DECTRIS®

detecting the future

MYTHEN PILATUS EIGER

Les détecteurs HPC pour la crystallo sous conditions extrêmes

Andreas Förster – Application Scientist Crystallography

ANF Яéciprocs – 2018-06-06

DECTRIS Ltd.
5405 Baden-Dättwil
Switzerland
www.dectris.com

Outline

Technology

Direct detection

Photon counting

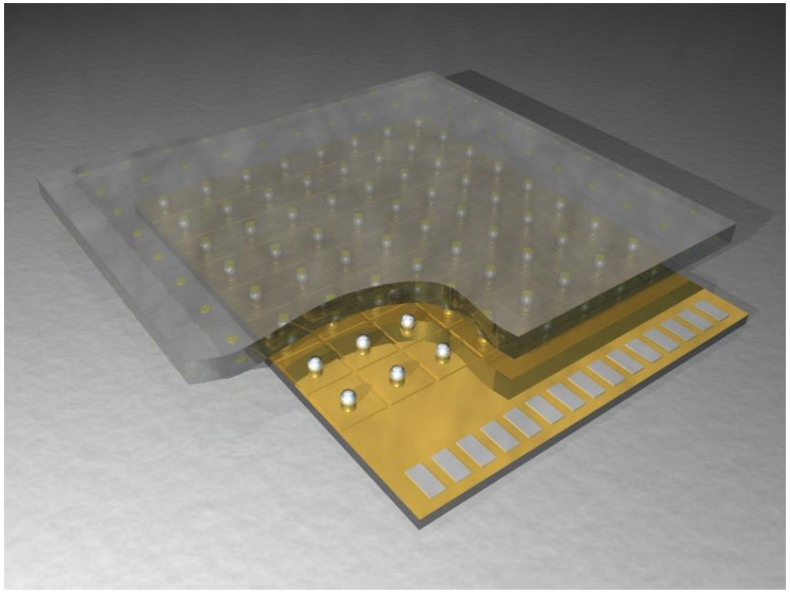
X-ray powder diffraction

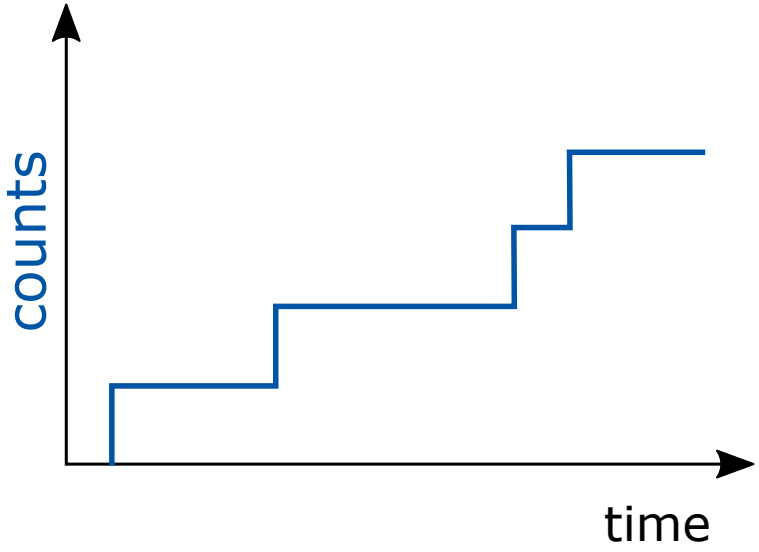
The power of the threshold

The future of high-pressure crystallography

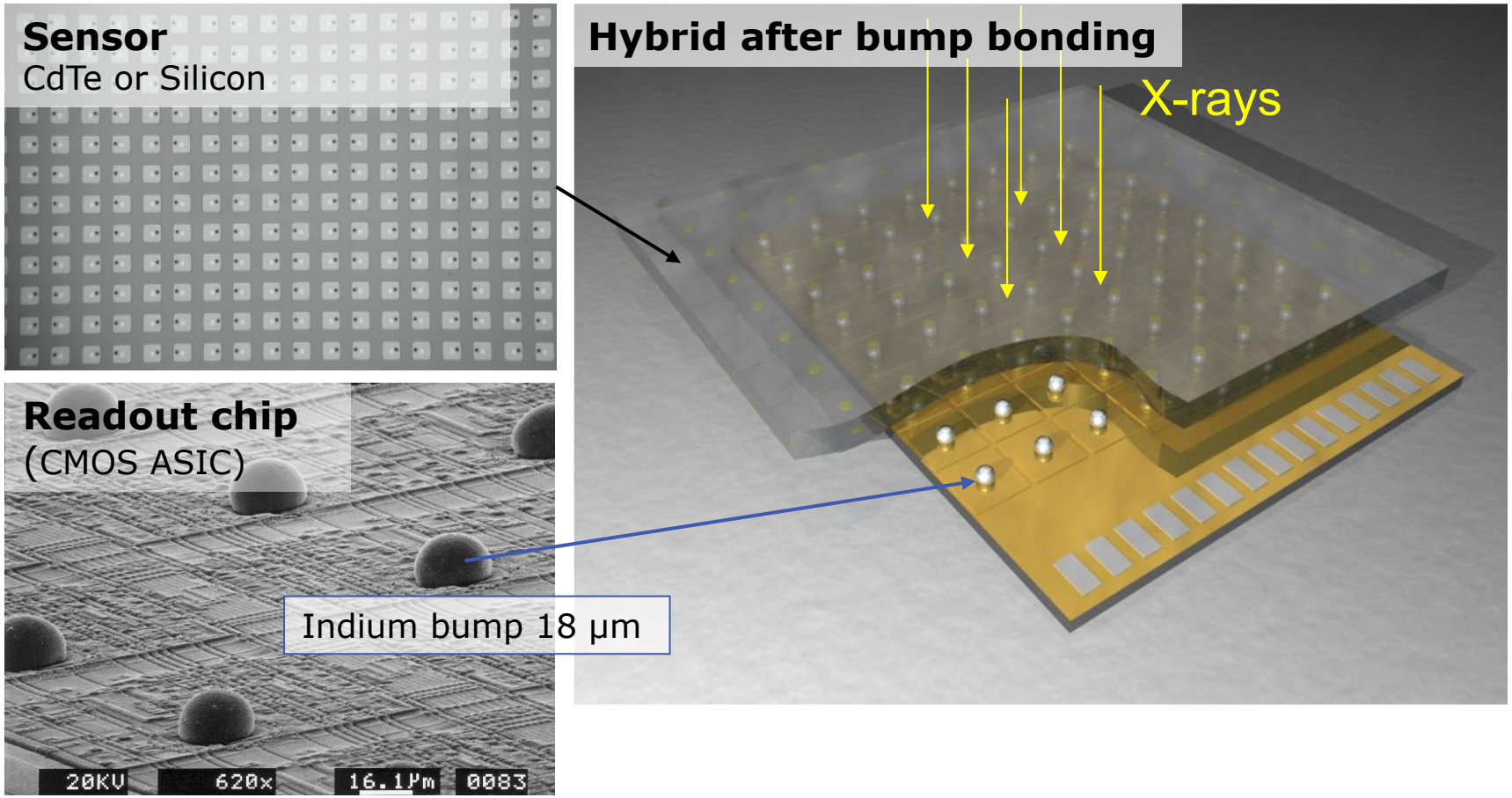
The meaning of HPC counting

Hybrid Pixels + Single Photon Counting

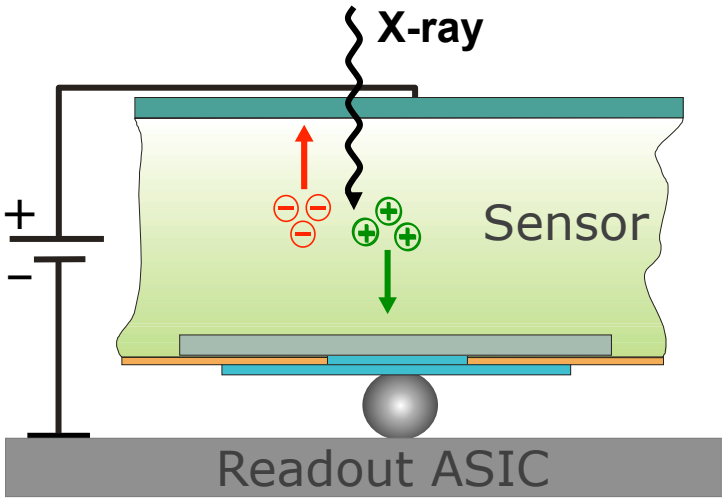




Hybrid pixel = sensor + readout



Direct detection in hybrid pixels



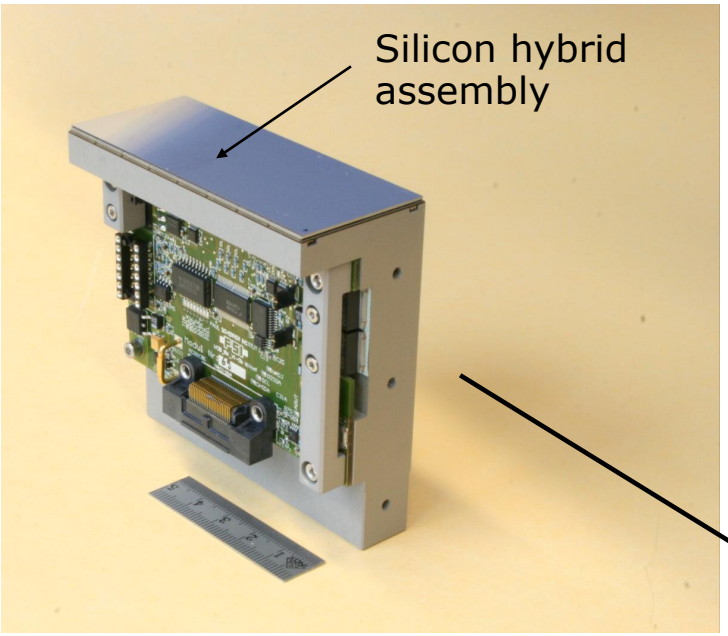
Sensor pixel

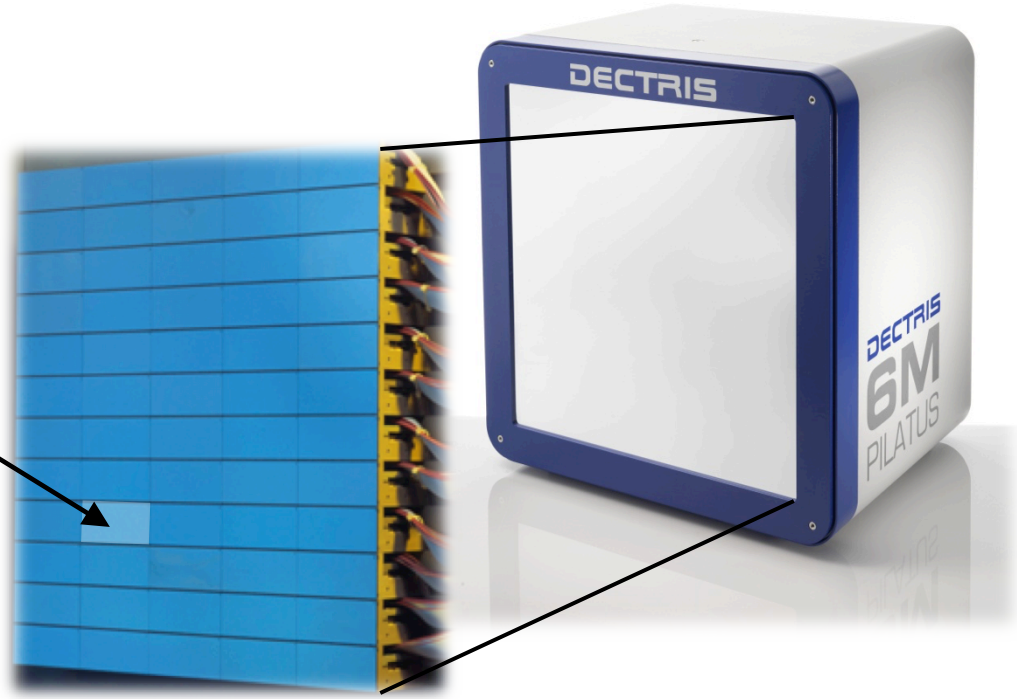
- *Absorption of X-ray photons*
  -> one e^-/hole pair per 3.6 eV
- *Charge is captured by electric field*

Pixel electronics

- *Counting of charge pulses*

Modular assembly of HPC detectors





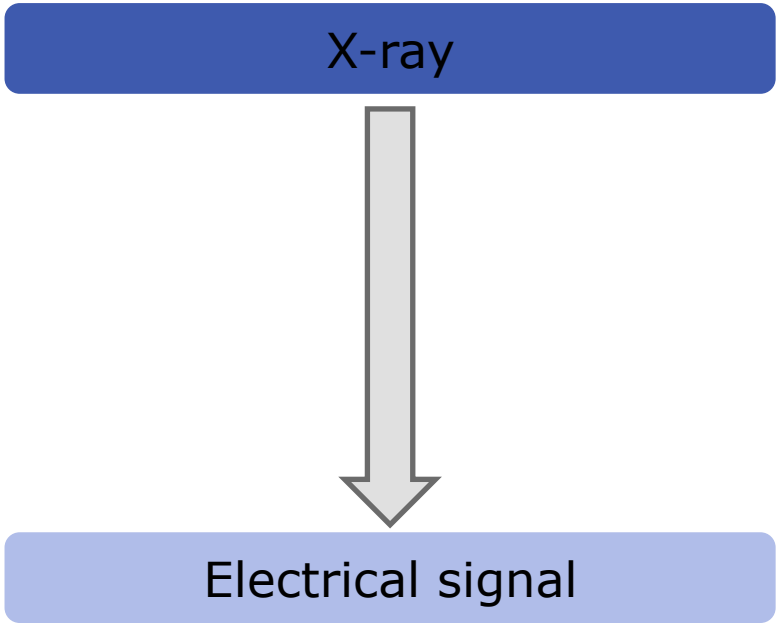
Module active area: 8 × 4 cm²

100k pixels on PILATUS (172 µm)

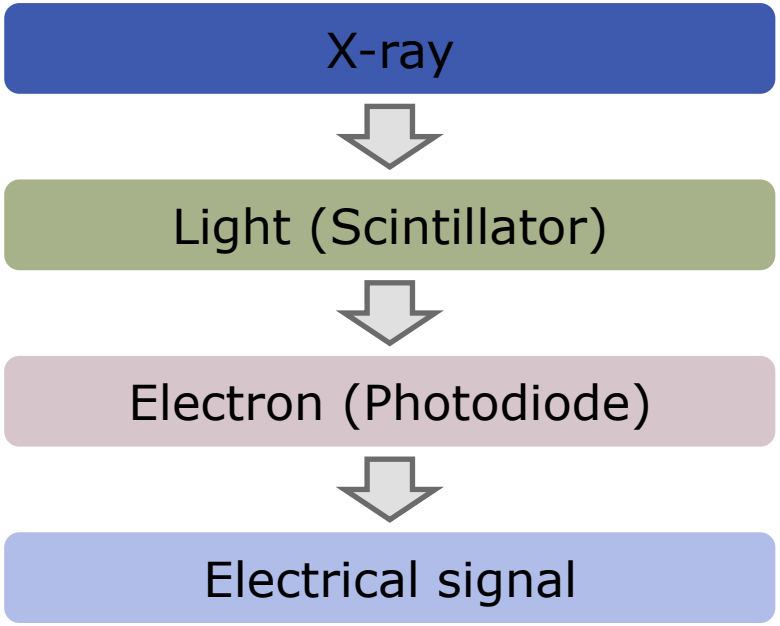
500k pixels on EIGER (75 µm)

Superiority of direct detection

Direct detection

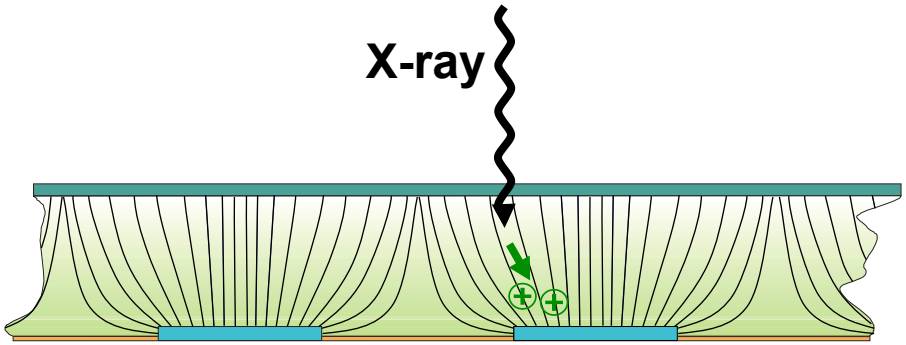


Indirect detection



Superiority of direct detection

Direct detection

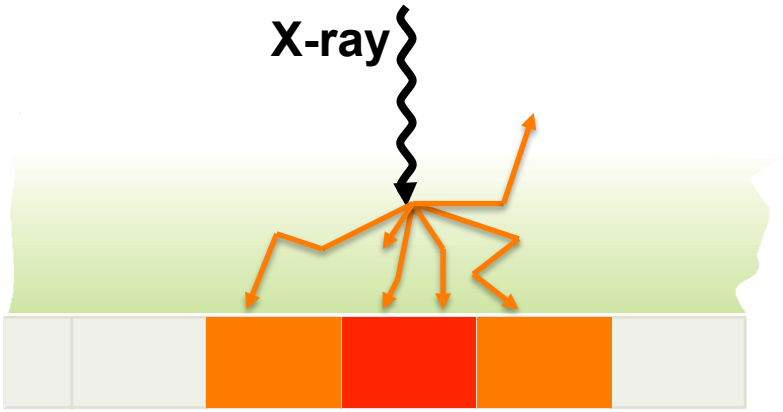


– *Charge captured in electric field*

=> ***All photons captured***

=> ***Signal remains in pixel***

Indirect detection



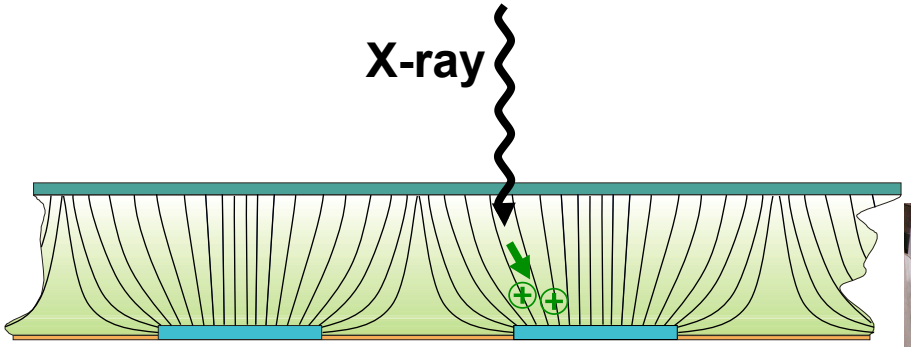
– *Secondary radiation scattered in scintillator*

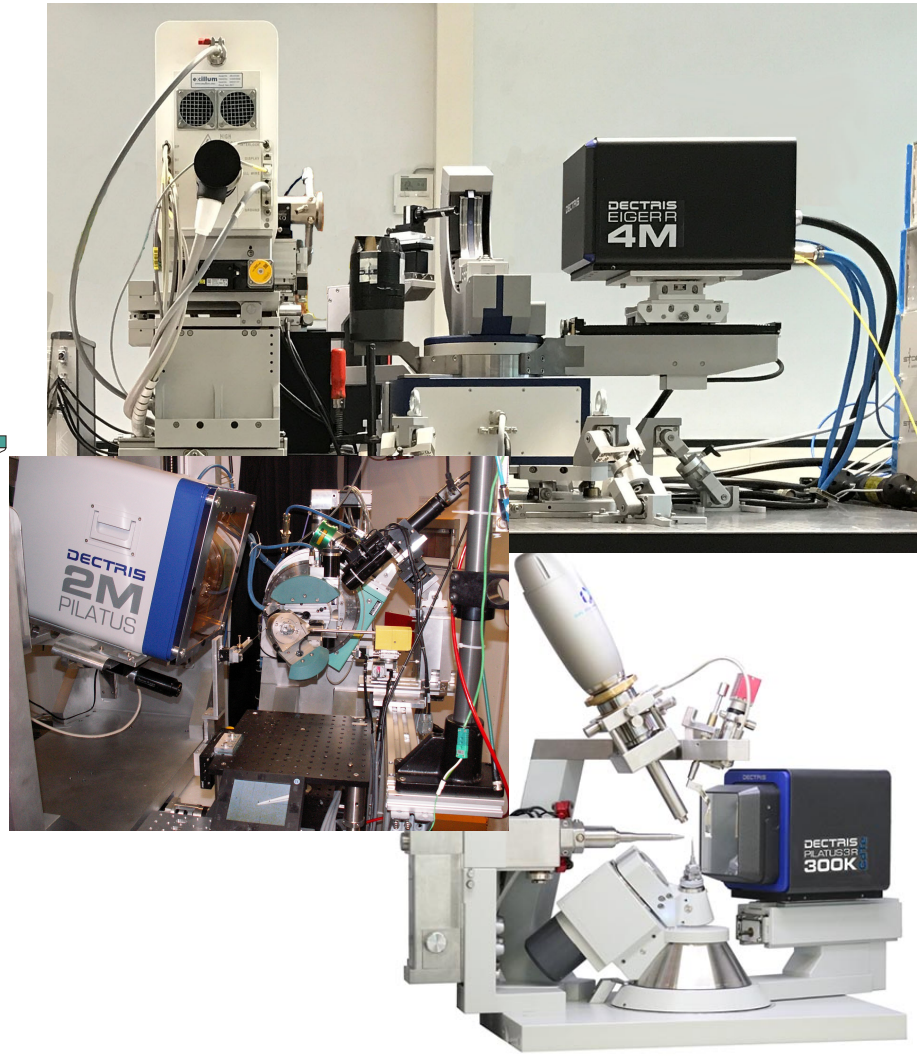
=> ***Signal spread across pixels***

=> ***Light partially lost***

Superiority of direct detection

Direct detection





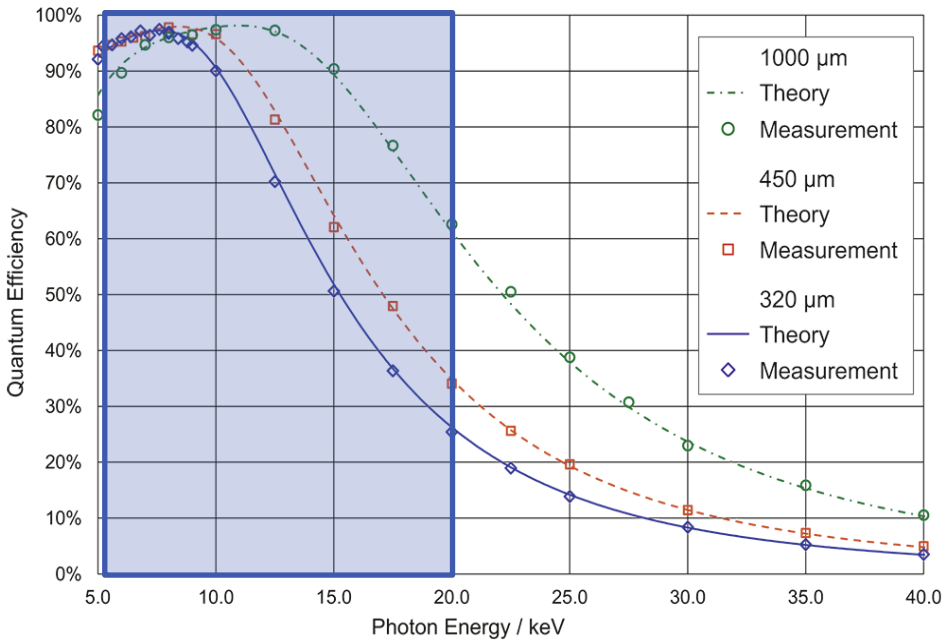
– *Charge captured in electric field*

=> ***All photons captured***

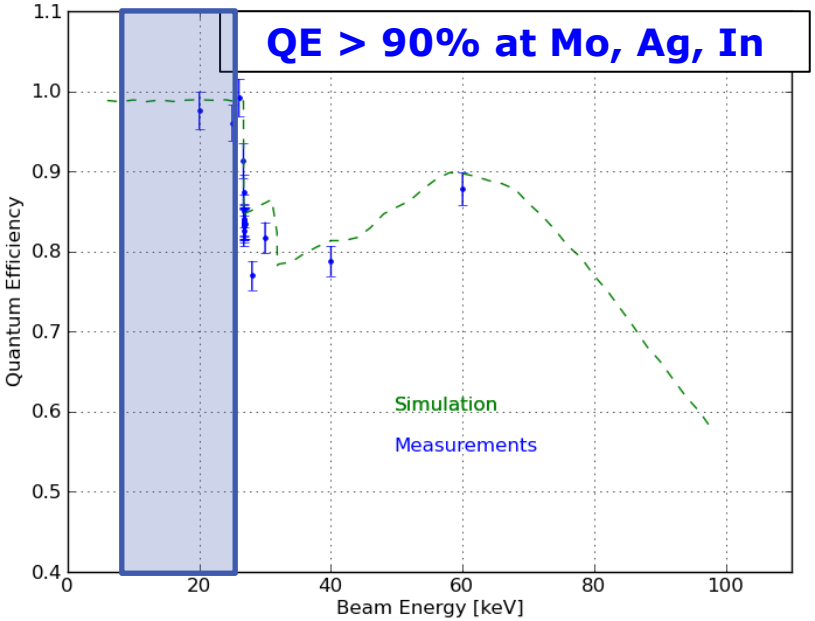
=> ***Signal remains in pixel***

Silicon and CdTe sensors

High quantum efficiency across a wide energy range



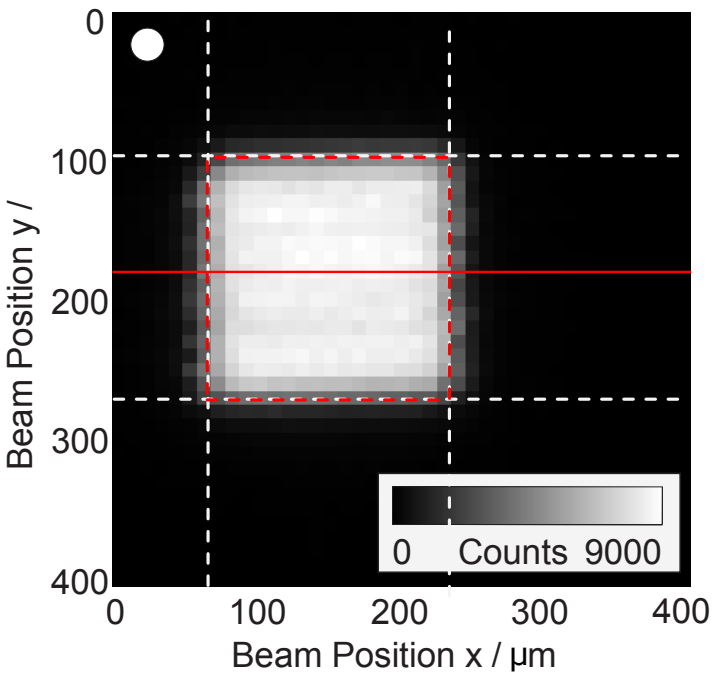
Silicon for low energies

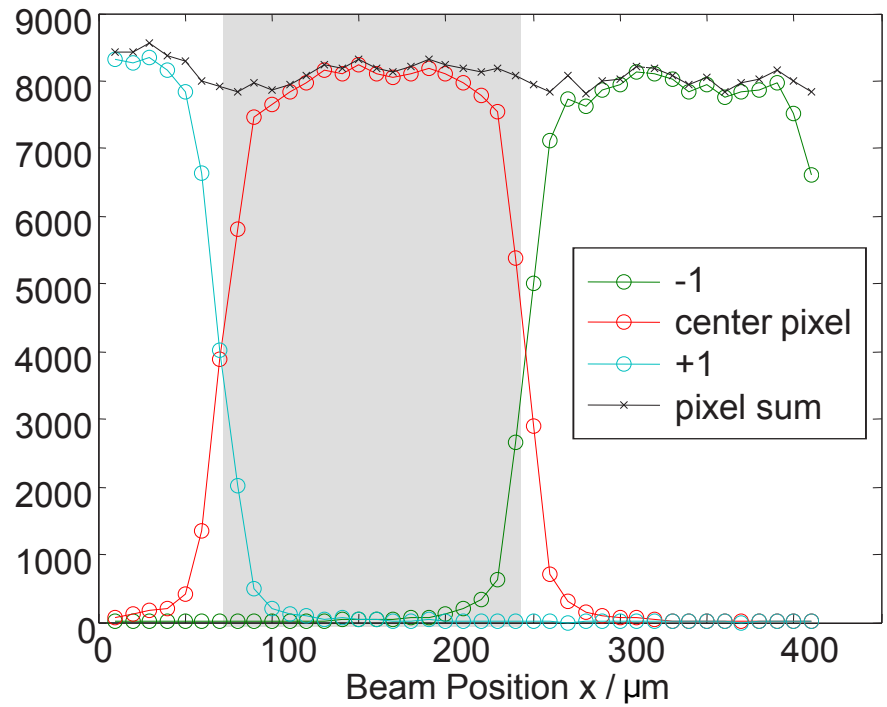


CdTe for high energies

Spatial resolution PILATUS3

All signal contained with pixel

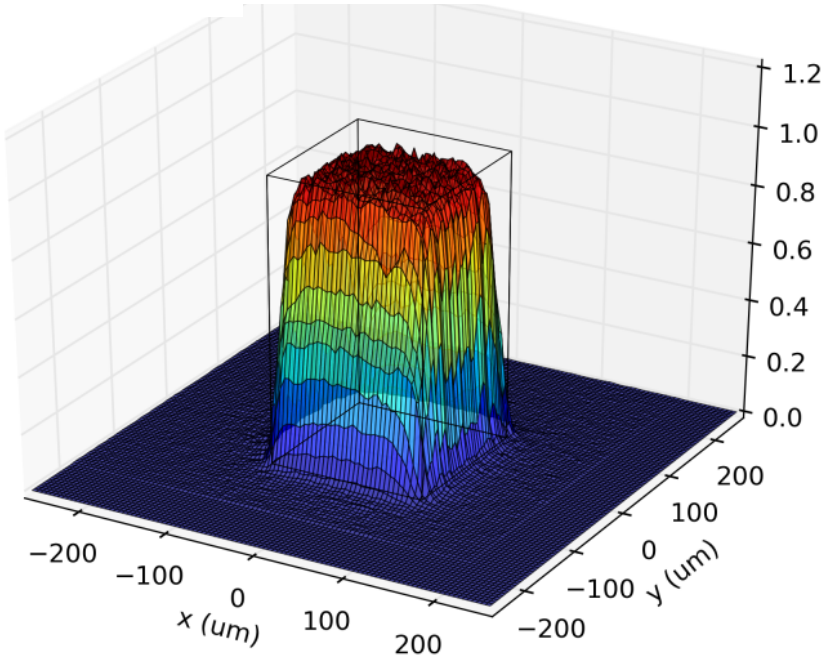




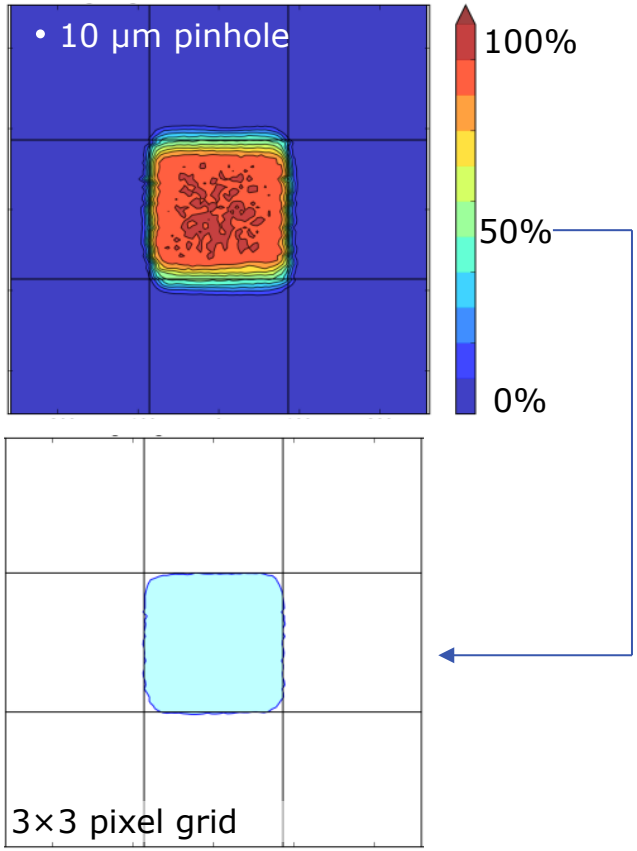
20 µm pencil beam, 10 µm rastering

Spatial resolution PILATUS3 CdTe

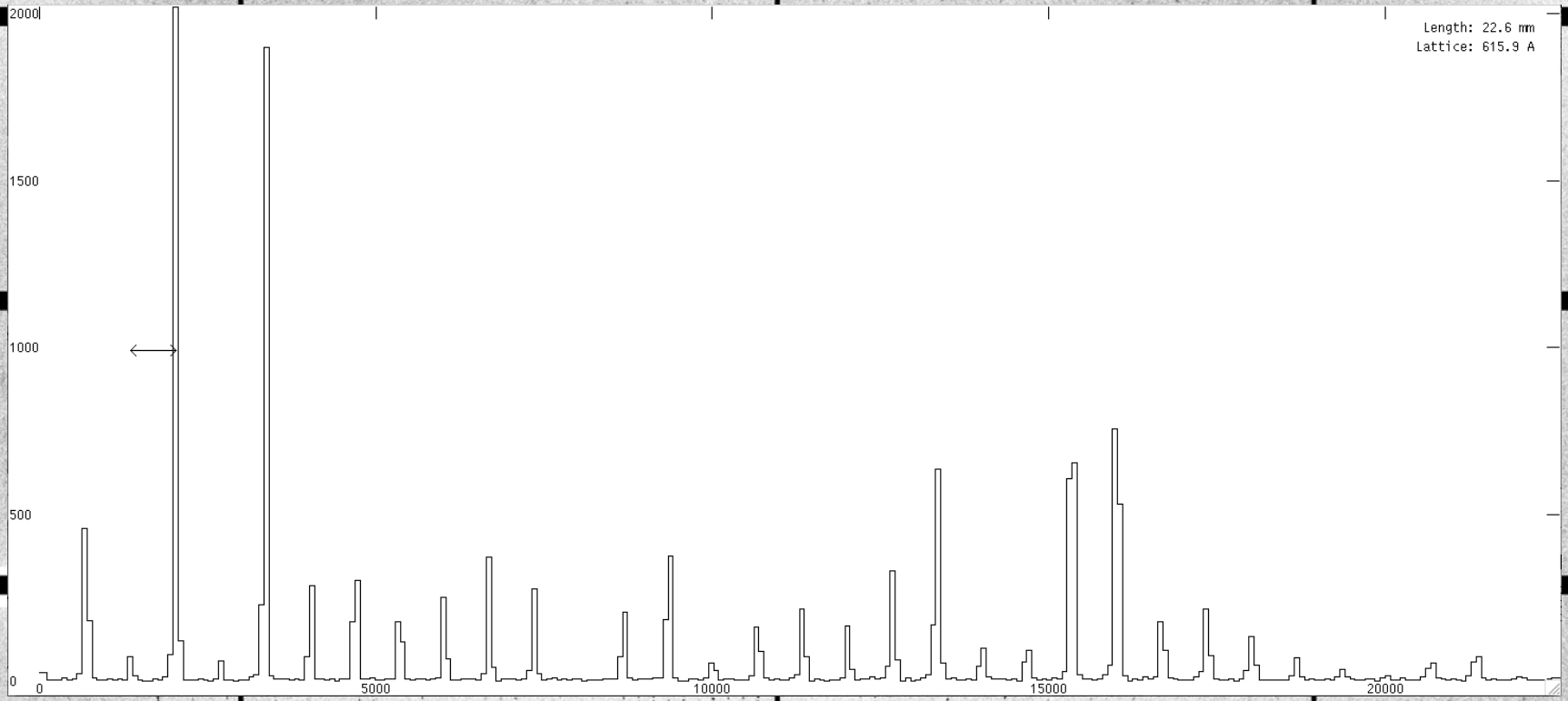
For CdTe as for silicon



10 µm pinhole at Ag Kα wavelength



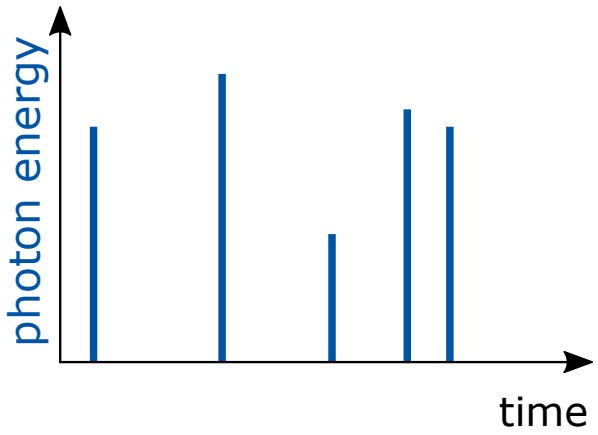
70S ribosome on EIGER X 16M



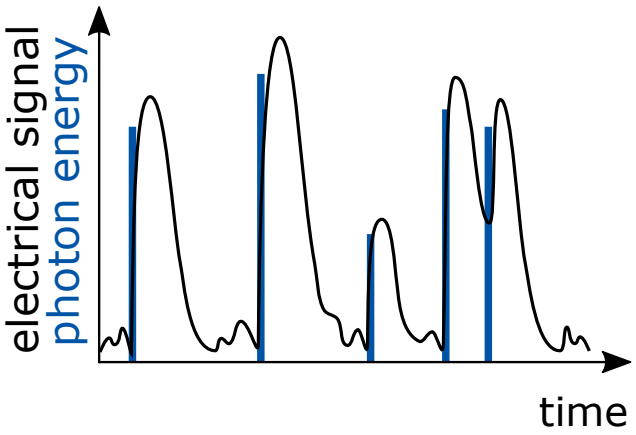
a = 210 Å, b = 450 Å, c = 620 Å
Diffraction to 2.3 Å, Y. Polikanov, UIC

Photon counting – one by one

– *Photons absorbed in sensor pixel*

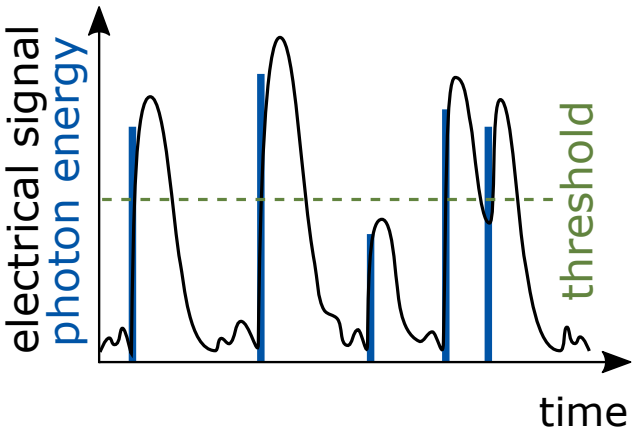


Photon counting – one by one



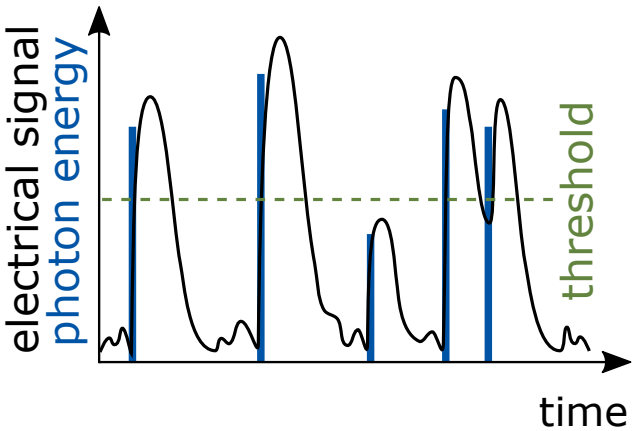
- *Photons absorbed in sensor pixel*
- *Charge pulse proportional to energy*

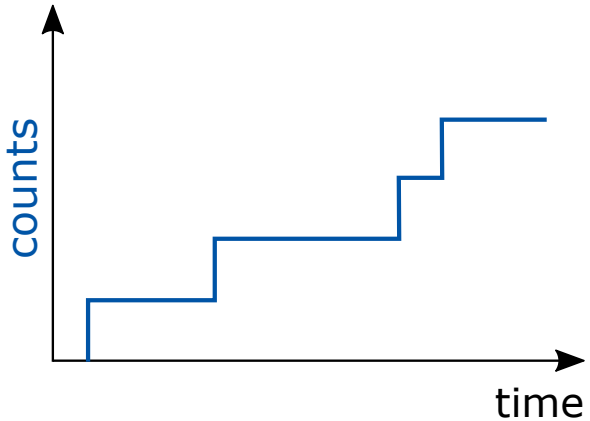
Photon counting – one by one



- *Photons absorbed in sensor pixel*
- *Charge pulse proportional to energy*
- *Threshold to discard noise*

Photon counting – one by one





- *Photons absorbed in sensor pixel*
- *Charge pulse proportional to energy*
- *Threshold to discard noise*

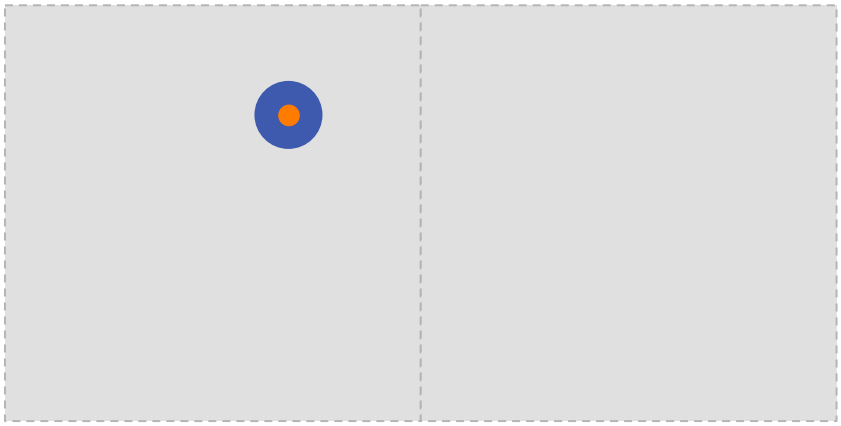
- *Signals above threshold are counted*
  - ***Suppression of dark signal***
  - *Suppression of fluorescence*
  - *Suppression of electronic noise*

- *On-the-fly digitization in digital counter*
  - ***No readout noise***
  - *Fast readout*
  - *High dynamic range*

Threshold at half photon energy

All signal counted within on pixel

Electronic noise + fluorescence filtered

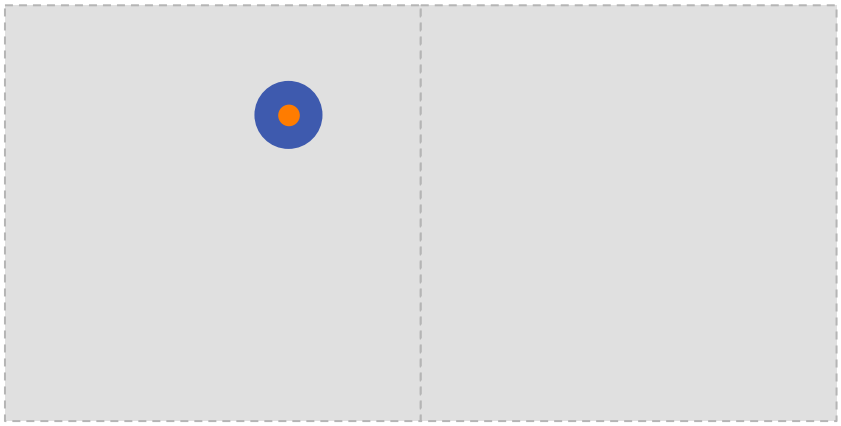


1 photon
100% energy
1 count -

Threshold at half photon energy

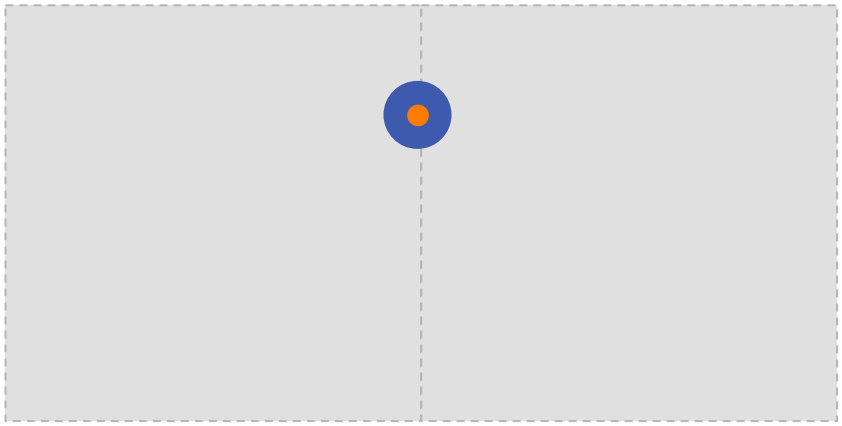
All signal counted within on pixel

Electronic noise + fluorescence filtered



1 photon
100% energy
1 count

-



1 photon
60% energy
1 count

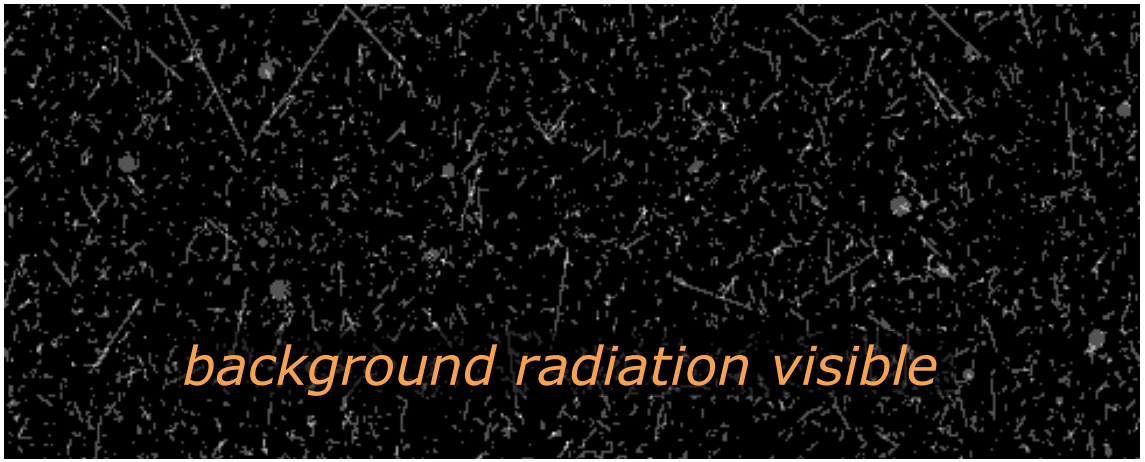
40% energy

-

Noise-free detection

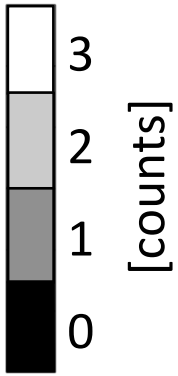
100 ms

1 hour



100K detector

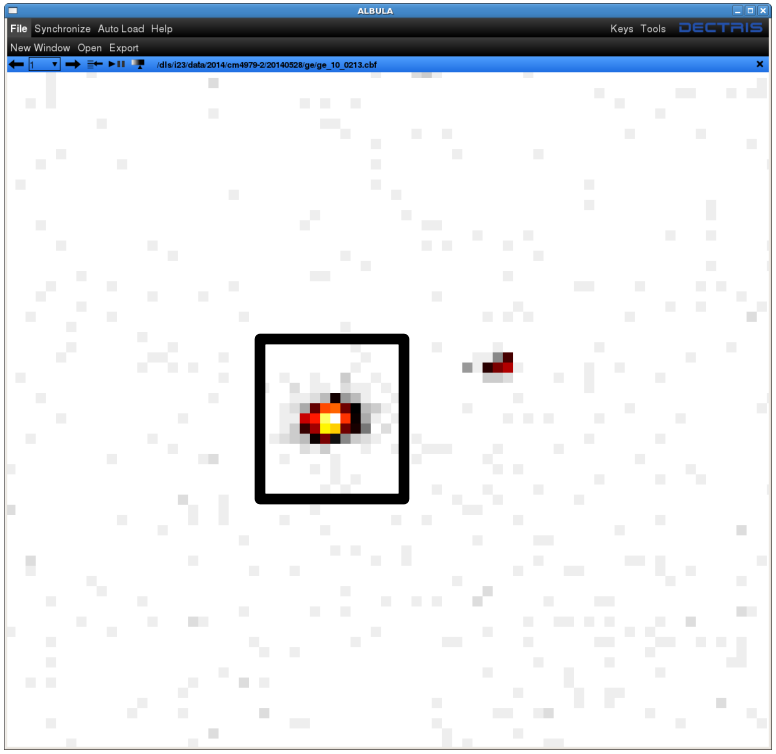
No readout noise
No dark signal

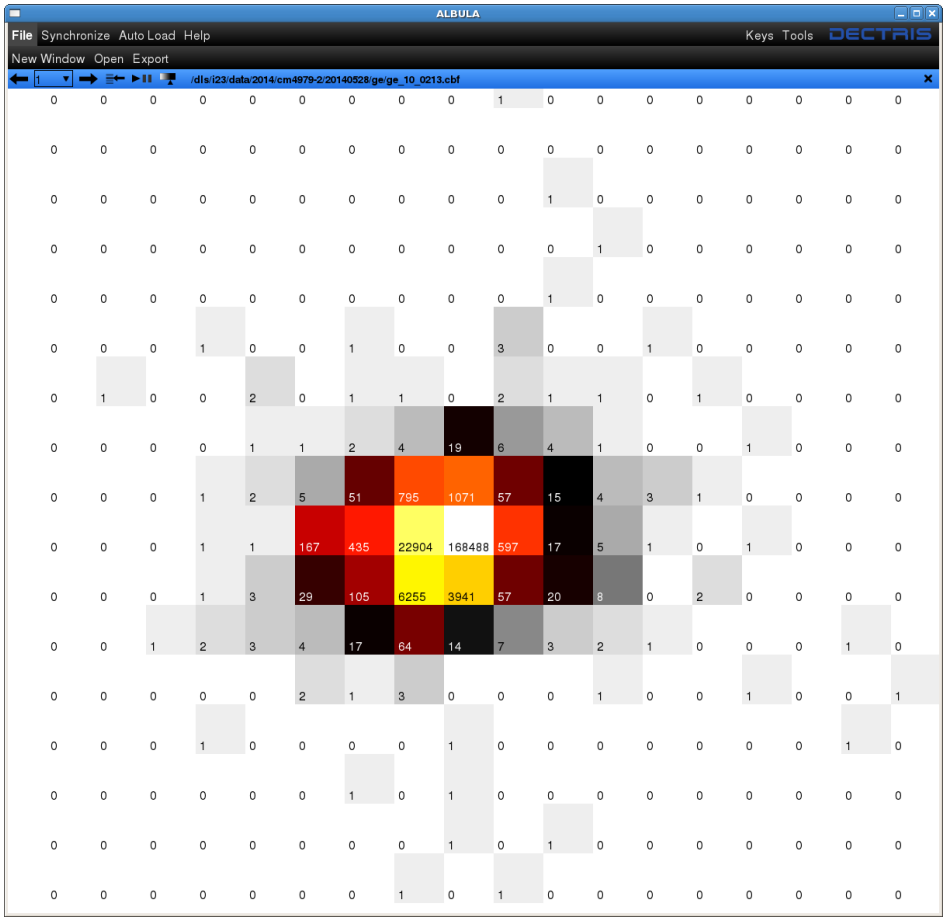


Negligible background for max. I/σ(I)

Crystallography in vacuum

Germanate @ DLS I23.

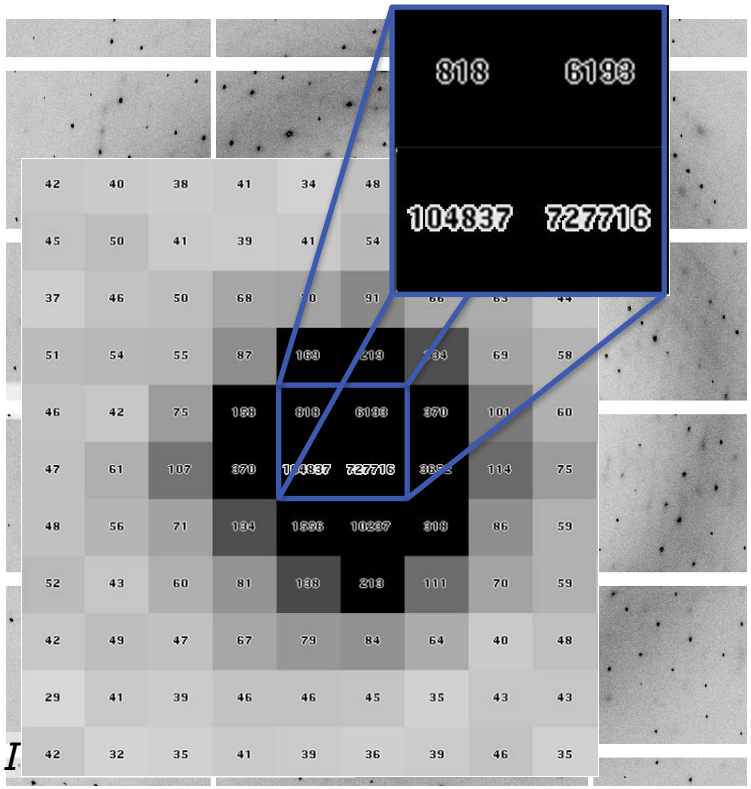




Dynamic range for strong reflections

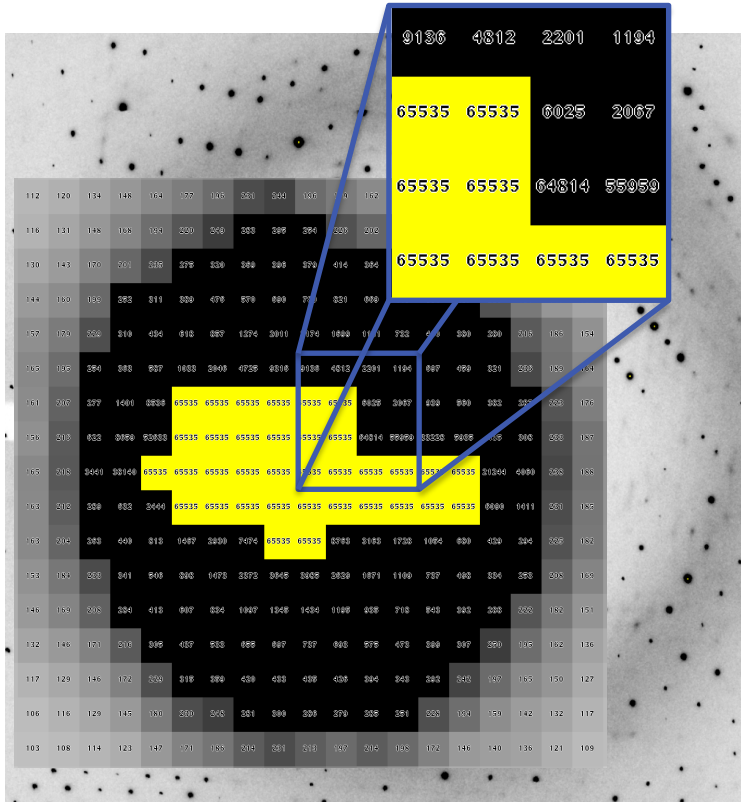
HPC detectors

PILATUS @ 20 bit (1 million counts)
EIGER @ 32 bits (4 billion counts)



Scintillator-based detectors

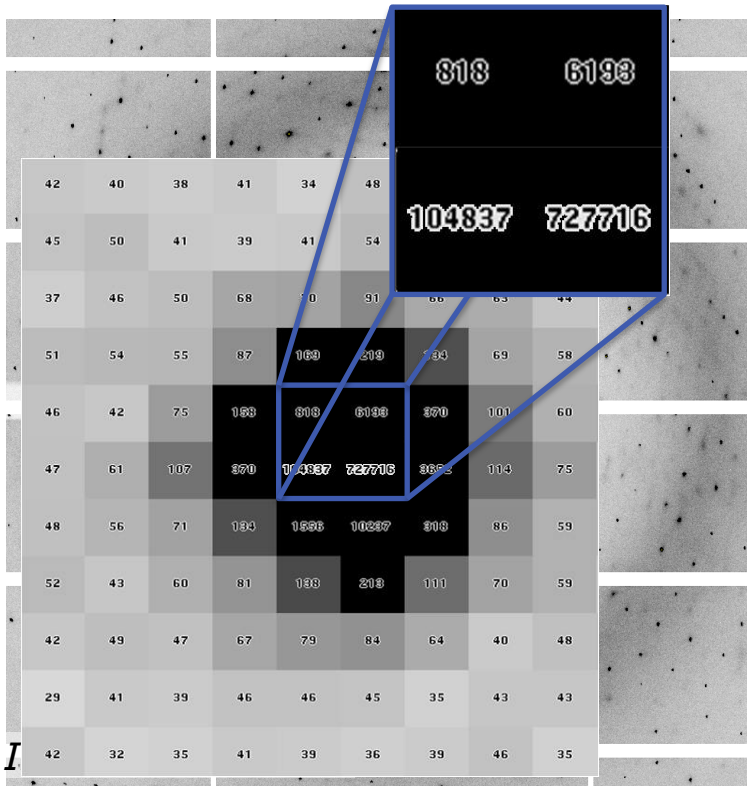
CCD @ 16 bits (65 thousand ADU)



Excellent spatial resolution

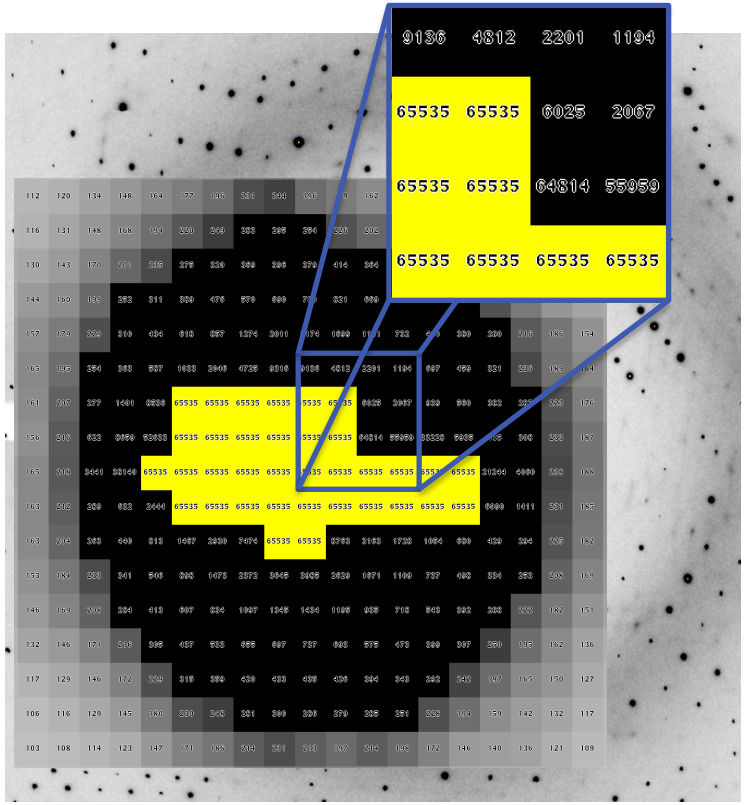
HPC detectors

Sharp point spread function



Scintillator-based detectors

Conversion dilutes signal



DECTRIS® detecting the future

MYTHEN2 – 1D detectors for PXRD

MYTHEN2 R
for laboratories

MYTHEN2 X
for synchrotrons

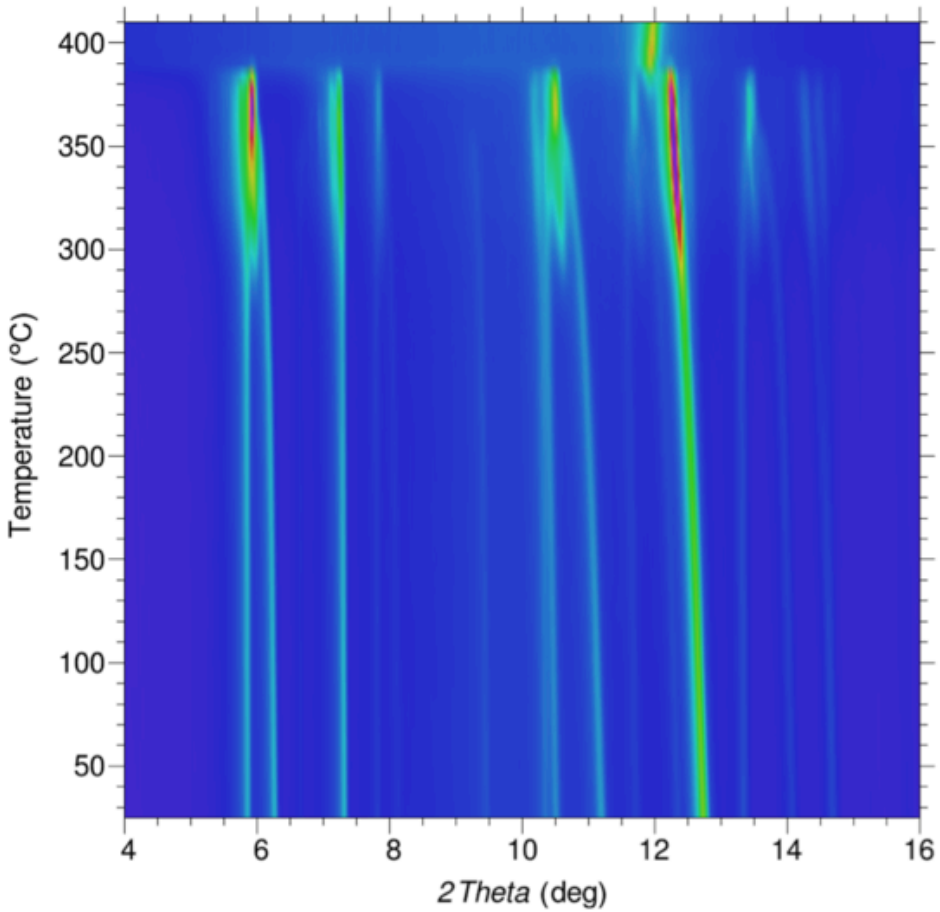
MYTHEN2 R/X 1D – 640 strips
MYTHEN2 R/X 1K – 1280 strips

50 µm strips
up to 4 detectors per DCS

PXRD at elevated temperatures

Response of adenine to heat

- *Simultaneous coverage of large angular range*
- *Collected data free of temporal inconsistencies*
- *Low noise for accurate data*

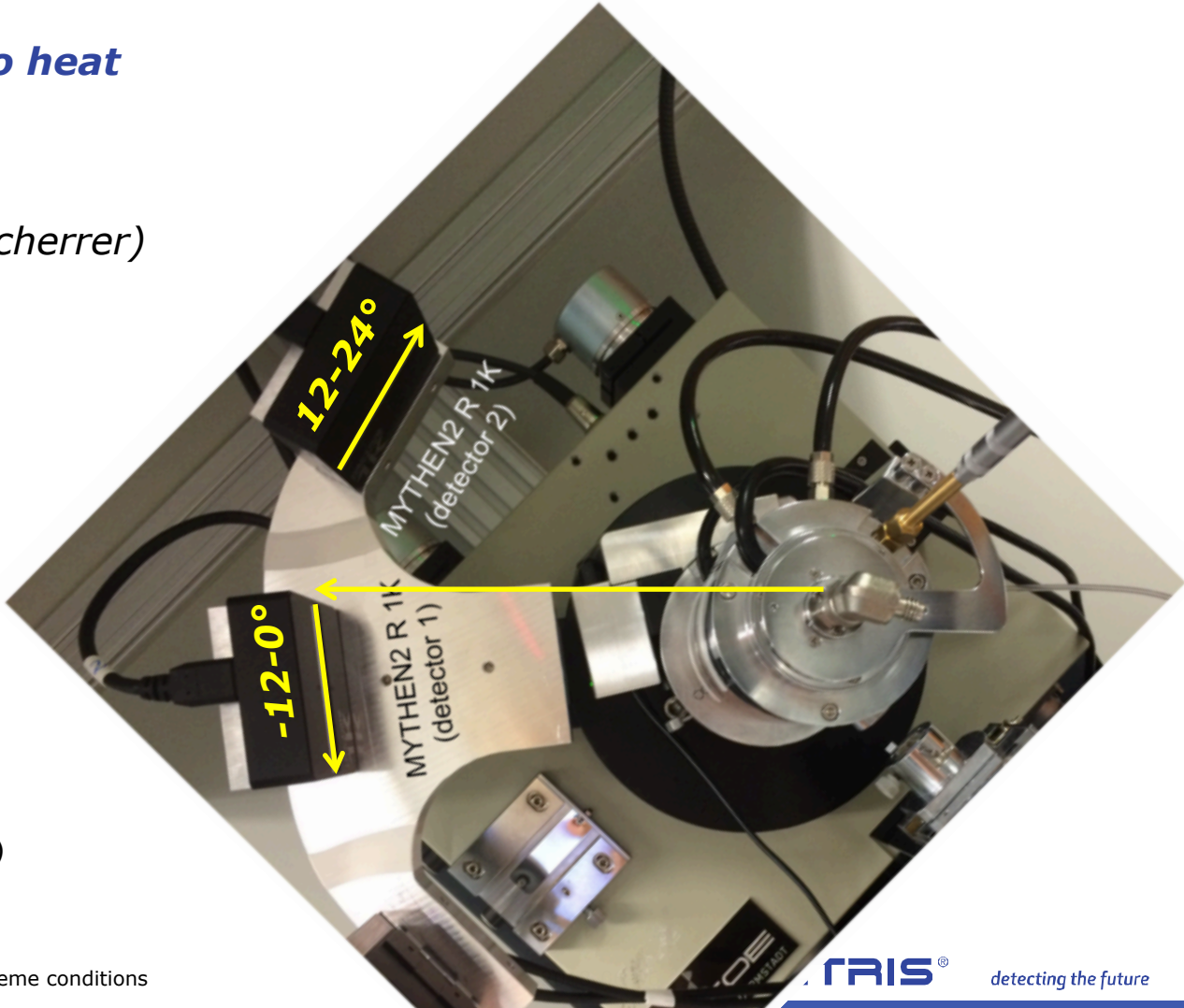


Courtesy Ivan Halasz (Zagreb)

PXRD at elevated temperatures

Response of adenine to heat

- *Mo source*
- *STOE INSITU HT2 oven*
- *STOE Stadi P (Debye-Scherrer)*
- *MYTHEN2 R 2K*

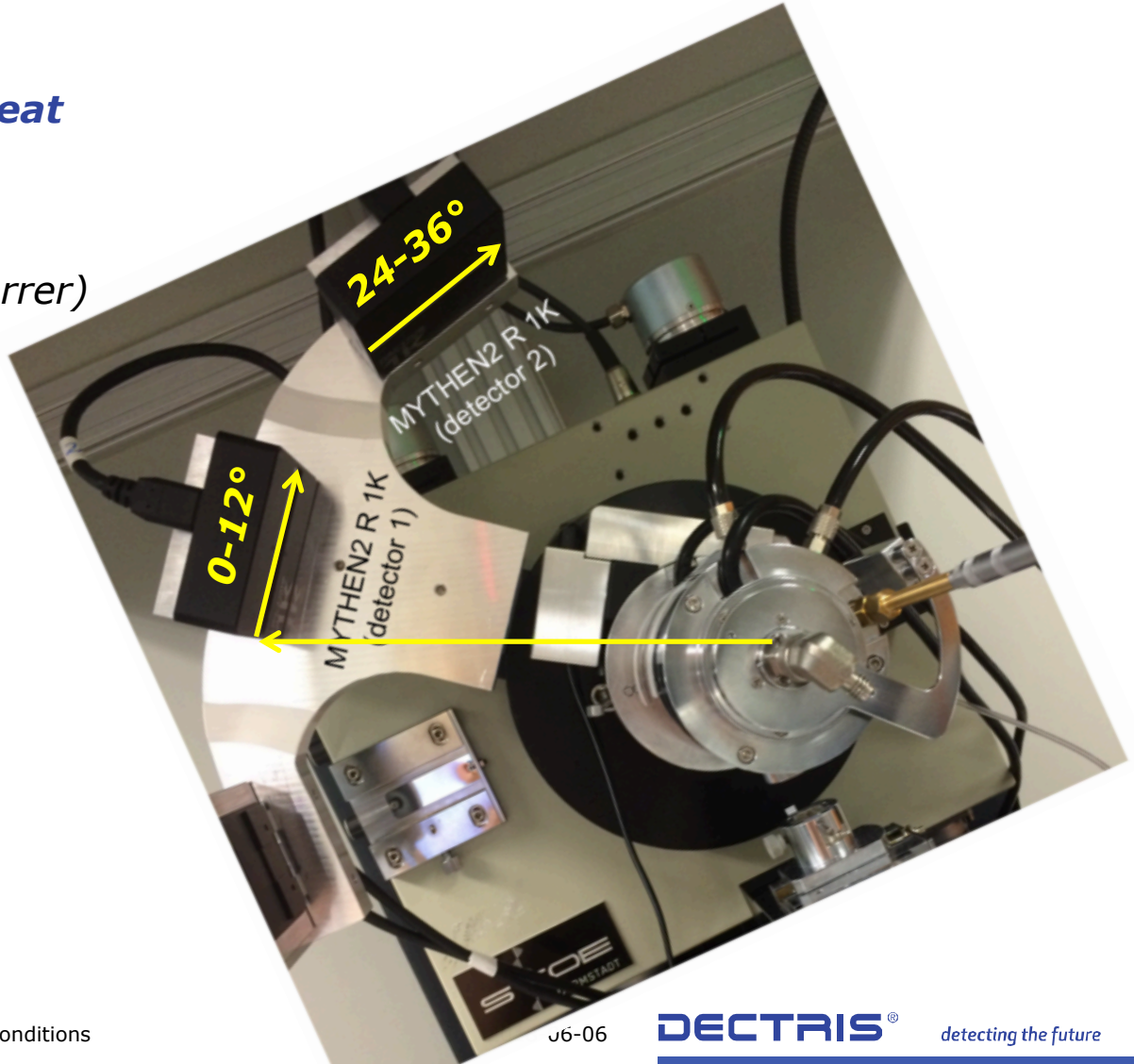


Courtesy Ivan Halasz (Zagreb)

PXRD at elevated temperatures

Response of adenine to heat

- *Mo source*
- *STOE INSITU HT2 oven*
- *STOE Stadi P (Debye-Scherrer)*
- *MYTHEN2 R 2K*

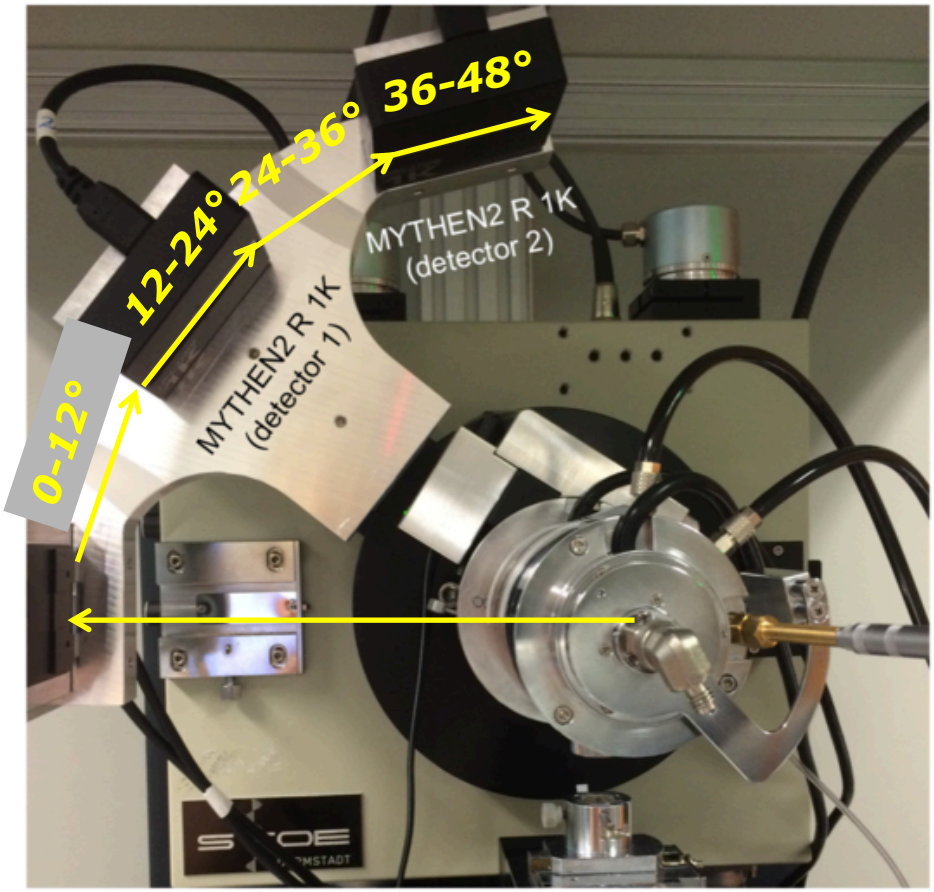


Courtesy Ivan Halasz (Zagreb)

PXRD at elevated temperatures

Response of adenine to heat

- *Mo source*
- *STOE INSITU HT2 oven*
- *STOE Stadi P (Debye-Scherrer)*
- *MYTHEN2 R 2K*
- *Two exposures of 12 min each*

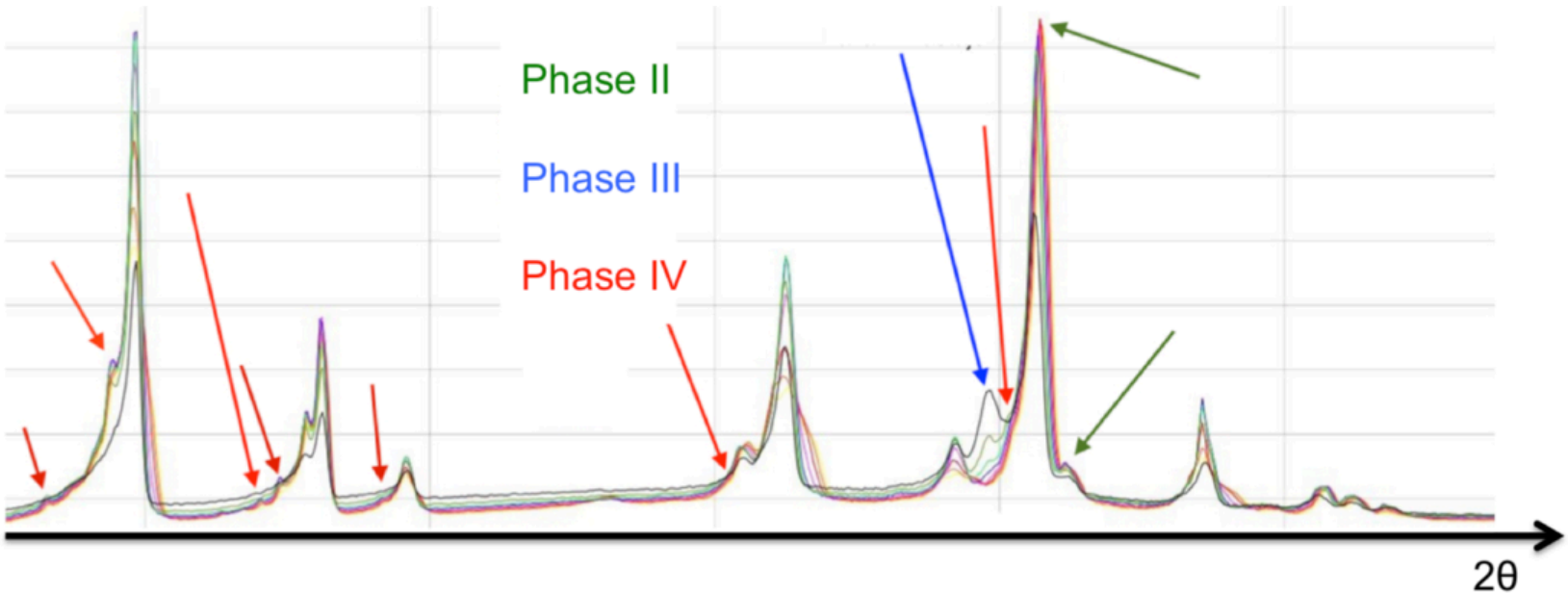


Courtesy Ivan Halasz (Zagreb)

PXRD at elevated temperatures

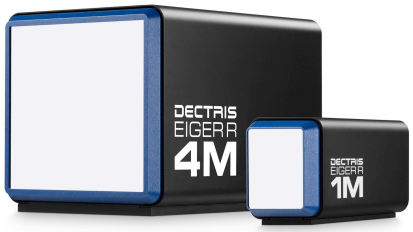
Multiple MYTHEN detectors

- *Phase I to phase II upon heating*
- *Phase III above 380°C*
- *Phase IV briefly before phase III*



PILATUS & EIGER with Si sensors

PILATUS3 R for laboratory

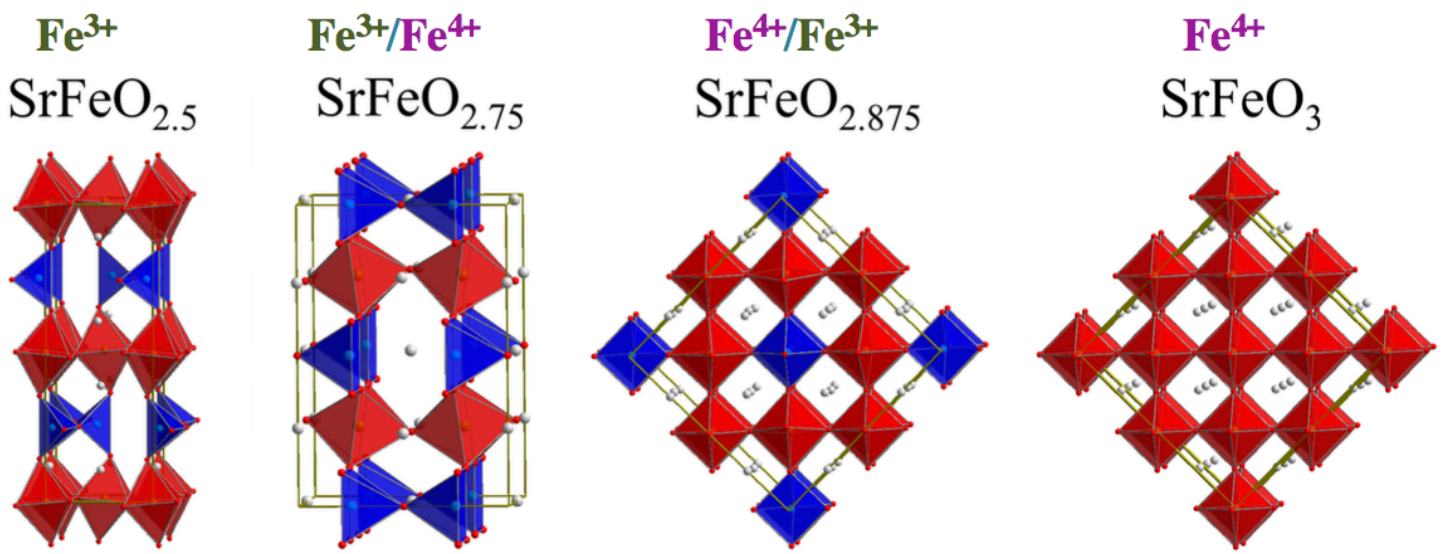


EIGER R for laboratory

EIGER2 R 500K for laboratory XRD

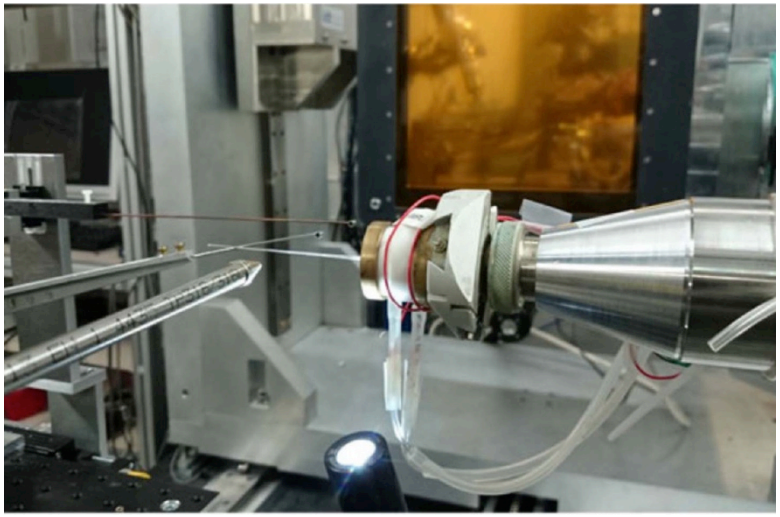
Faster (and bigger) instruments for synchrotrons

Electrochemical oxidation of $SrFeO_{2.5}$



Courtesy Werner Paulus (Montpellier)

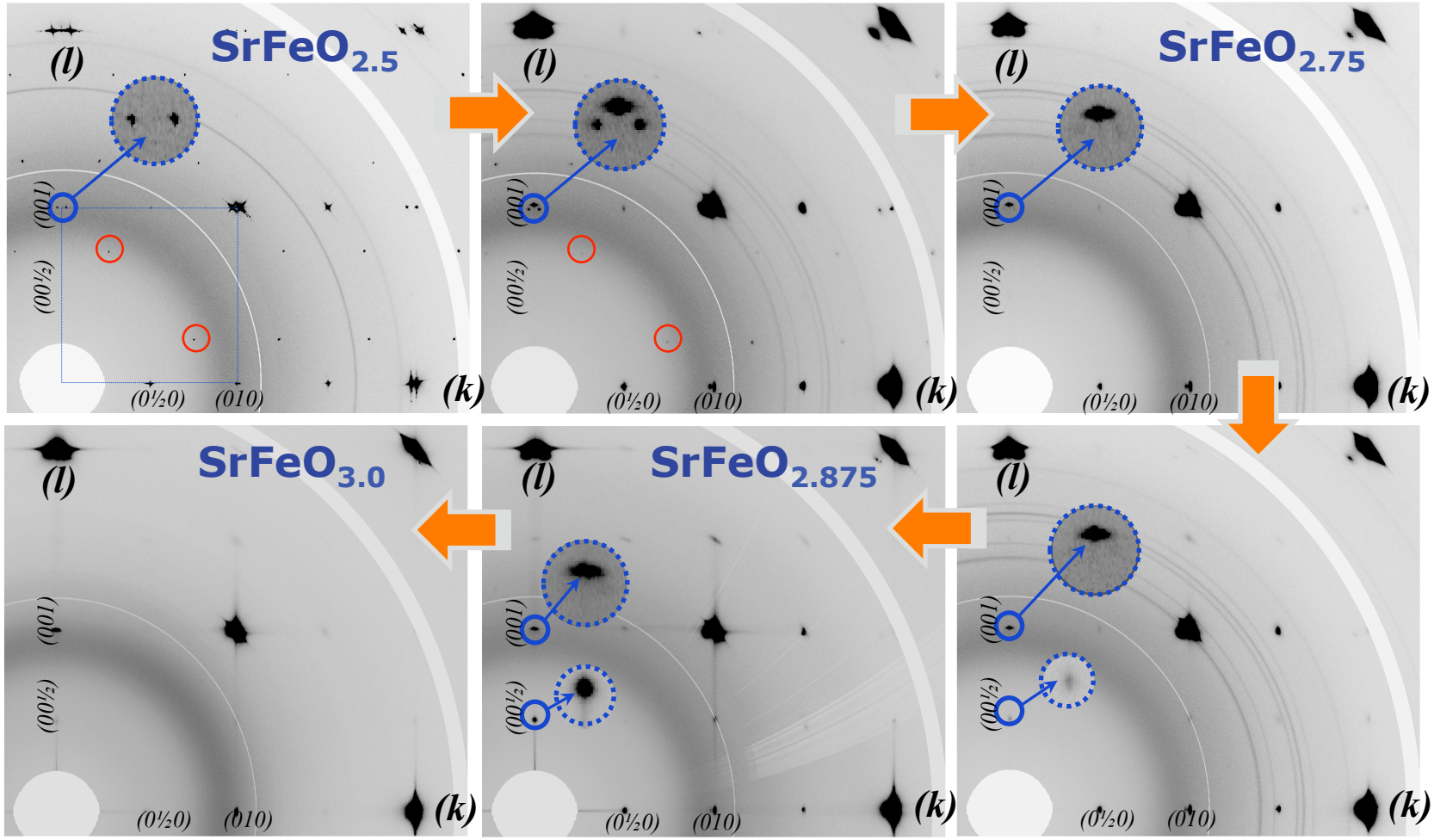
Electrochemical cell at BM01



Swiss-Norwegian beamline (BM01) at ESRF

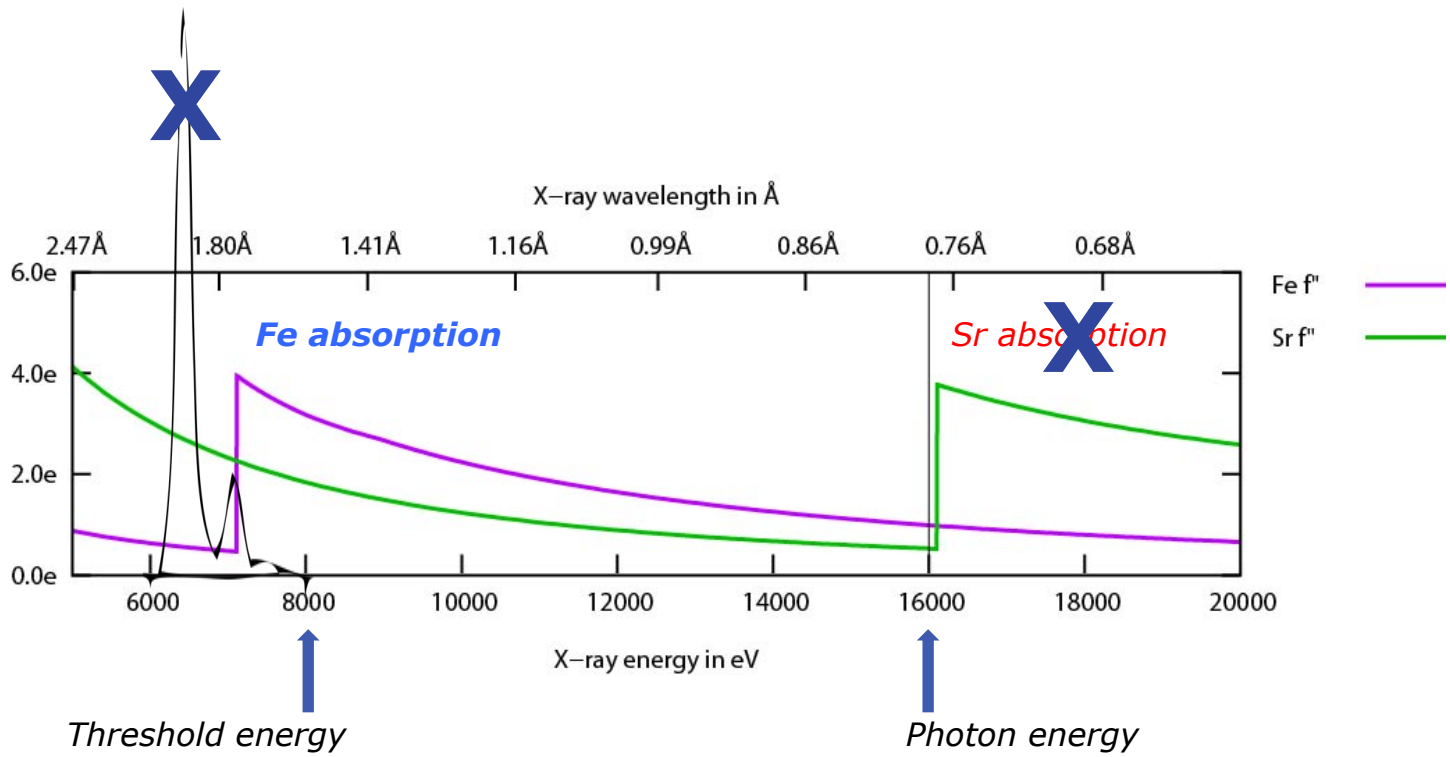
also on STOE Stadivari Mo
with PILATUS 200K

Evolution of reflections in 0kl plane



t = 5 days

Threshold suppresses fluorescence



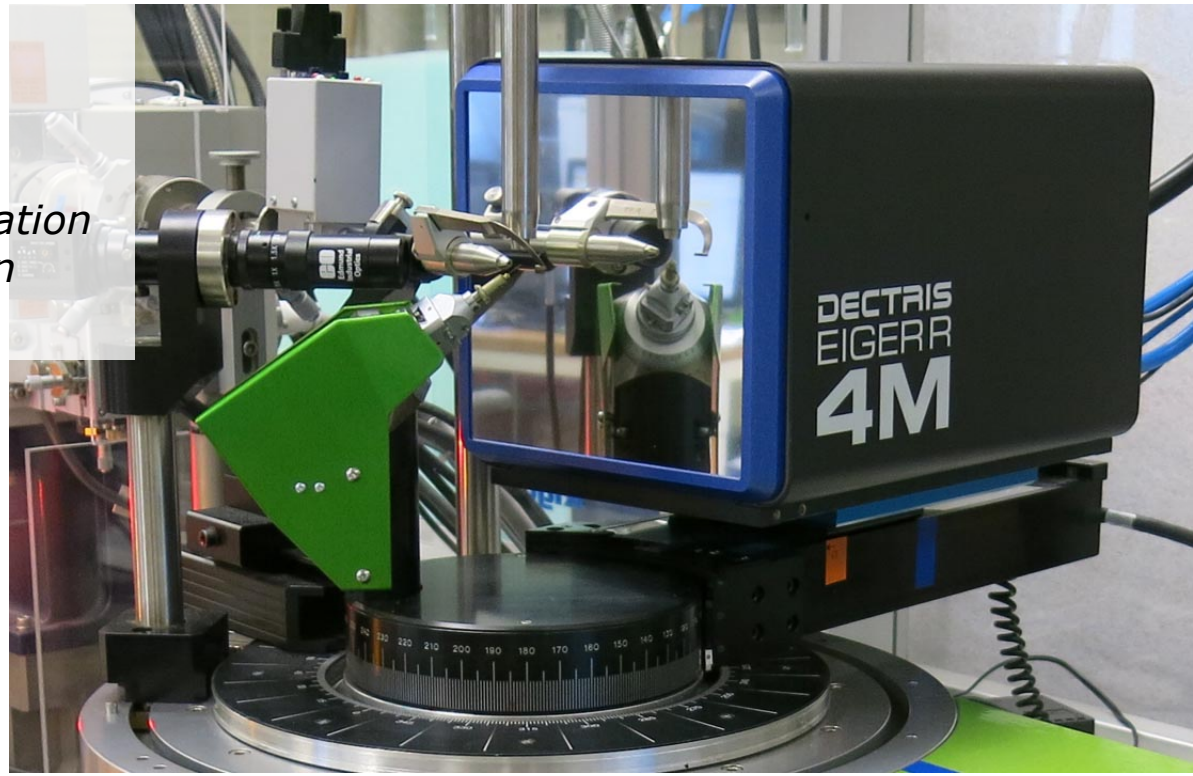
$E_{ex,Fe}$ = 7.1 keV

$E_{ex,Sr}$ = 16.1 keV

Fluorescence suppression in the lab

Small molecule with Europium

- *$E_{em,Eu}$ = 5.8 keV*
- *Structure determination impossible with integrating detectors*
- *Excellent data with Cu radiation and threshold at 6.2 keV on EIGER R 4M*

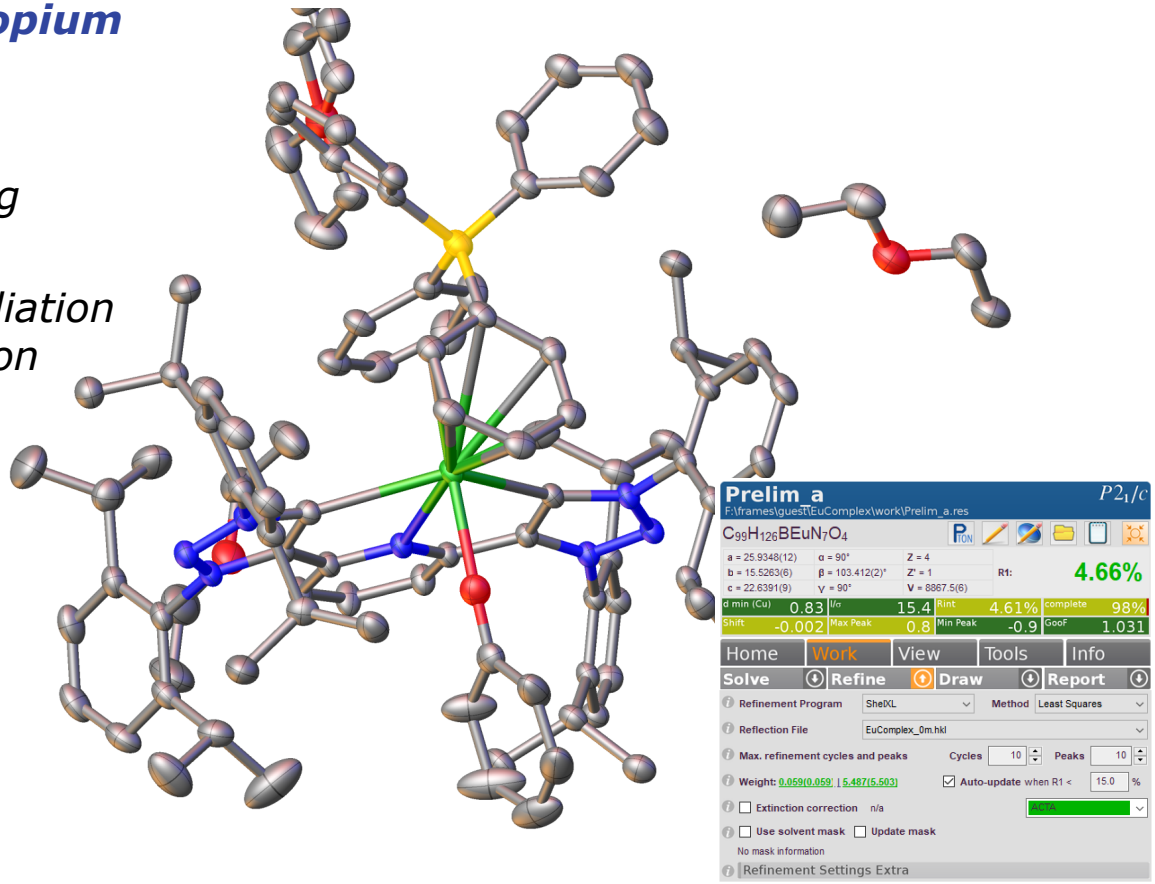


Courtesy Curtis Moore (UCSD)

Fluorescence suppression in the lab

Small molecule with Europium

- *$E_{em,Eu}$ = 5.8 keV*
- *Structure determination impossible with integrating detectors*
- *Excellent data with Cu radiation and threshold at 6.2 keV on EIGER R 4M*

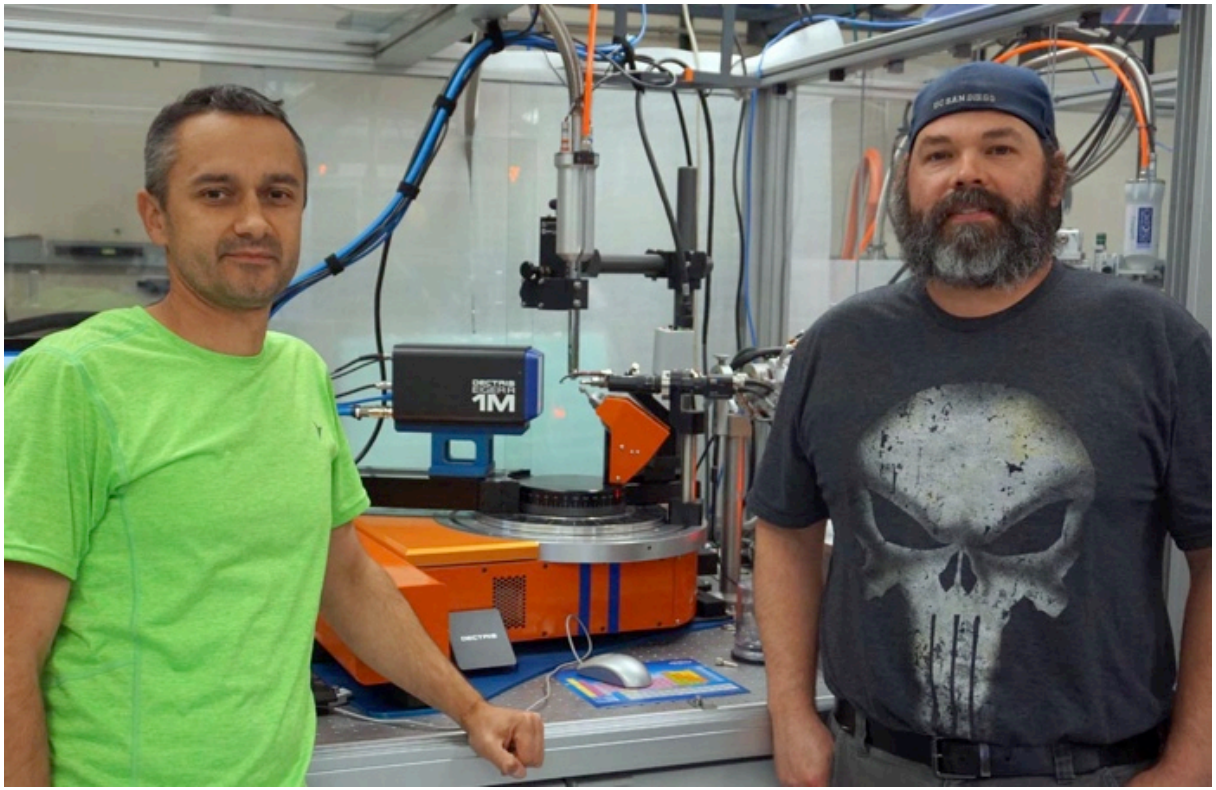


Courtesy Curtis Moore (UCSD)

Crystallography at USCD

Curtis Moore and Milan Gembicky

– EIGER R 1M



PILATUS3 CdTe detectors

PILATUS3 R CdTe
for laboratories

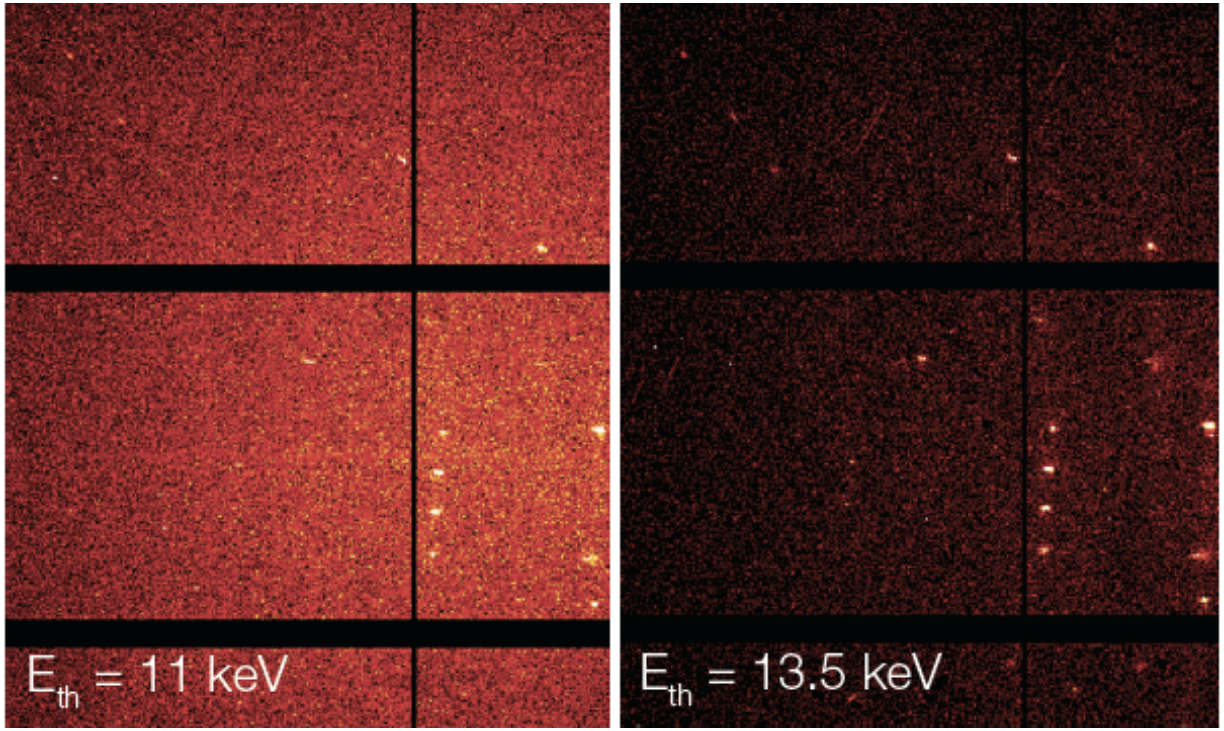
PILATUS3 X CdTe
for synchrotrons

25 x 28 cm²

Fluorescence suppression in the lab

Charge density studies

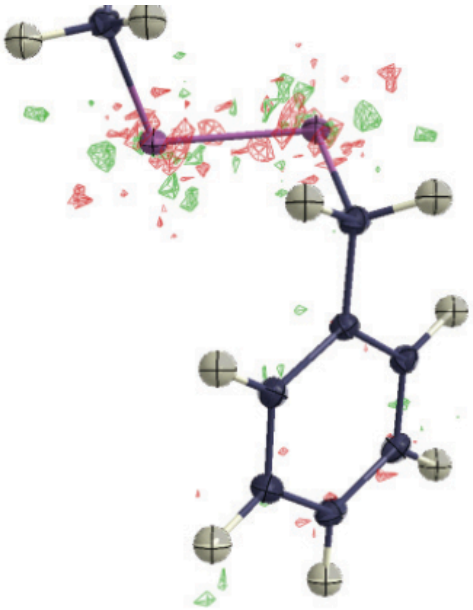
- *Dibenzyl diselenide*
- *Ag source*
- *$E_{em,Se}$ = 11.2-12.5 keV*
- *Data collection with Ag source (22.2 keV)*
- *No fluorescence noise with threshold at 13.5 keV*



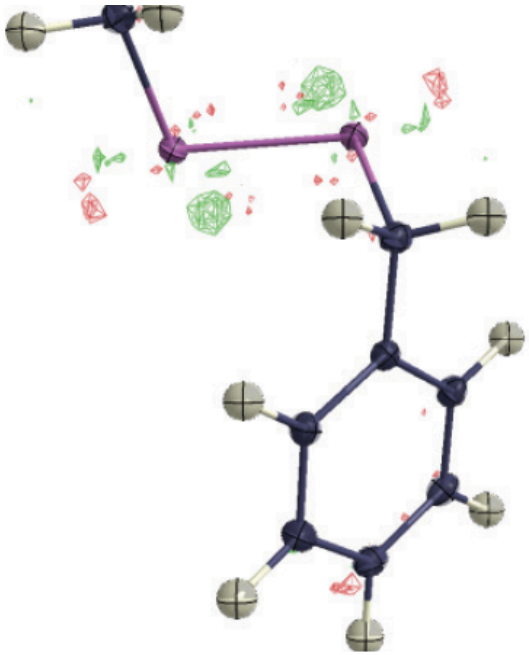
High-quality charge density

Dibenzyl diselenide

– *Isolevels at ± 0.11 e Å^{-3}*



CCD



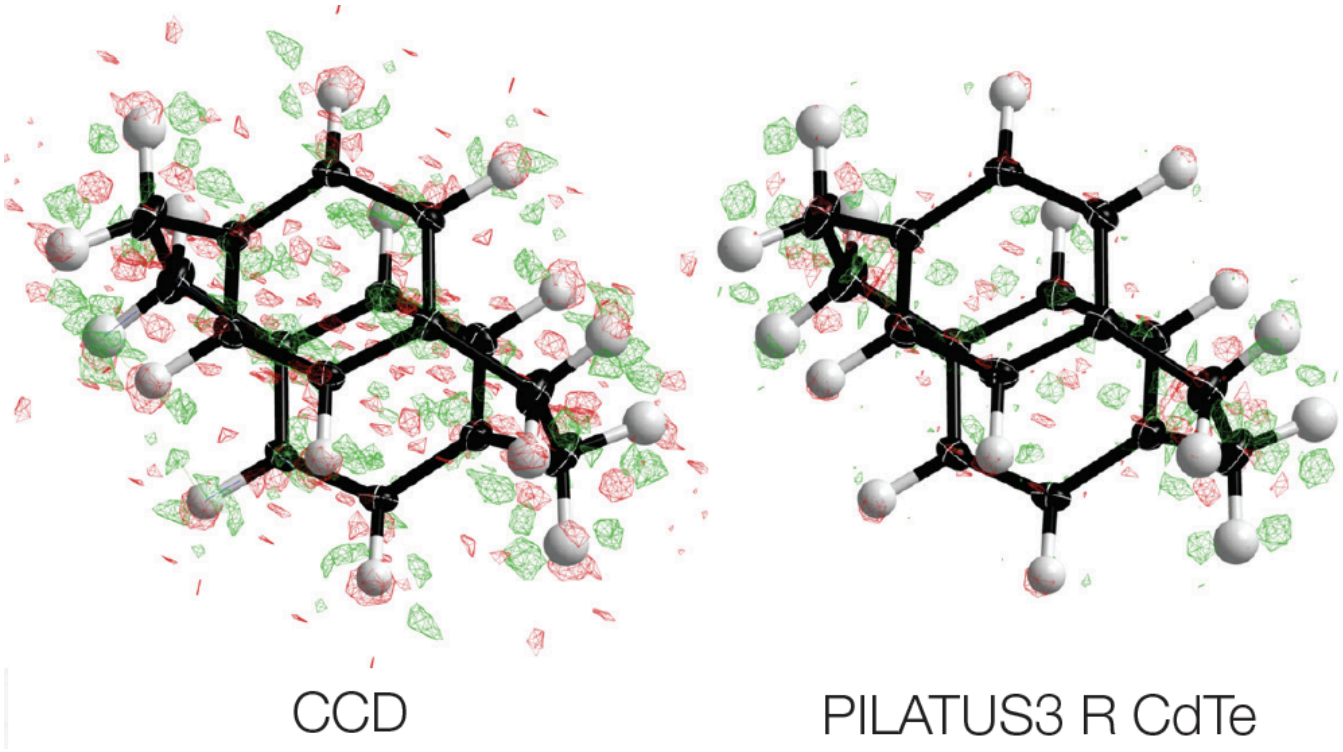
PILATUS3 R CdTe

Courtesy Dietmar Stahlke (Göttingen)

High-quality charge density

Paracyclophane

– *Isolevels at ± 0.047 e Å^{-3}*

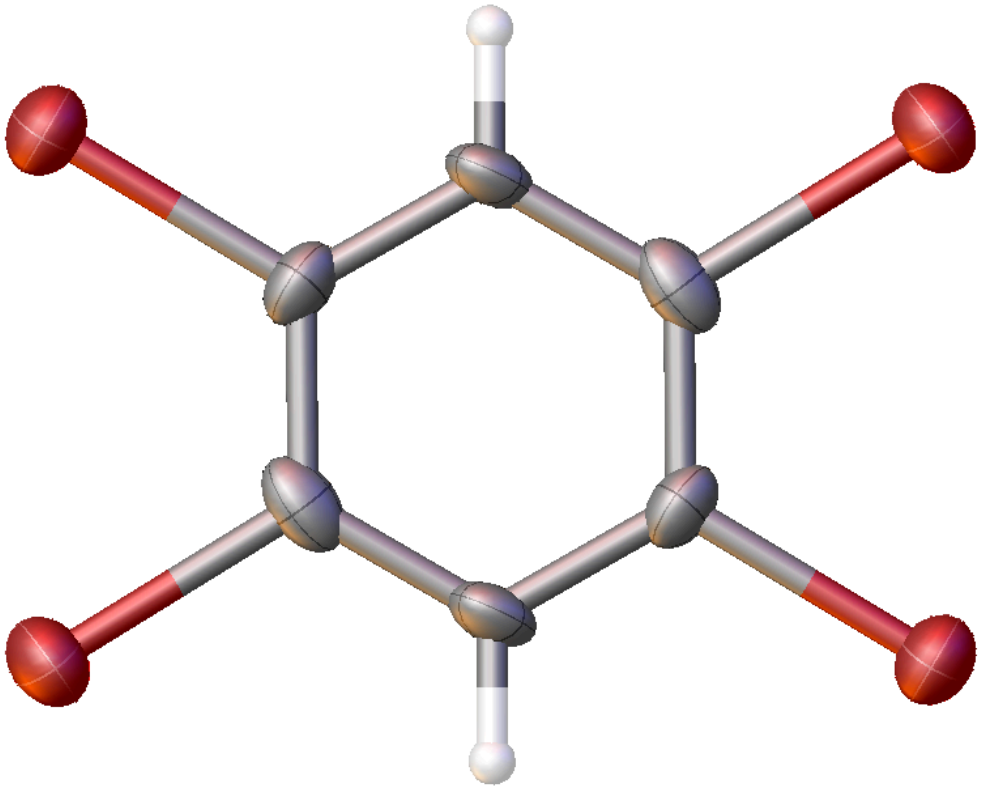


Courtesy Dietmar Stahlke (Göttingen)

Crystallography at high pressure

1,2,3,4-tetrabromobenzene

- *Material with mechanical response to heating*
- *Data collected in diamond anvil cell at 0.4 GPa*

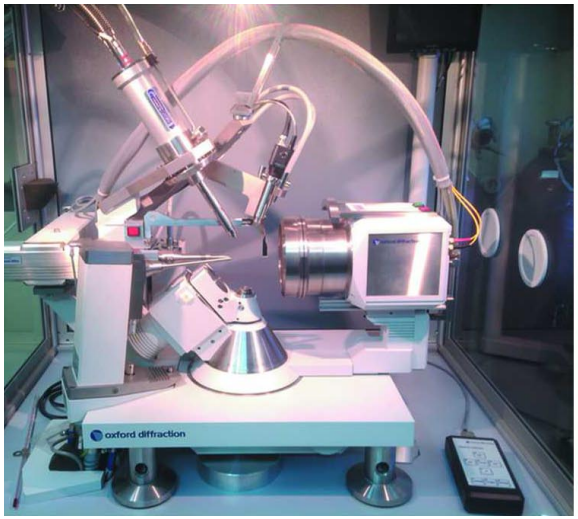


Courtesy Elena Boldyreva (Novosibirsk)

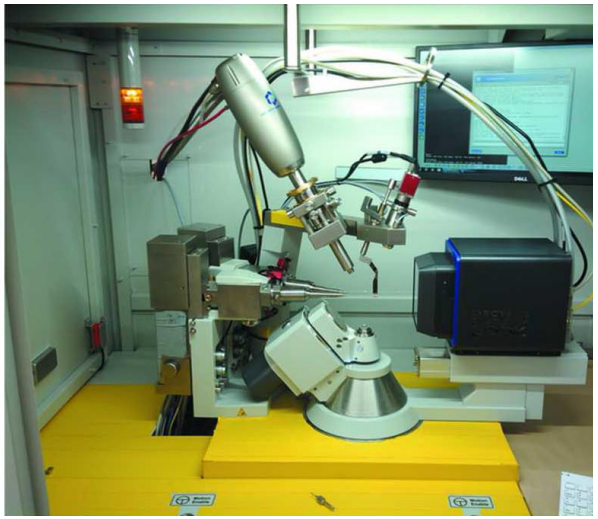
Better kit for better data

Ten years of innovation

- *Ag radiation for higher reciprocal space coverage*
- *HPC detector for higher-accuracy intensities*
- *CdTe sensor for high quantum efficiency*



Oxford Diffraction Gemini
Mo radiation
Ruby CCD



Rigaku OD XtalLAB Synergy-S
Ag radiation
PILATUS3 R CdTe 300K

Acta Cryst. (2018). E74, 613–619

Better kit for better data

Ten years of innovation

- *Ag radiation for higher reciprocal space coverage*
- *HPC detector for higher-accuracy intensities*
- *CdTe sensor for high quantum efficiency*

	(*e*) Mo, *ABSORB-7*	(*b*) Ag, *ABSORB-7*
Crystal data		
a, b, c (Å)	3.9431 (5), 10.7566 (18), 9.964 (2)	3.9390 (9), 10.781 (4), 9.944 (4)
β (°)	100.557 (15)	100.49 (3)
V (Å^3)	415.47 (13)	415.2 (2)
Radiation type	Mo $K\alpha$	Ag $K\alpha$, λ = 0.56087 Å
No. of reflections for cell measurement	514	748
θ range (°) for cell measurement	2.8–22.4	2.2–22.9
μ (mm^{-1})	19.29	10.33
Absorption correction	Gaussian	Gaussian
T_{min}, T_{max}	0.361, 0.434	0.486, 0.562
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2116, 531, 313	2445, 870, 496
R_{int}	0.103	0.048
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.663	0.801
Range of h, k, l	$h = -5 \rightarrow 5$, $k = -12 \rightarrow 11$, $l = -10 \rightarrow 10$	$h = -5 \rightarrow 6$, $k = -14 \rightarrow 14$, $l = -11 \rightarrow 12$
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.071, 0.169, 1.05	0.037, 0.073, 0.93
No. of reflections	531	870

Experiment time:
Ruby CCD: 32 hrs
PILATUS3: 6 hrs

Summary

Direct detection for sharp spots

Photon counting for accuracy and speed

Adjustable threshold to cut fluorescence

CdTe sensors for high X-ray energies

DECTRIS®

detecting the future

Merci pour votre attention!

www.dectris.com

andreas.foerster@dectris.com

DECTRIS Ltd.
5405 Baden-Dättwil
Switzerland
www.dectris.com