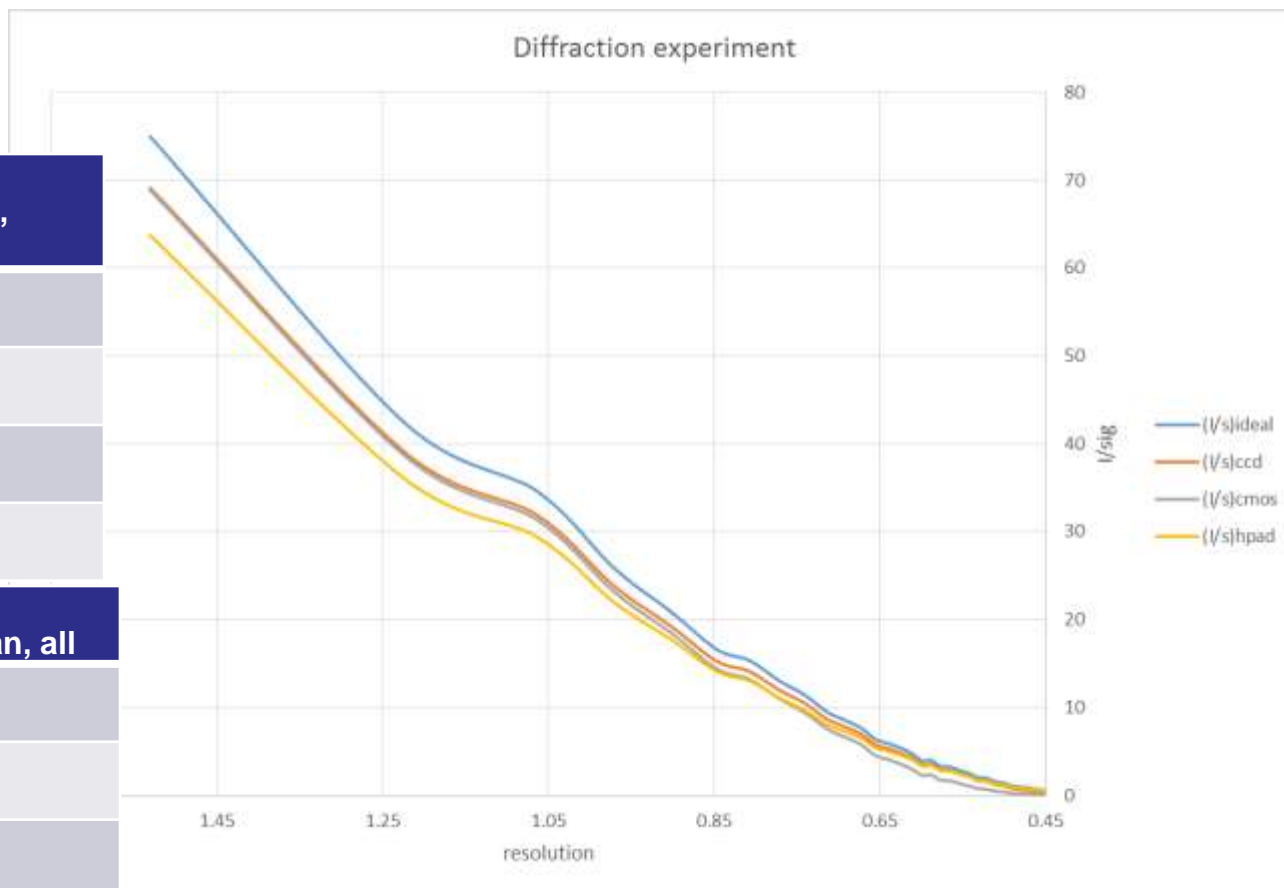


Importance of weak data

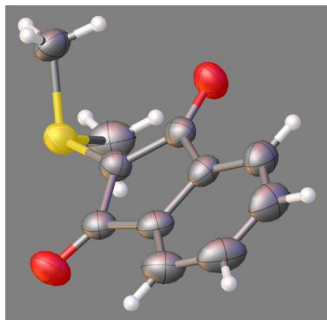
Comparison of tech



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

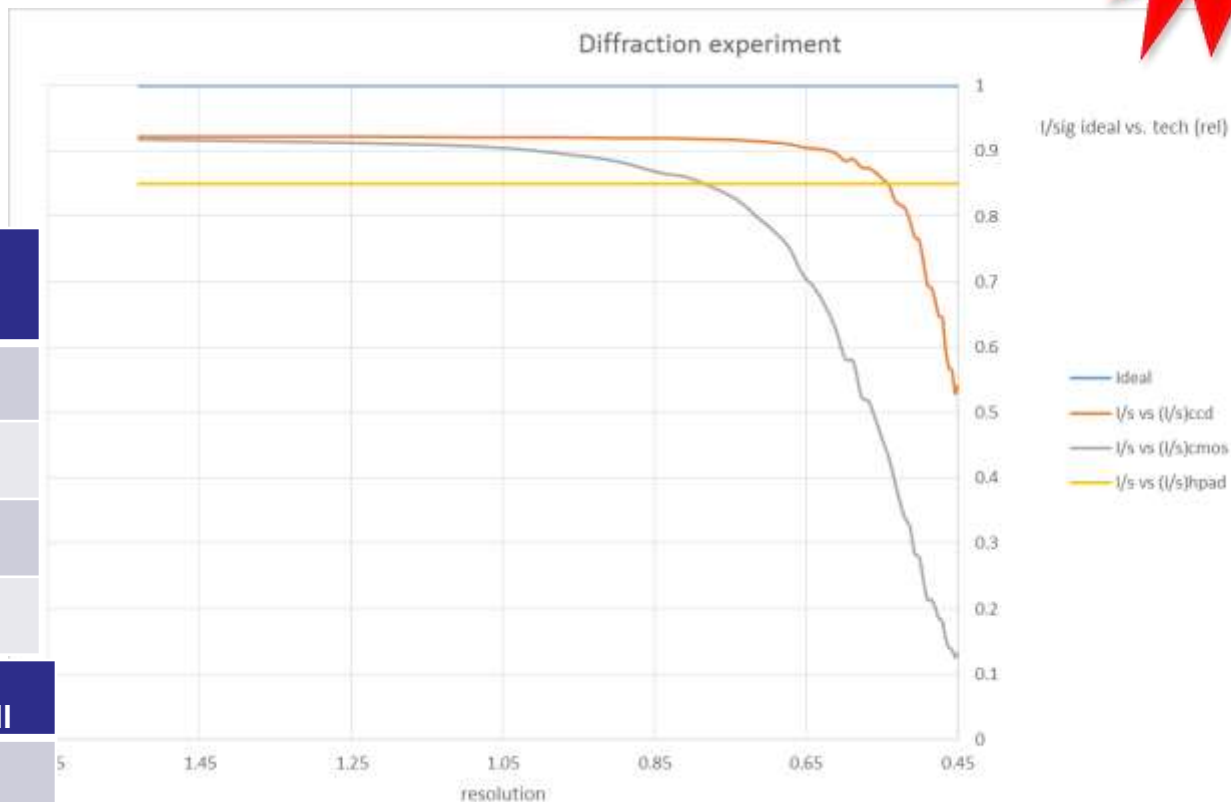


Tech	$(I/\sigma)_{\text{mean, all}}$
Ideal	8.6
CCD	7.8
CMOS	7.0
HPAD	7.3



Importance of weak data

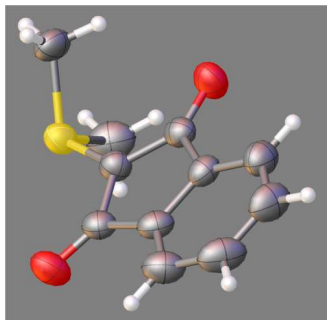
Comparison of tech



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



Importance of weak data

Conclusion



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

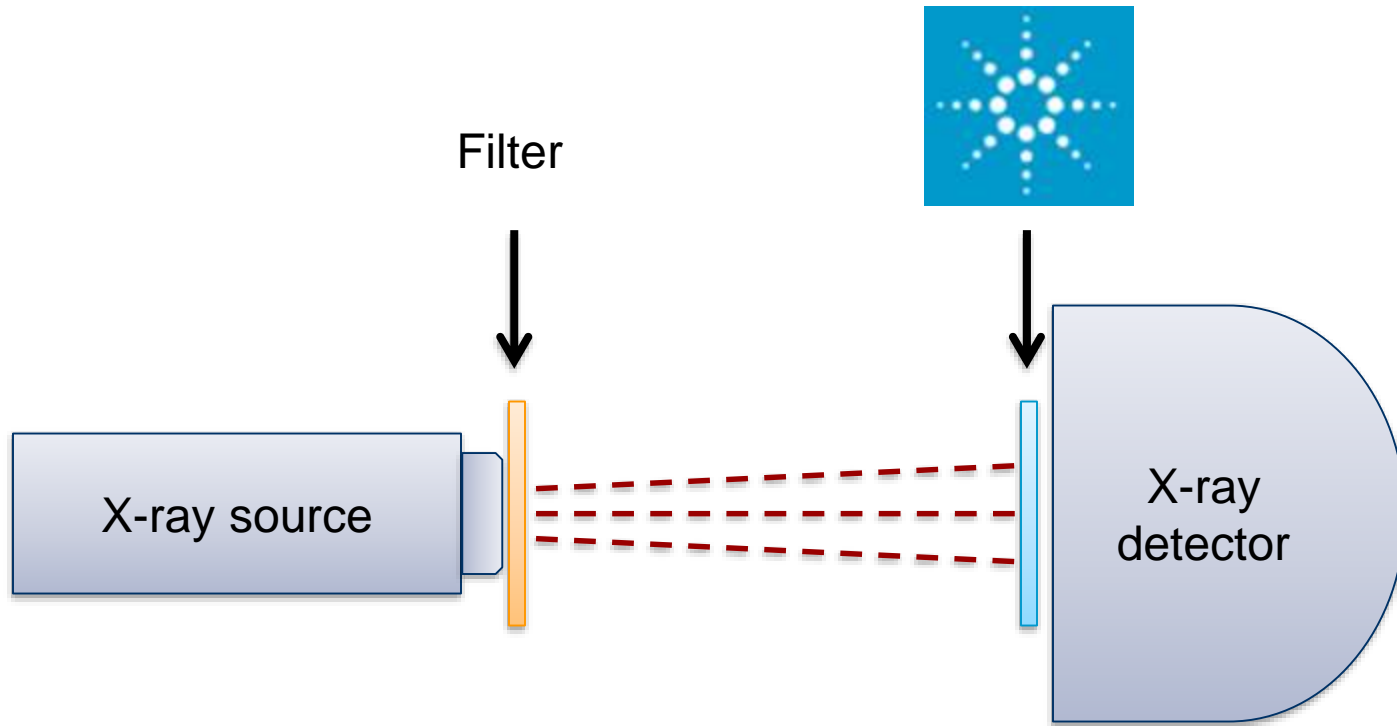


Feature	Atlas S2 CCD	APS CMOS (Oxford Diffraction R&D)
Active area, Taper	100x100mm, taper 2:1	100x100mm, taper 1:1
Gain [e-/MoK α]	180	261
Sensor	Truesense* Imaging CCD	Teledyne Dalsa RadEye 100 CMOS
Noise [MoK α -photons]	~0.05	~0.5

*Formerly Kodak

CCD vs. APS CMOS

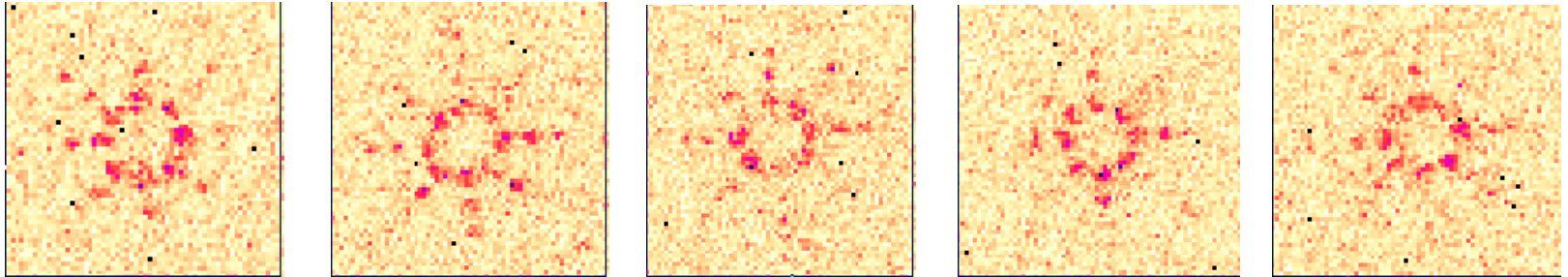
Comparative Detectivity Measurements



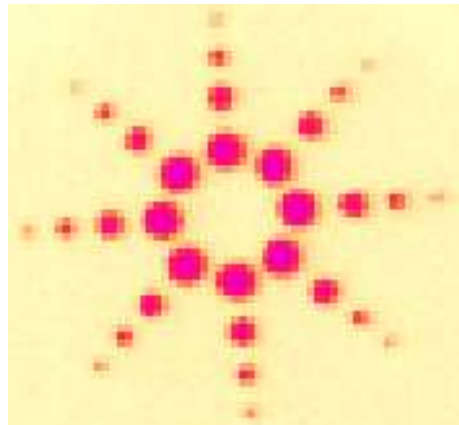
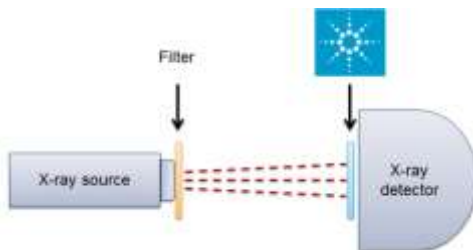
CCD vs. APS CMOS

Comparative Detectivity Measurements

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



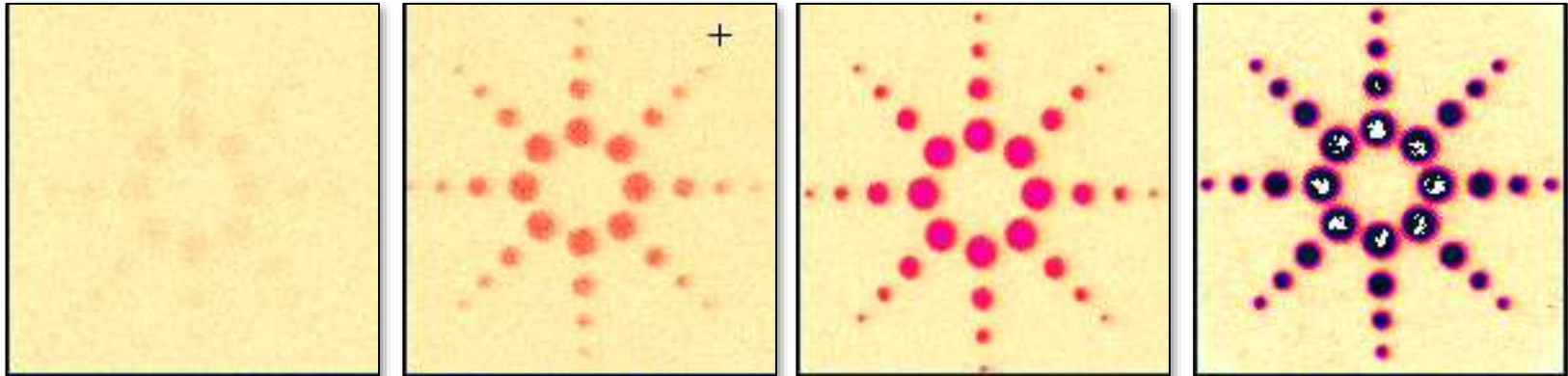
- In order to visualize signal-to-noise differences 100 images are averaged and scaled so that the noise level is the same for all modes of operation



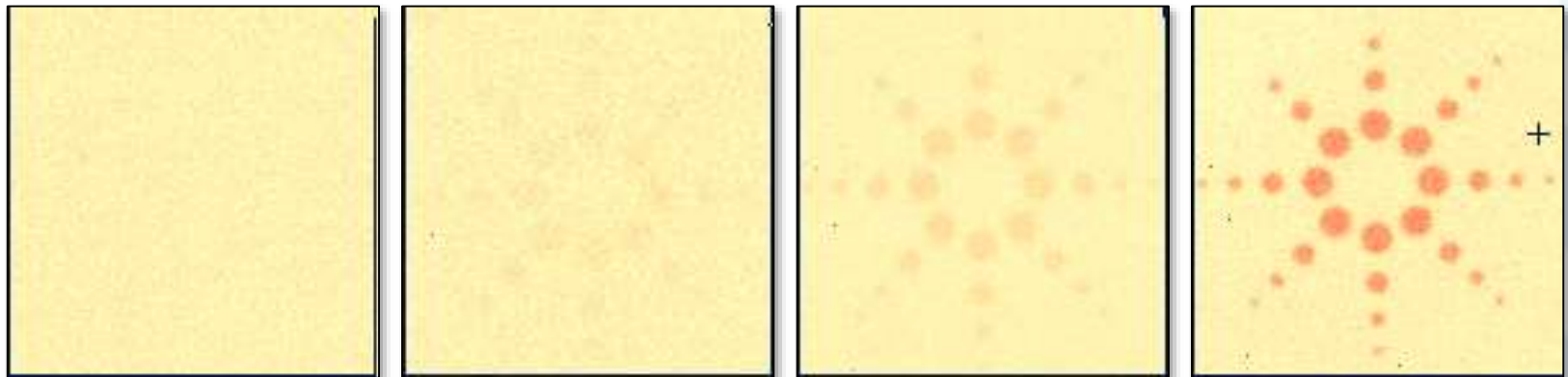
CCD vs. APS CMOS

Comparative Detectivity Measurements

Atlas
CCD



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



Exposure
time [s]

0.1

0.5

1

5

HW – Detector technology: Key metrics

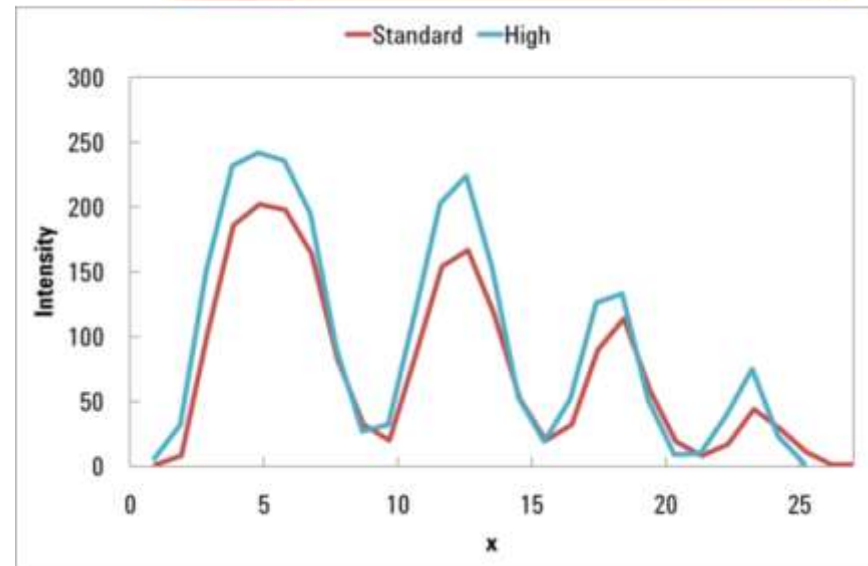
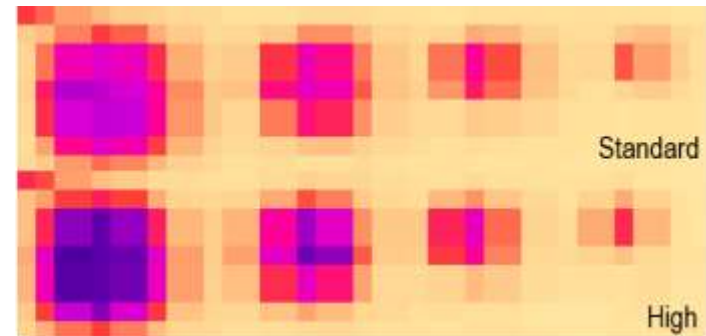


- Detectivity
- Dynamic range
- Speed
- Size
- Price

S2 CCD Detectors: INTELLIGENT MEASUREMENT SYSTEM

Smart Sensitivity Control (SSC)

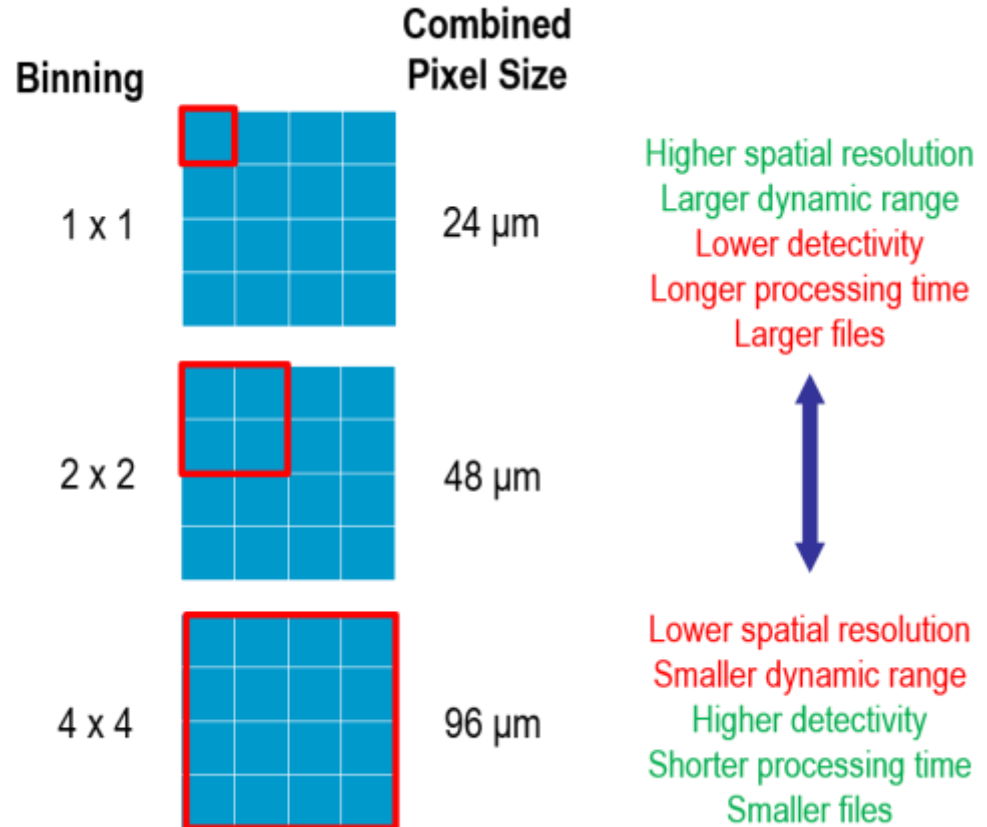
- **Self-optimizing** detector amplification based on strength of observed data (similar to ISO settings in digital photography)
- Standard, Medium and High SSC modes
- Maximises dynamic range for strong data
- Improvement in signal-to-noise for weak data
- A **unique** feature of Rigaku Oxford Diffractions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



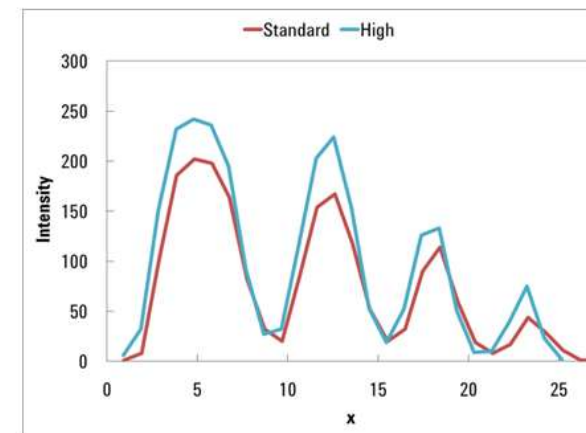
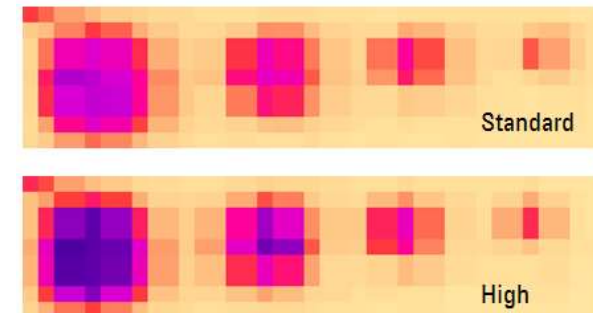
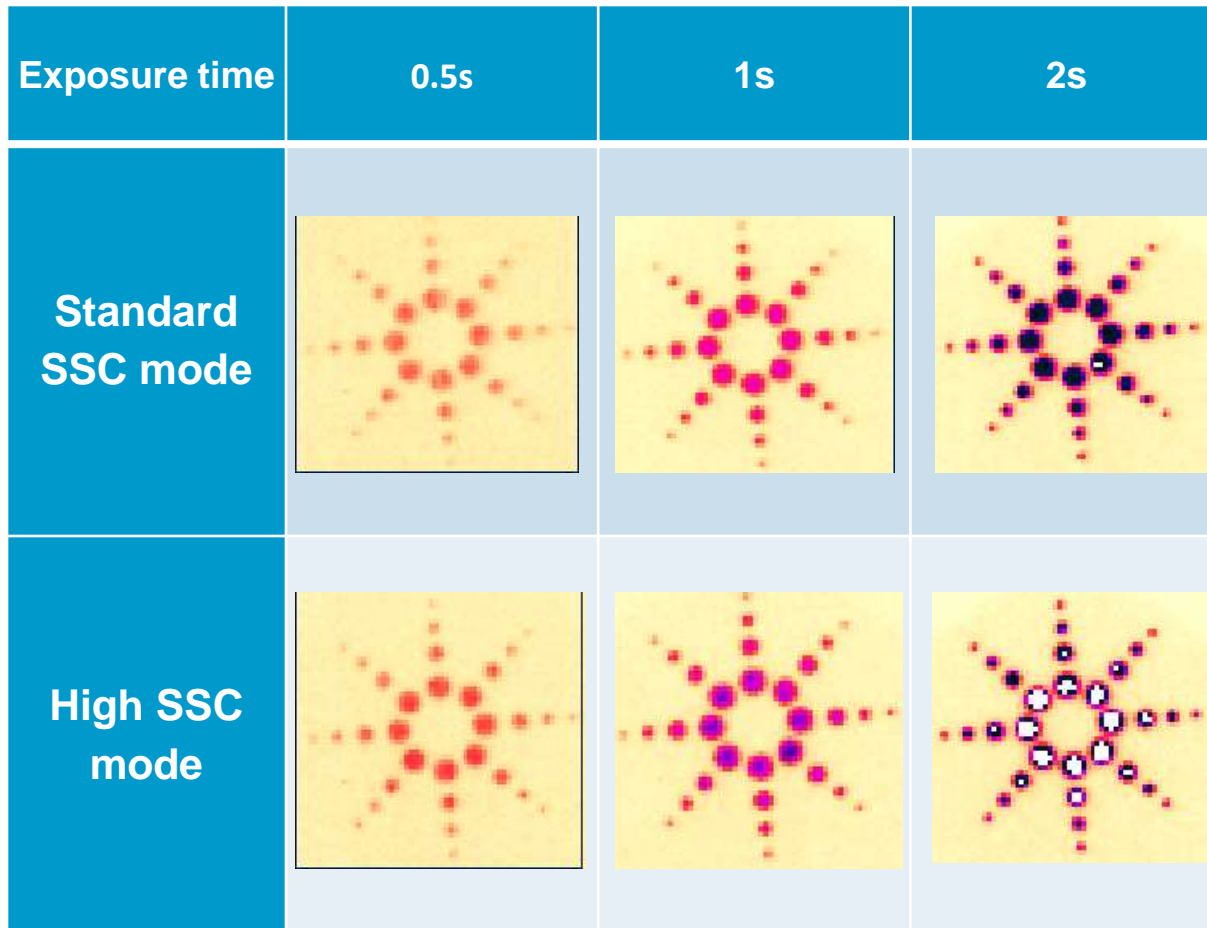
S2 CCD Detectors: INTELLIGENT MEASUREMENT SYSTEM

Instant-switching
hardware binning:


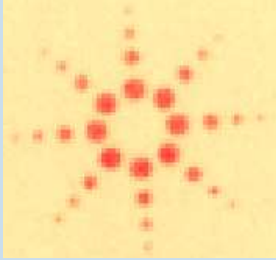
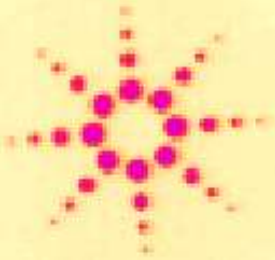
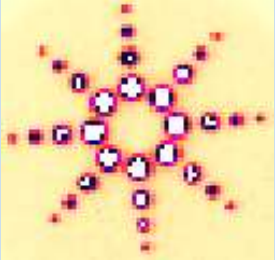
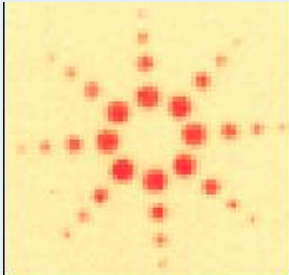
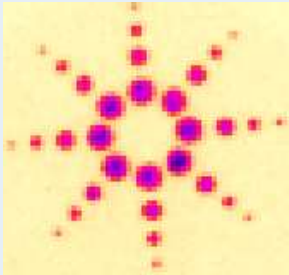
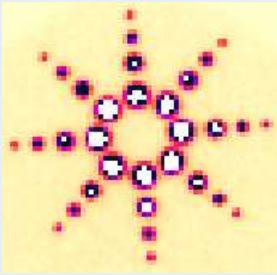
- **Adjustable** pixel sizes for variable resolution
- Flexibility in dynamic range
- Fast re-measurement of overflowed reflections
- Theta-dependent binning
- Automatic software switches binning modes **instantly**



S2 CCD Detectors: INTELLIGENT MEASUREMENT SYSTEM



S2 CCD Detectors: INTELLIGENT MEASUREMENT SYSTEM

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

S2 CCD Detectors: INTELLIGENT MEASUREMENT SYSTEM: IMS

Experiment Strategy (1.2.1), automode suggests exposure time t=2.0sec, scan width: 1.0 deg

Experiment Strategy

Unit cell for Strategy Calculation (CSD: 17-4439):
Cell: 5.969(5) 9.054(6) 18.381(18) 90.04(6) 90.04(7) 90.03(6) 993(1) oP

P-lattice 95.71% (87 of 70 reflections) [Lattice Wizard](#)

Strategy parameters

- Resolution Theta 2Theta
- Line group Other
- Friedel mates are equivalent (uncheck for high quality absolute configuration data)

Detector Distance [Advanced](#)

Strategy mode

Complete data (default mode)

limit [BICr limit](#) Max 99.89 %

Generates runs that reach completeness limit

Time prediction based on data to 0.837 Ang

- Fill time
- Fill 1/sigma
- The same time for all theta positions
- Different time for each theta position

theta	time	individual 1/sigma	merged 1/sigma	theta binning
[0.00]	2.00	75.66	75.66	del
[-42.50; 50.26]	2.35	25.48	32.29	del
[-42.50; 118.53]	2.25	6.38	8.80	del

Predicted resolution beyond 0.84
Scan width: Total 1/sigma:

Use theta-dependent binning

Manual experiment settings: [Depth to start](#) [Correlated frames](#) [AutoSuggest/Move Crystal](#)

Options: [AutoSuggest/Move Crystal](#)

Current Strategy

No. runs/frames: 12/553
Total experiment time: 0h 36m
Expected experiment finish time: Mon Oct 21 11:57:53 2013

[Calculate New Strategy](#) [Manually Edit Run List](#)
[Update Completeness](#)

Completeness/Coverage curves Completeness/Coverage tables

Completeness in mmm

Redundancy for completeness

Full sphere (P1)

Redundancy for coverage

help Start saved experiment Start experiment Cancel

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

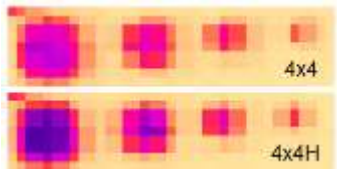
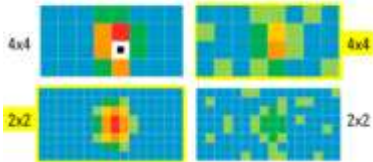
Strategy

Intelligent Measurement System – IMS for CCD



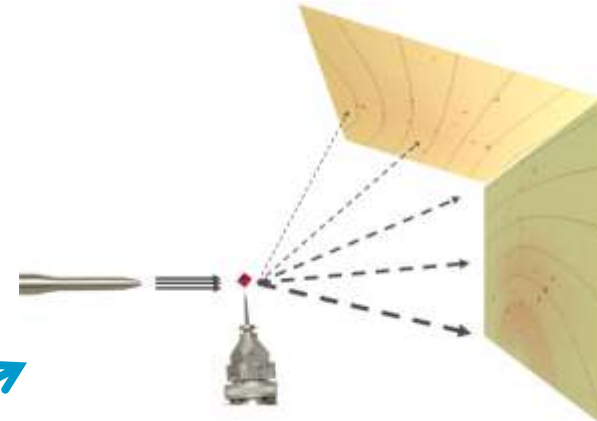
Base binning

Strong Weak



Smart sensitivity control

IMS



Theta dependent binning

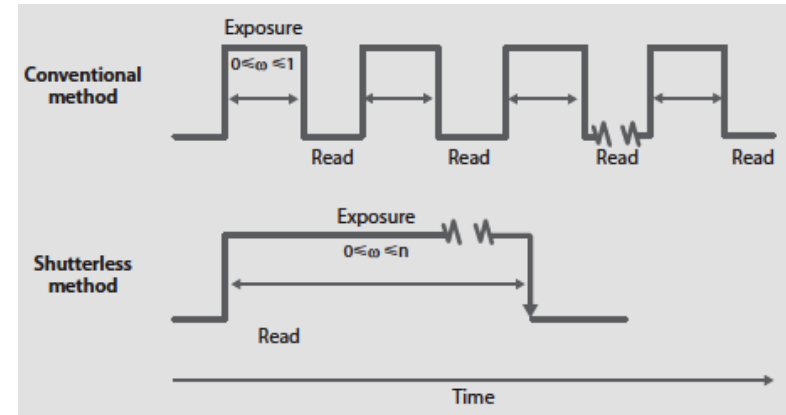


Gonio/scan settings correlation

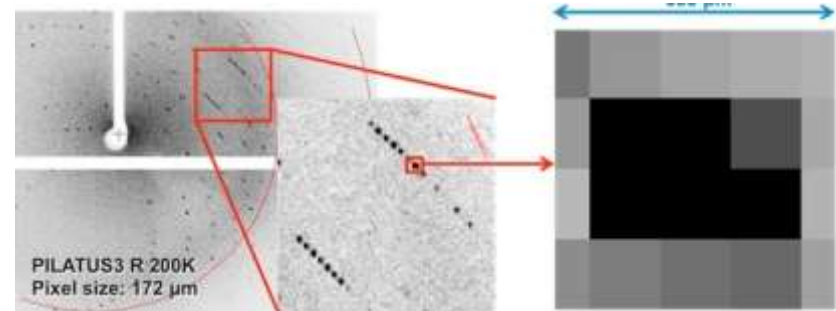
HPC – Hybrid Pixel Counters:



- 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



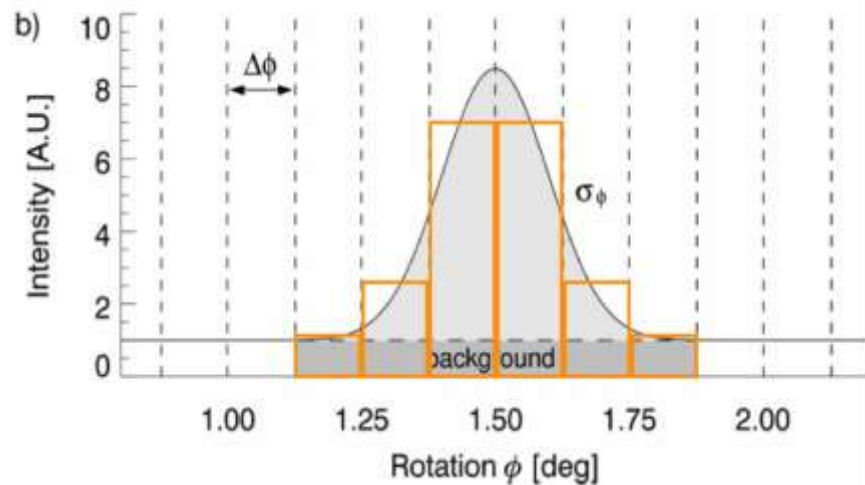
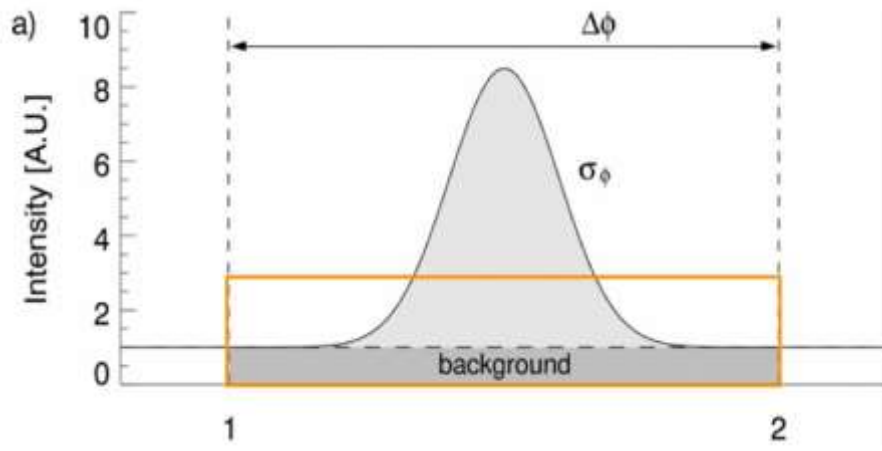
Shutterless data collection



Single pixel point spread function

Fine slicing and count rate correction

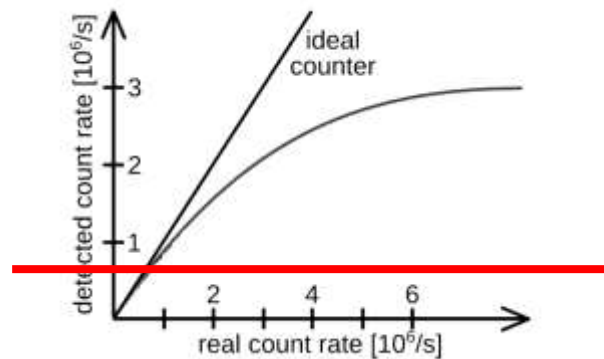
Wide slicing



Fine slicing

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

$$\text{Rate} = \text{counts/time}$$

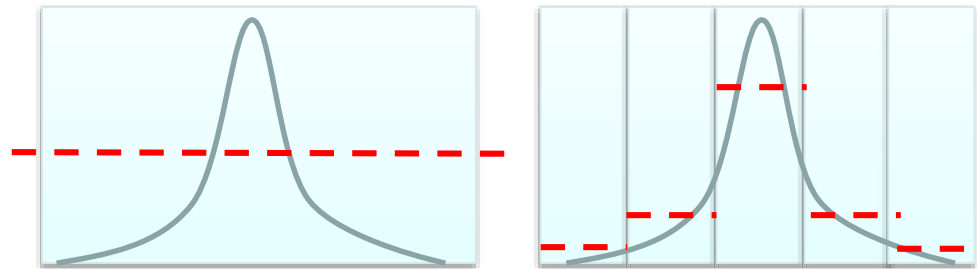


HPC – IMS: Feature



Feature:

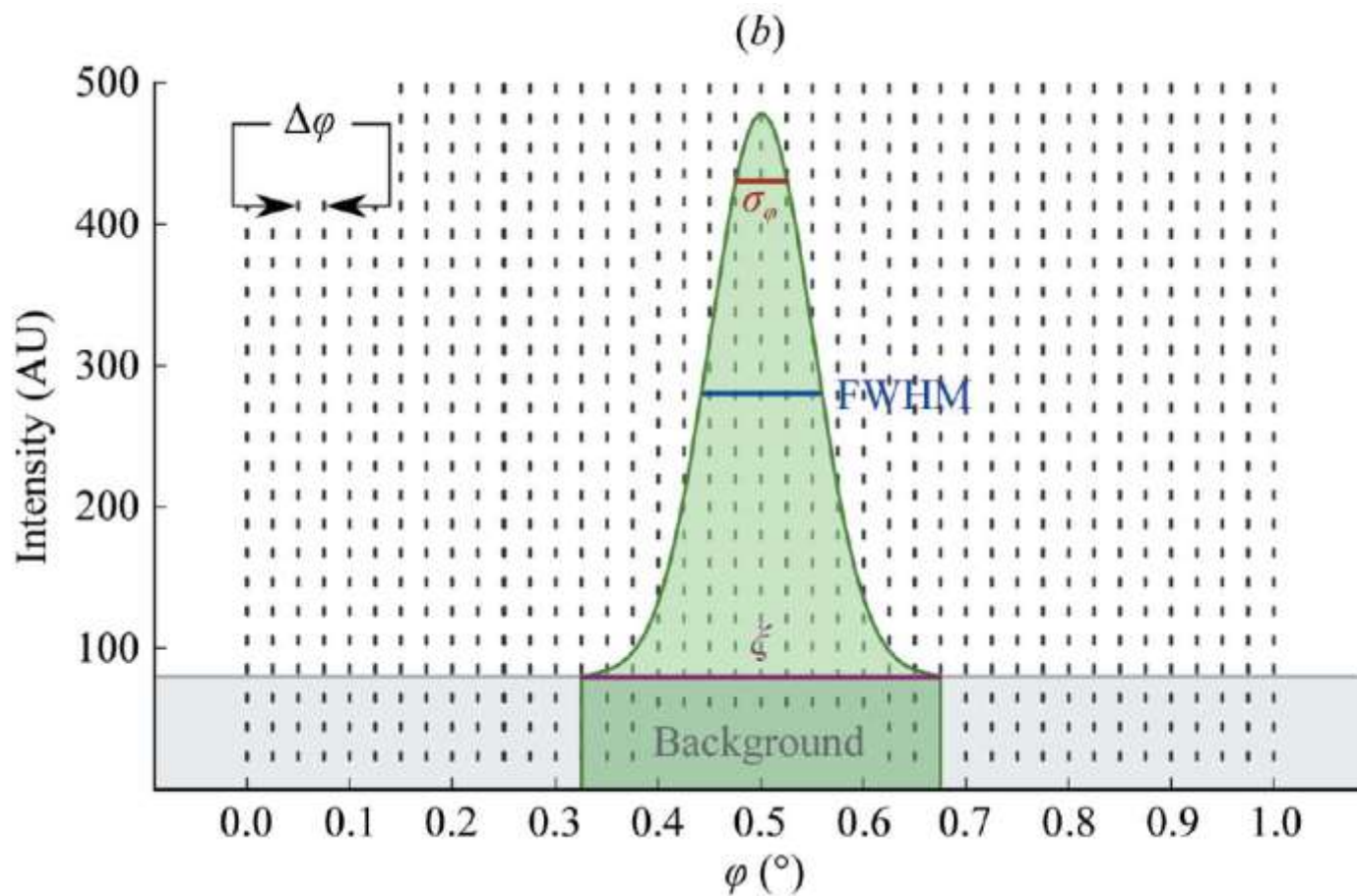
- Strong reflections may be affected by coincidence-loss (dead time correction): rates $> 400\text{k/pix.s}$
- Fine-slicing may be required for more accurate count-rate correction



- 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

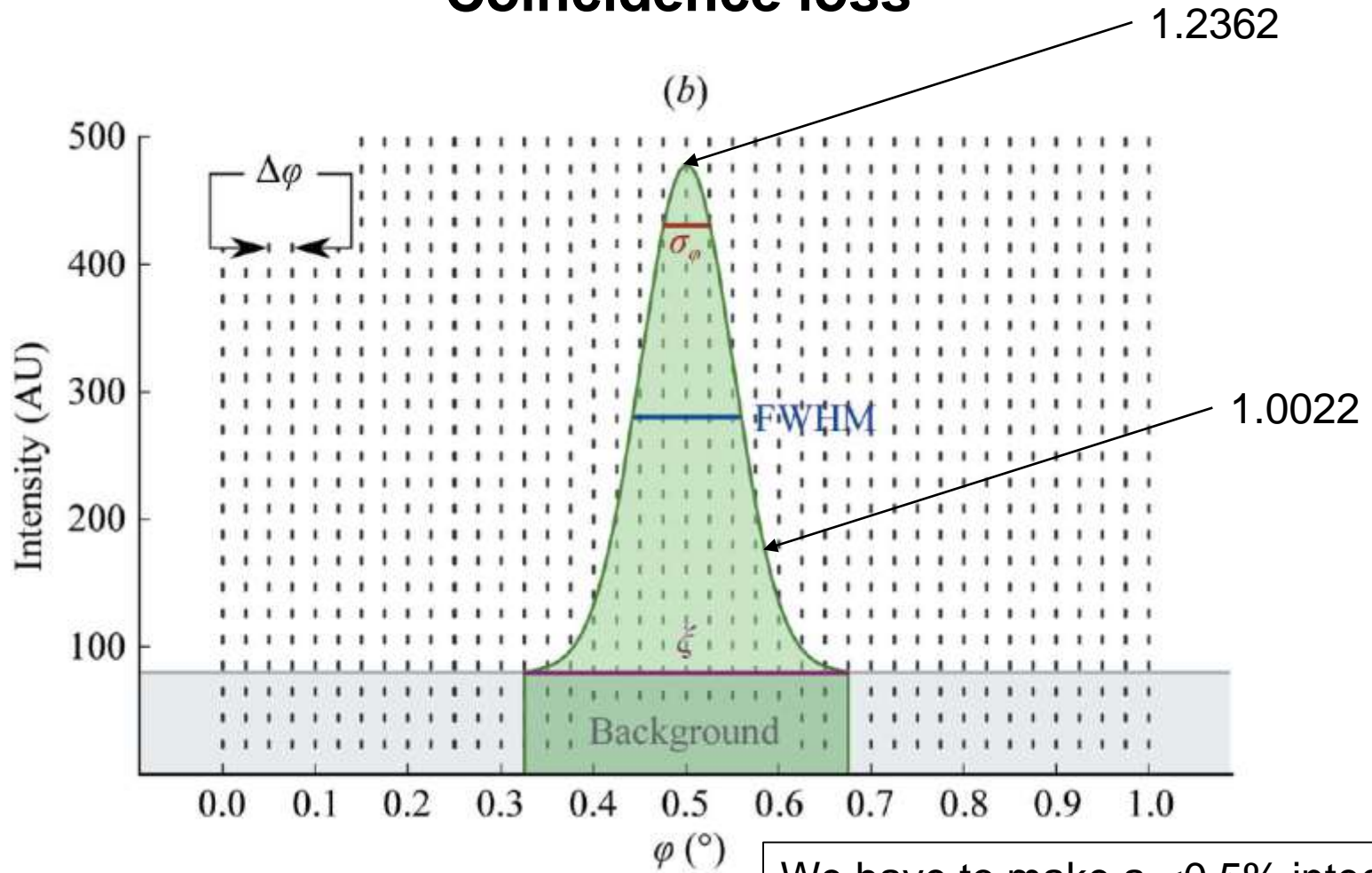
Data quality and count rates

Coincidence loss



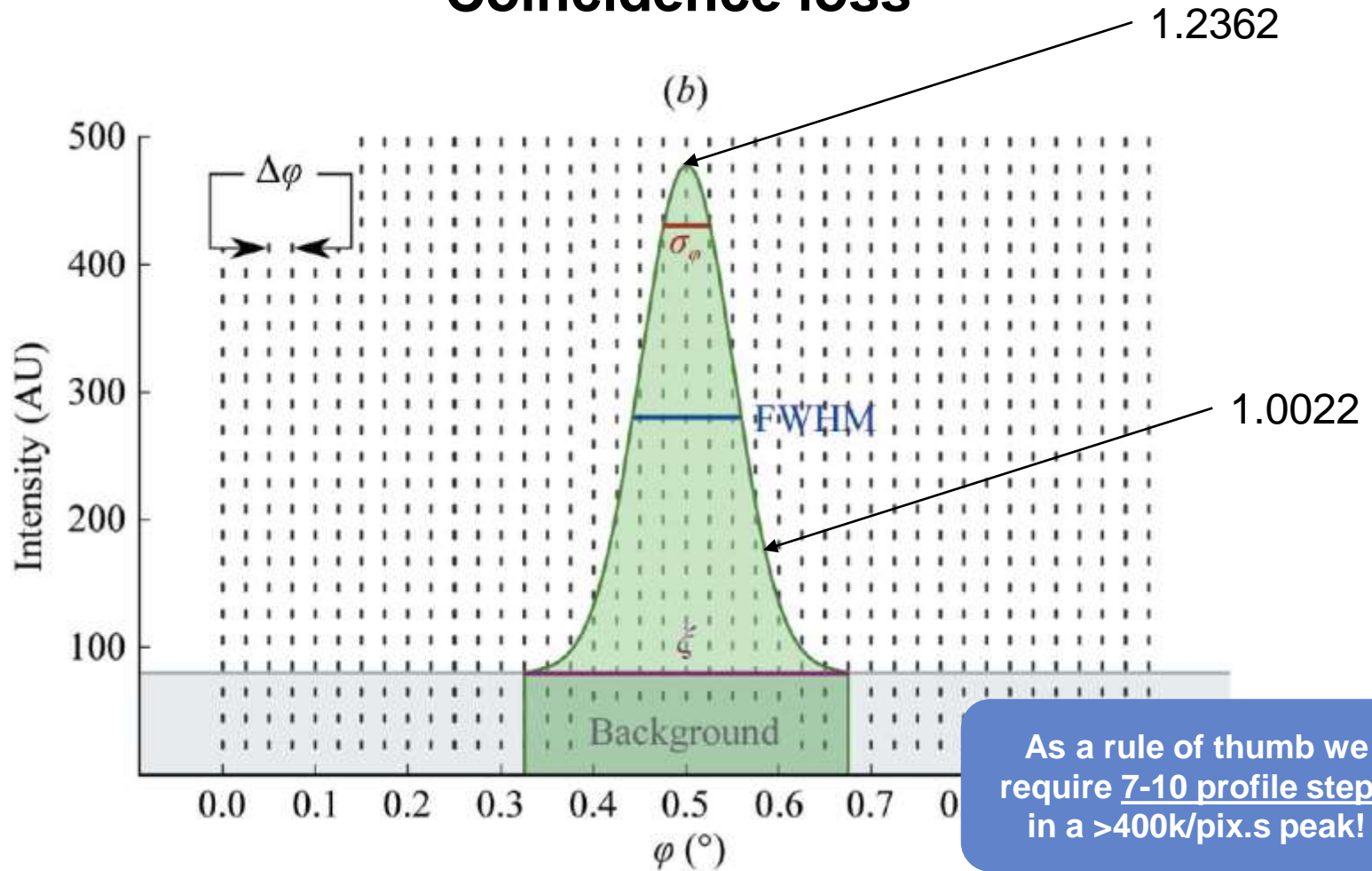
Data quality and count rates

Coincidence loss

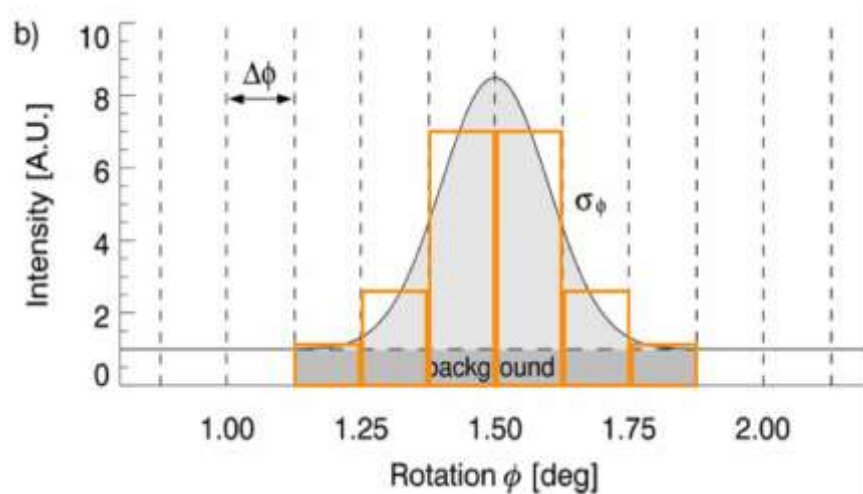


Data quality and count rates

Coincidence loss



Fine slicing and count rate correction

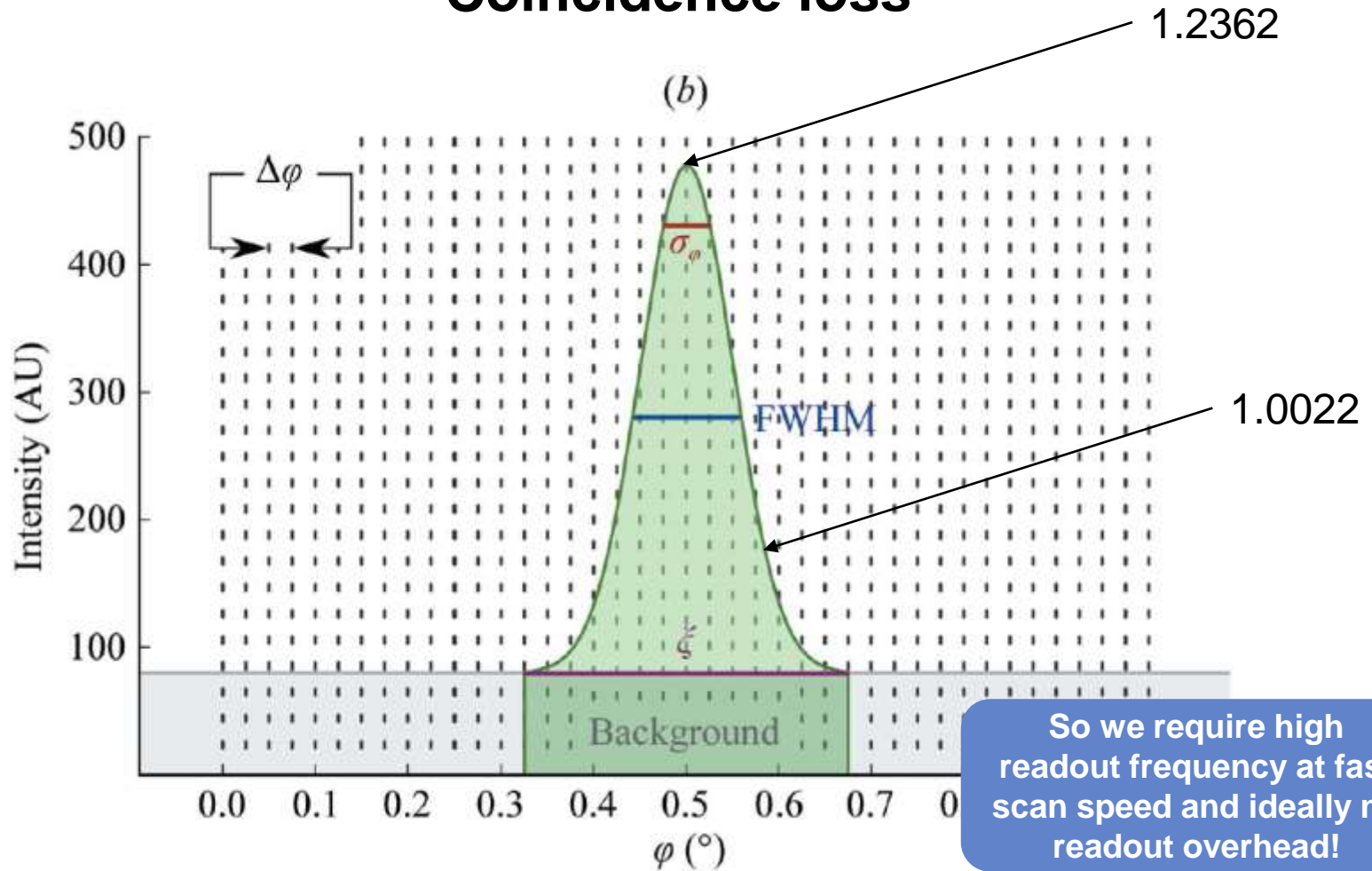


Rate = counts/time

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

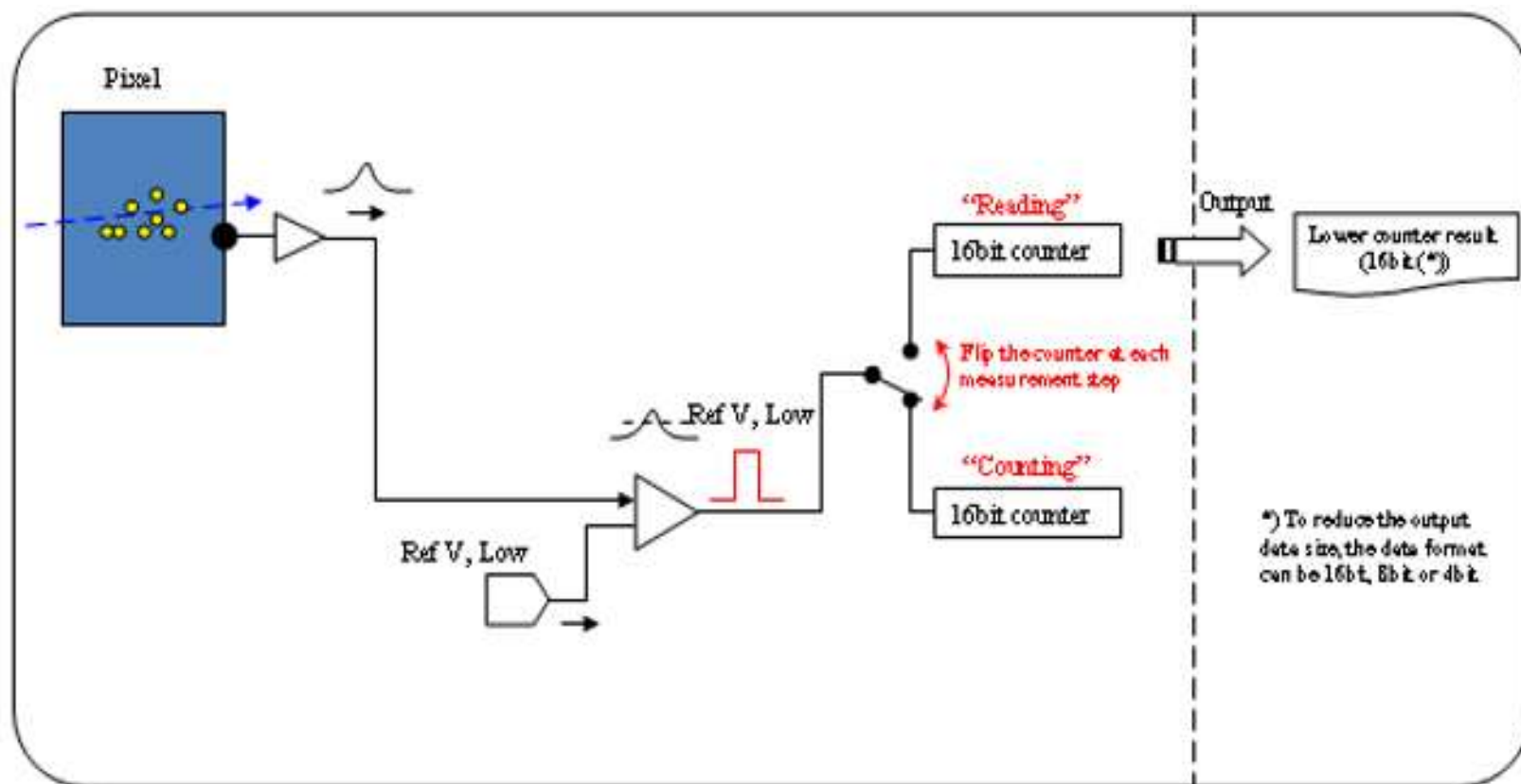
Data quality and count rates

Coincidence loss



New Hybrid Photon Counting Detector

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



HPC – Hybrid Pixel Counters: IMS



Experiment Strategy (1.3.0), autmode suggests exposure time t=0.5sec, scan width: 0.30 deg

Experiment Strategy CRYALIS™

Unit cell for Strategy Calculator: (200.10, 20.1)

Cell: 5.9663(17) 9.851(3) 11.375(4) 89.98(2) 89.97(2) 89.97(3) 992.2(5) °P

P-lattice 100.00% (159 of 159 reflections) Lattice Wizard

Strategy parameters

- Resolution Theta ZTheta 0.837
- Lawe group: Other: mmm
- Friedel mates are equivalent (uncheck for high quality absolute configuration data)
- Detector Distance: 50.00 Advanced

Strategy mode

Complete data (default mode)

limit: 100.0 HCy limit Max 99.89 %

Generates runs that reach completeness limit

Time prediction based on data to 0.837 Ang

Resolution	exp time	individual I/sigma	merged I/sigma
0.30	0.30	144.15	232.30
[-38.89; 38.89]	0.30	327.30	426.38
[-98.50; 105.29]	0.30	83.07	147.04

Predicted resolution beyond 0.84

Scan width: 0.30

Total I/sigma: 144.15 232.30

Repeat pre-experiment with longer exposure time as current time prediction can be inaccurate.

Automatic experiment settings

Mode: not supported Frequency: 20.0Hz Autochem/Mouse/Cryolink

Options

Current Strategy

No. runs/frames: 9/1092

Total experiment time: 0h 11m

Expected experiment finish time: Thu Jun 16 16:18:24 2016

Calculate New Strategy Manually Edit Run List

Update Completeness

Completeness/Coverage curves Completeness/Coverage tables

Completeness in mmm

Redundancy for completeness

Full sphere (P1)

Redundancy for coverage

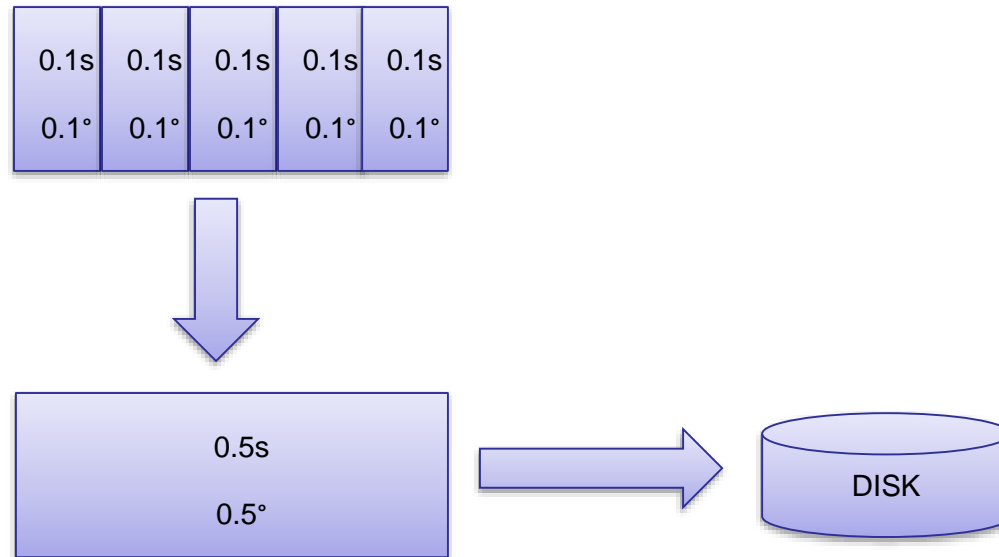
Help Start named experiment Start experiment Cancel

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

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

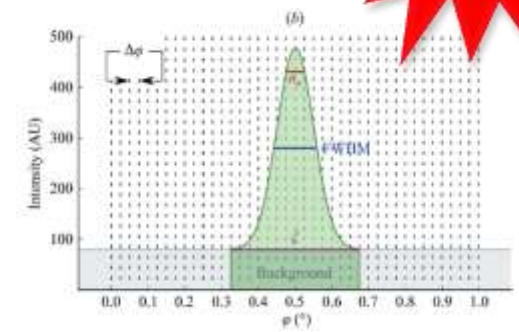
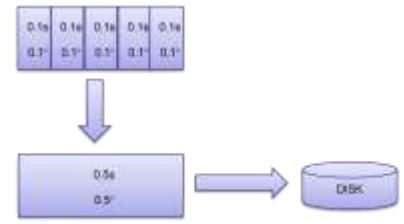


Strategy

Intelligent Measurement System – IMS for HPC

New
39

Base scan width



IMS
HPAD

Rate limits
Counter limits

HPAD op mode

Generator setting

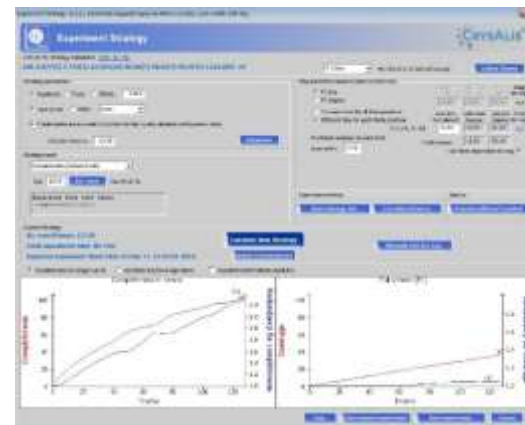


Advances from where?



HW – instrumentation

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



Procedure



Data reduction
Corrections

HW – Sources

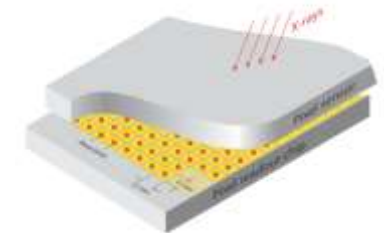
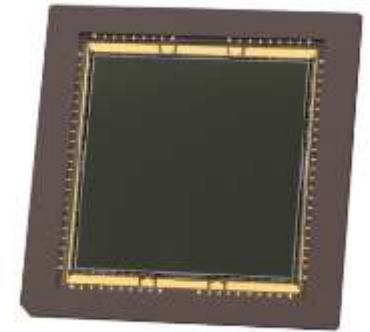
Same sample 0.3mm, normal tubes (2kW, 0.5mm collimator), micro-focus (50W), 007HF (1200W)



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

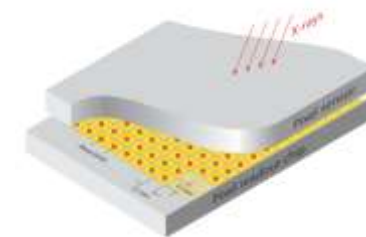
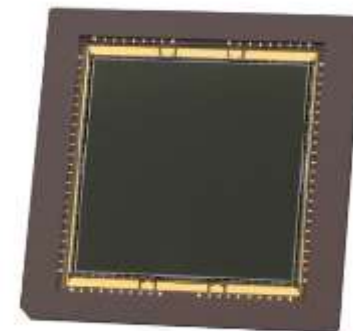
HW – Fullwell/Dynamic

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



HW – Detector Size

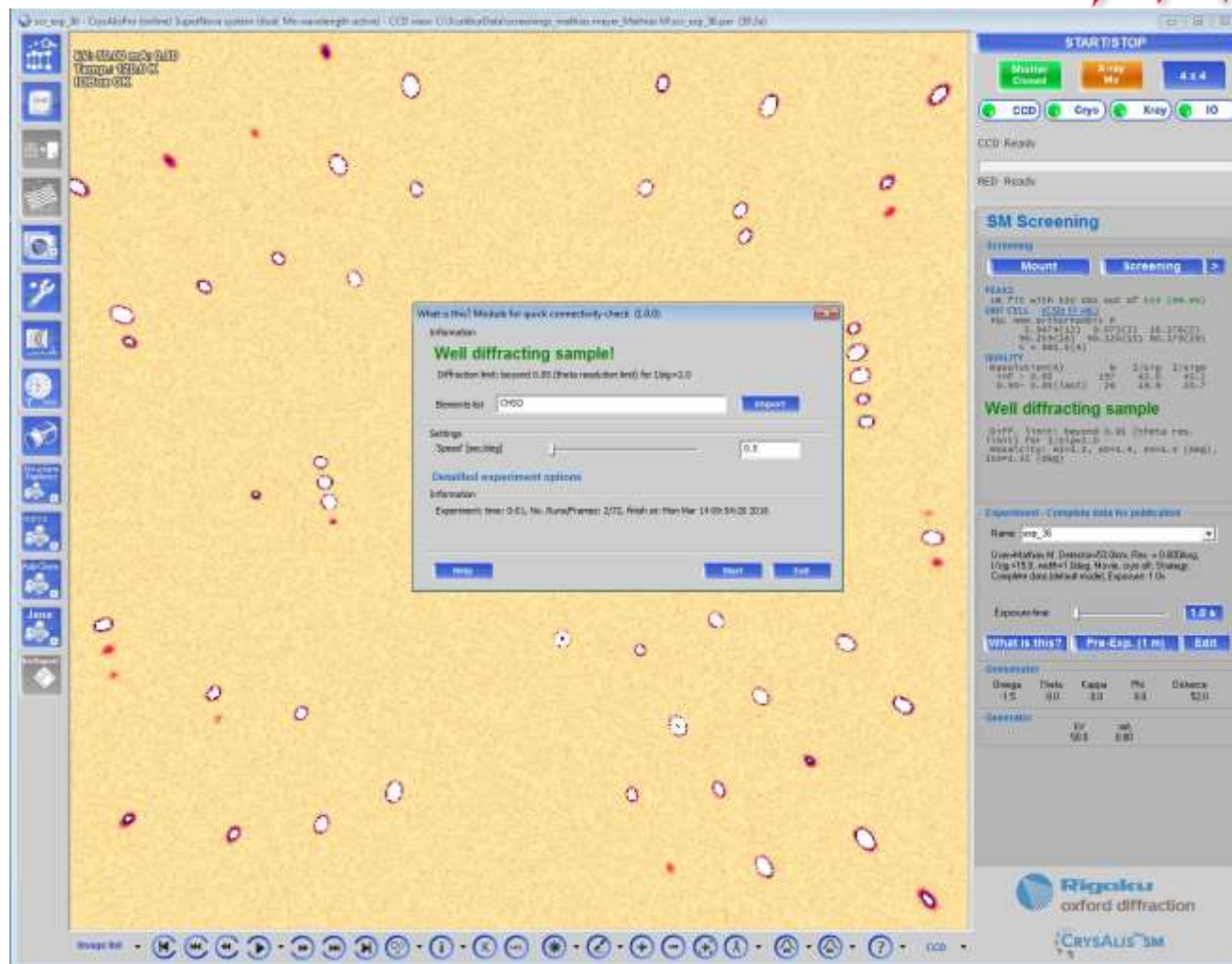


Detector relative size	Unique speed	Observation speed
Eos 1	1	1
Atlas 2.4	1.3-1.6	1.6-1.8
Titan 3.7	1.4-1.8	2.0-2.2

‘What is this?’ tool



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



What is this?' tool: 70s later...

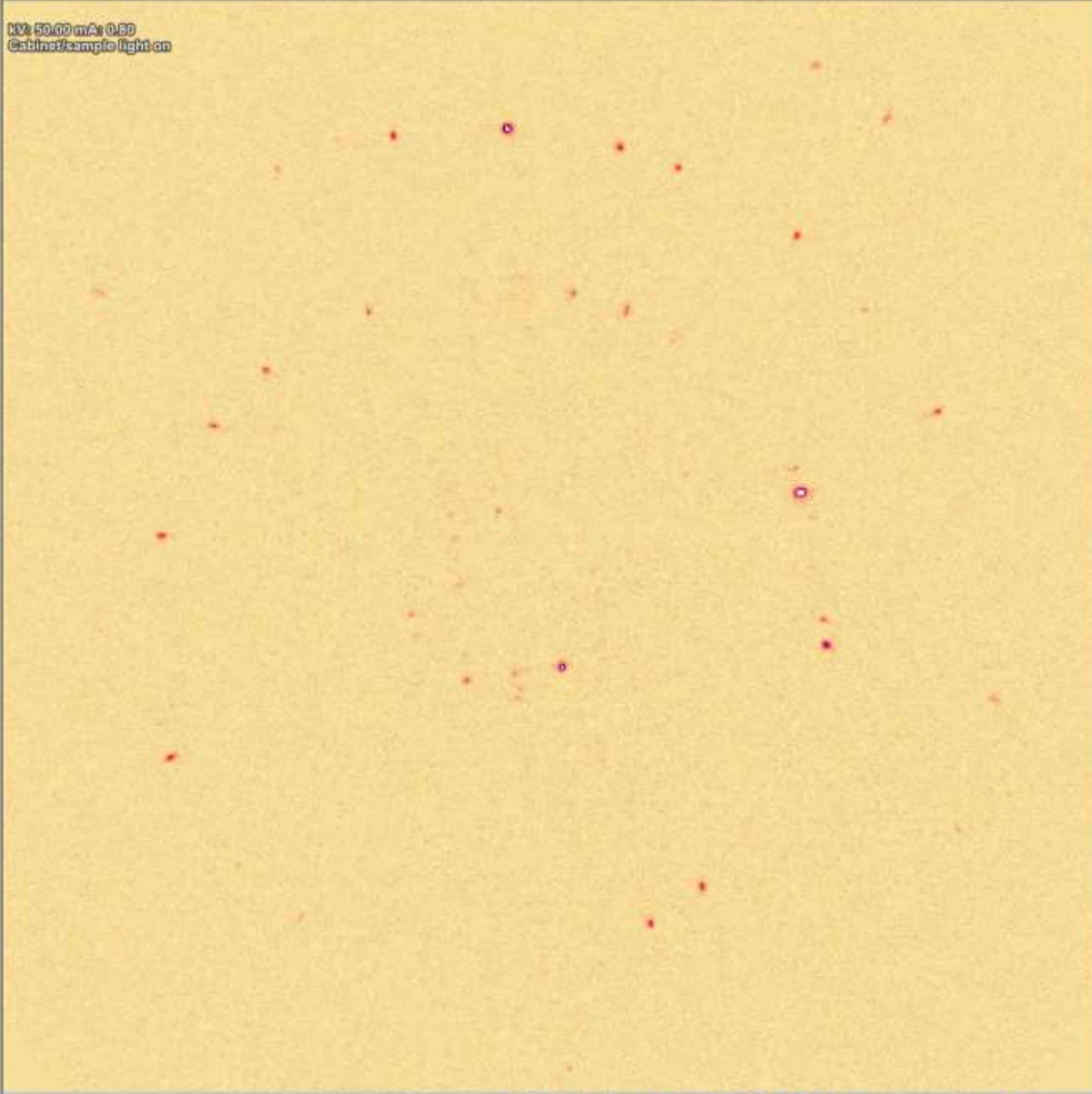
New
38

The screenshot displays the CrysAlis Pro software interface. The main window shows a diffraction pattern with several spots highlighted in red and white. The interface includes a top menu bar, a left sidebar with various tool icons, and a right sidebar with control panels. The right sidebar contains a 'START/STOP' section with buttons for 'Shutter Closed', 'Start Run', and '4 x 4'. Below this are status indicators for 'CCD', 'Cryo', 'Kray', and 'IO'. The 'Crystal' section shows 'CCD' and 'Data Collection' buttons. The 'Data Reduction' and 'AutoChem' sections are also visible. The 'AutoChem' section includes a 'Olex2' button and a 'Restart AutoChem' button. A 3D ball-and-stick model of a molecule is shown in the 'AutoChem' section. Below the model is a 'SCHEMATIC STATISTICS' table with the following data:

SCHEMATIC STATISTICS	
Chemical Formula:	C ₁₄ H ₁₀ O ₂ S
Space Group:	P2 ₁ (1)2 ₁ (1) #14
Formula weight:	226.26
Z:	2.000
Vol:	4.076
D _{calc} :	1.38
Absorption:	0.238 mm ⁻¹
Comp. ID (R1A):	19.76 (Paw)
Atom sum of Z:	14.00 (P222)
Peak and Hole:	0.127 and -0.018
Flack:	-0.000

Below the statistics is the 'STRUCTURE FILE' section, which shows the filename: 'P197a01.cif'. The bottom of the interface features a toolbar with various navigation and control icons. The Rigaku oxford diffraction logo is visible in the bottom right corner of the software window.

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 >

REMARKS
UB FIT with 0 obs out of 0 (0.0%)
UNIT CELL C50H42N10
PG: mm orthorhombic P
a = 8.34(4) b = 8.030(18) c = 18.33(2)
alpha = 90.19(12) beta = 90.4(2) gamma = 90.7(3)
V = 583(7)
QUALITY
Resolution(A) N I/sig I/s190
1/117 = 1.10 7 5.9 34.0
0.00- 0.00(1ast) 0 0.0 0.0

Well diffracting sample
Mosaicity: $\omega=0.3$, $\alpha=1.0$, $\beta=0.3$ (deg), $I_{000}=0.92$ (deg)

Experiment - Complete data for publication

Name: exp_1755

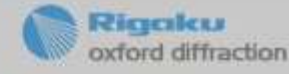
User: ps, Detector: 53.0mm, Res = 0.600Ang, I/sig=40.0, width=1.0deg, Movie, cryo-off, Strategy: Complete data (default mode), Exposure: 2.5s

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



What is this?' tool: Connectivity solved! <70s

New
38

Wt_Exp_38 - CrysAlis Pro (online) SuperNova system (Sat, Mo wavelength: actia) - CCD view: C:\XtalData\Mathia_Pt_scm\exp_38\wt_exp_38.p4

Wt 3000 mA; 2.00
Temp: 110.0 K
IP: 20.0 kV

START/STOP
Shutter Closed 3.00 Min 4 x 4
CCD Crys Kray IO
CCD Ready
RED Ready

Crystal
Data Collection
Data Reduction
AutoChem
Olex2 Restart AutoChem

C1=CC=C(C=C1)S(=O)(=O)C2=CC=CC=C2

WILSON STATISTICS
Chemical Formula: C₈H₈O₂S
Space Group: P2₁(21)21 #19
Formula weight: 160.16
Z: 2.000
V_{cell}: 4.076
GOOF: 1.08
Absorption: 0.285 mm⁻¹
Comp. ID: 81A) 59.76 (Paw)
Atom sum: 14.86 (P22)
Peak and Hole: 0.127 and -0.018
Flack: -0.000

STRUCTURE FILE
Filename: wt_exp_38

Wavelength: 0.71073 Å
Omega: -4.50 Theta: 0.00 Kappa: 0.00 Phi: 0.00 Distance: 610.0
GONIOMETER
Omega: -4.50 Theta: 0.00 Kappa: 0.00 Phi: 0.00 Distance: 610.0

WtExp_38

Rigaku oxford diffraction
CRYALIS™
3.8

XtaLAB Synergy: PhotonJet R, HyPix6000HE



XtaLAB Synergy and HyPix6000HE: *100 μ m pixel, 100Hz operation, 10deg/s scans*

The screenshot displays the XtaLAB Synergy control software interface. The main window is divided into several sections:

- Top Left:** Status information: `kV: 40.00 mA: 30.00` and `Interlock open (HV). Please press 'HV ON/Start' button, Sample light on`.
- Center:** A large blue area showing a diffraction pattern with several bright spots. To the right of this area is a circular goniometer dial with a scale from 0 to 12 and a digital display showing `00:00:00`.
- Right Panel:** A control panel with a **START/STOP** section containing a green **Shutter Closed** button. Below this are status indicators for **CAM**, **CRYO**, **X-RAY**, and **STATUS**. Further down, it shows `CCD Ready` and `RED Ready`. A **Crystal** section displays experiment details: `EXPERIMENT: wit_exp_158`, `CHEMICAL FORMULA: C12H10S2`, `LATTICE: Current cell: C2/c1`, and `UNIT CELL: a: 1.9822 b: 3.0923 c: 14.9177`, `alpha: 90.1187 beta: 90.1 gamma: 90.0181`, and `V = 1000.0881`. A `REMARKS` section notes: `BB FOC with 0 cm out of 0 (TOTAL) 0.35% (med) 0.00%`.
- Bottom Right:** A vertical menu with `Data Collection`, `Data Reduction`, and `AutoChem` options. The **Rigaku oxford diffraction** logo is visible at the bottom right of this panel.
- Bottom Left:** A toolbar with various icons for image processing and navigation.
- Bottom Center:** `IMAGE: wit_exp_158_2_72_rodhypix (run: 2 frame: 72)`, `Omega: 52.72 Theta: 55.47 Kappa: 0.00 Phi: 184.00 Distance: 32.50`, and `GONIOMETER: Omega: 0.00 Theta: 35.00 Kappa: 0.00 Phi: 0.00 Distance: 32.50`.

Synergy: XtaLAB Synergy

New
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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.



Synergy: XtaLAB Synergy

New
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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.

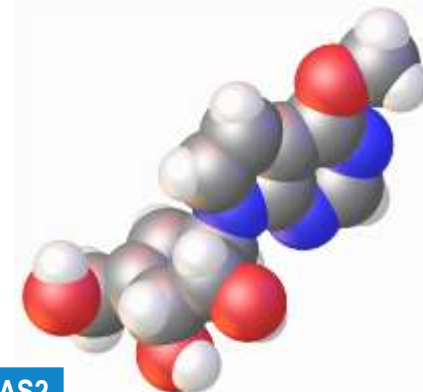
Unique telescoping 2 θ arm provides total flexibility for your diffraction experiment.

Enhanced kappa goniometer design with symmetrical 2 θ positioning

Synergy: XtaLAB Synergy

New
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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

Synergy: XtaLAB Synergy



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!

Find out more at

www.rigaku.com

Support of Rigaku instruments via CAP

Step 2 - Choose platform

Machine: XtaLAB PRO Kappa

Source: n/a

Controller: CrysAlisPro

Detector: n/a

Goniometer: MM007 Cu, MM007 Mo, MM007DW CuMo, MM003 Cu (home) SST Mo Fine (offset), MM003 Cu (home) MM003 Mo (offset), MM003 Ag (home) SST Mo Fine (offset), MM003 Cu (home), MM003 Mo (offset)

Si layer: n/a

Instruction Previous step



Step 2 - Choose platform

Machine: XtaLAB mini

Source: SST Mo 600W

Controller: CrysAlisPro

Detector: MERCURY3

Goniometer: n/a

Instruction Previous step Next step

