

**DECTRIS<sup>®</sup>**

*detecting the future*

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MYTHEN

PILATUS

EIGER

# ***Hybrid Photon Counting detectors for synchrotron and lab sources***

***ANF Réciprocs – 2016-10-11***

***Andreas Förster – Application Scientist***

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Switzerland  
[www.dectris.com](http://www.dectris.com)

# ***Outline***

***Technology***

***Direct detection***

***Photon counting***

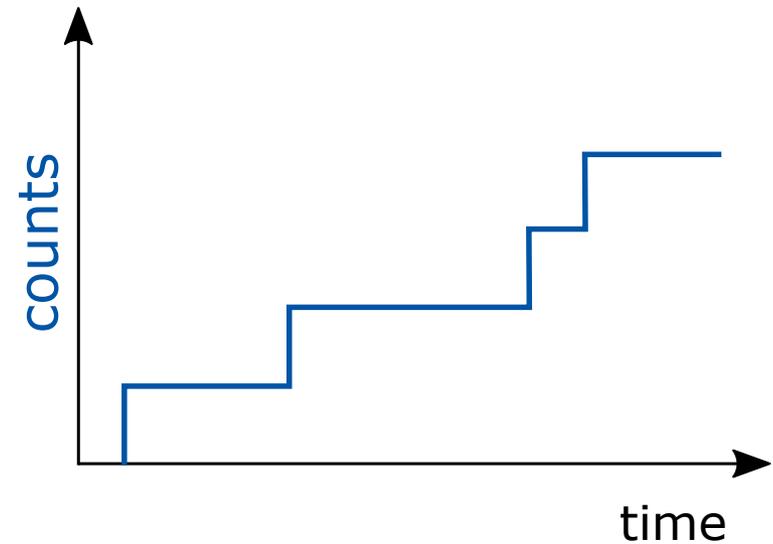
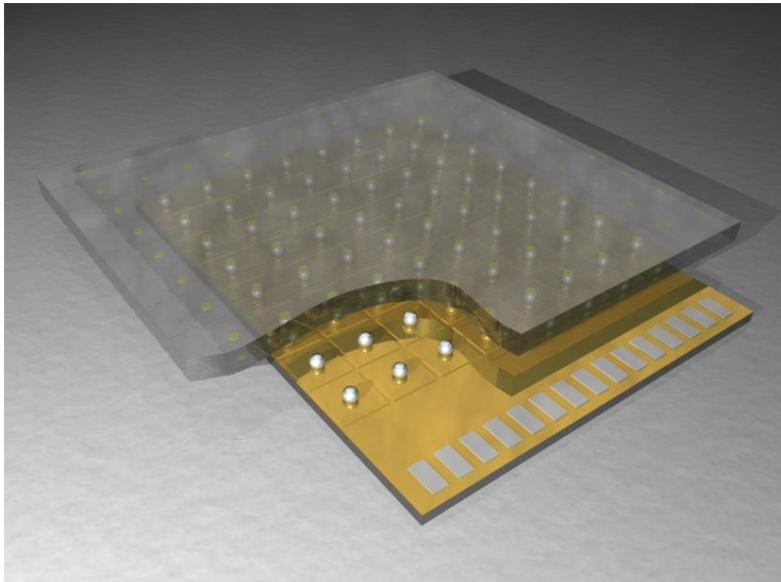
***X-ray powder diffraction***

***The power of the threshold***

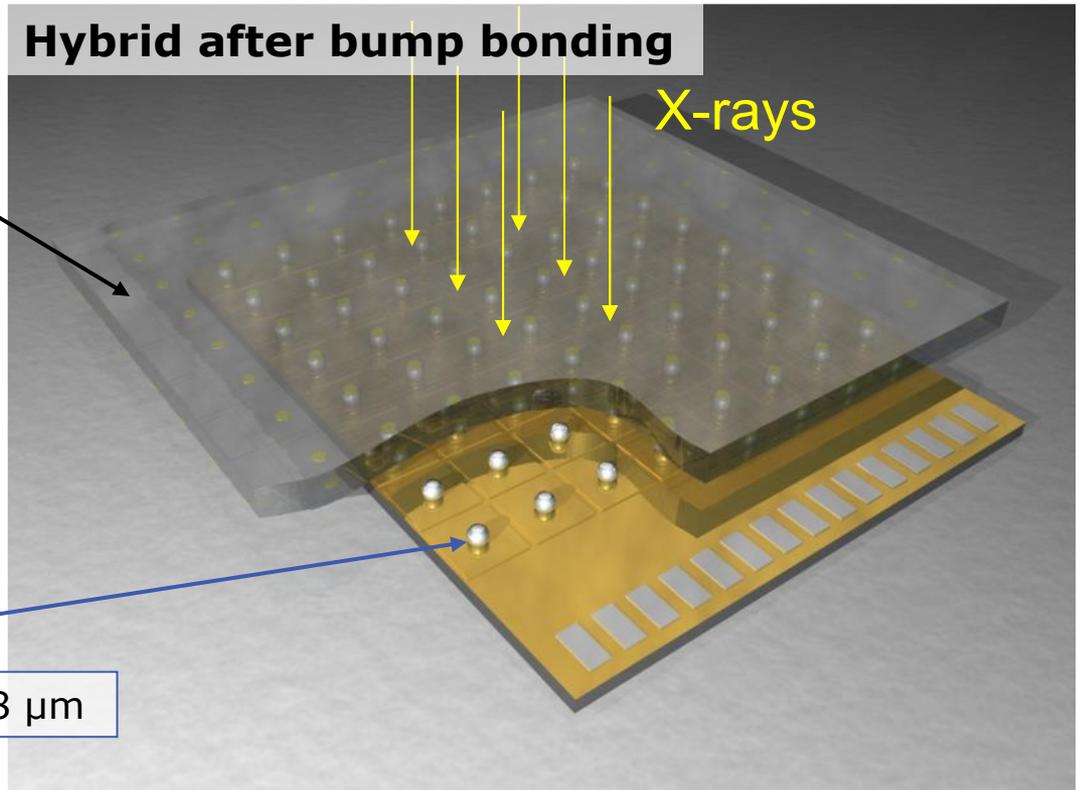
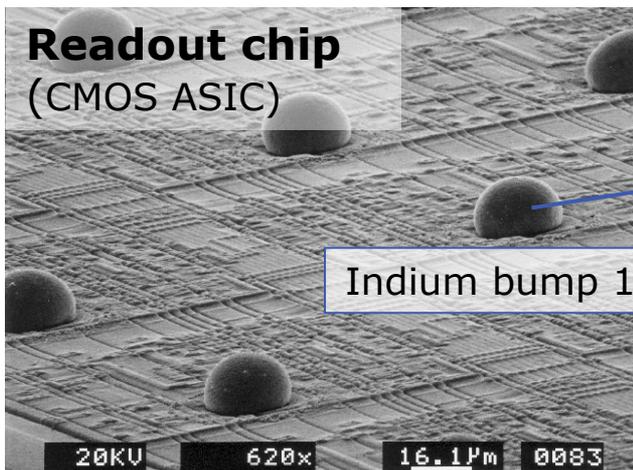
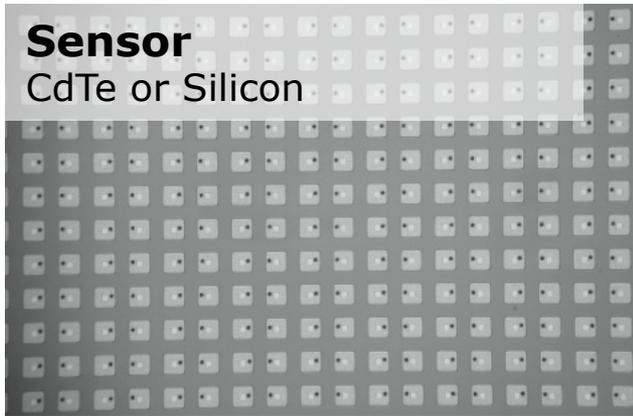
***Challenging MX in the lab***

# *The meaning of HPC counting*

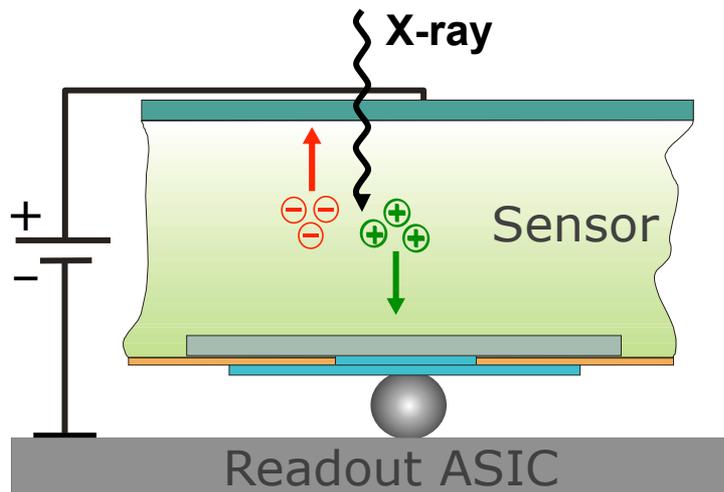
## *Hybrid pixel + single Photon Counting*



# Hybrid pixel = sensor + readout



# Direct detection in hybrid pixels



## Sensor pixel

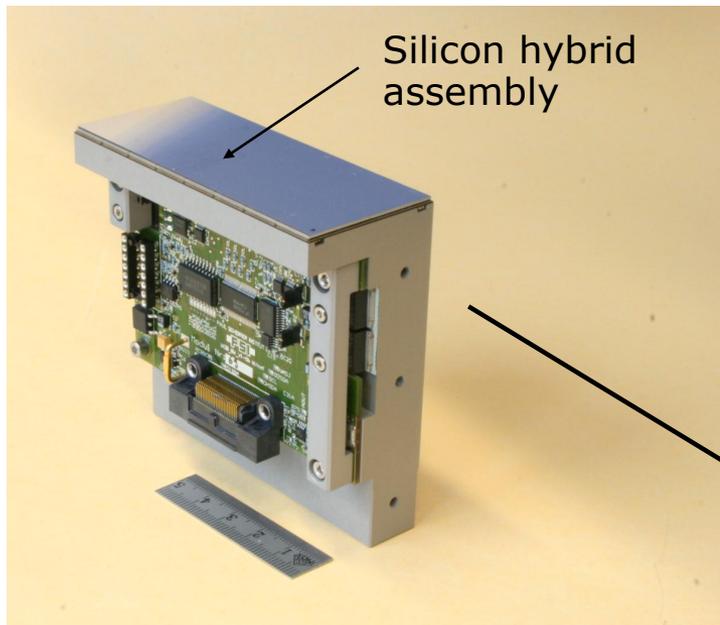
- Absorption of X-ray photons  
-> 3.6 eV per electron
- Charge is captured by electric field

## Pixel electronics

- Counting of charge pulses

# Modular assembly of HPC detectors

*PILATUS detectors are arrays of PILATUS modules.*



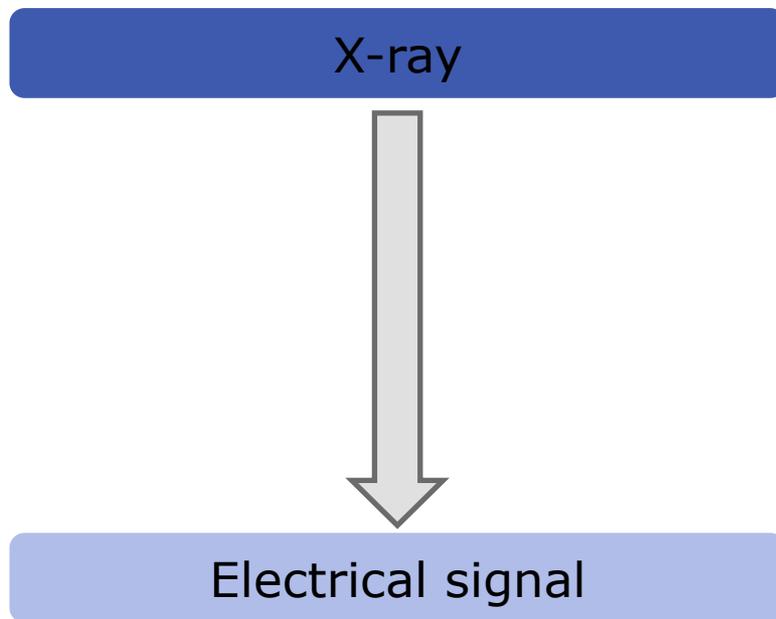
*Module active area:  $83.8 \times 33.6 \text{ mm}^2$*

*$487 \times 195 \text{ pixels} = \sim 100\text{k pixels}$*

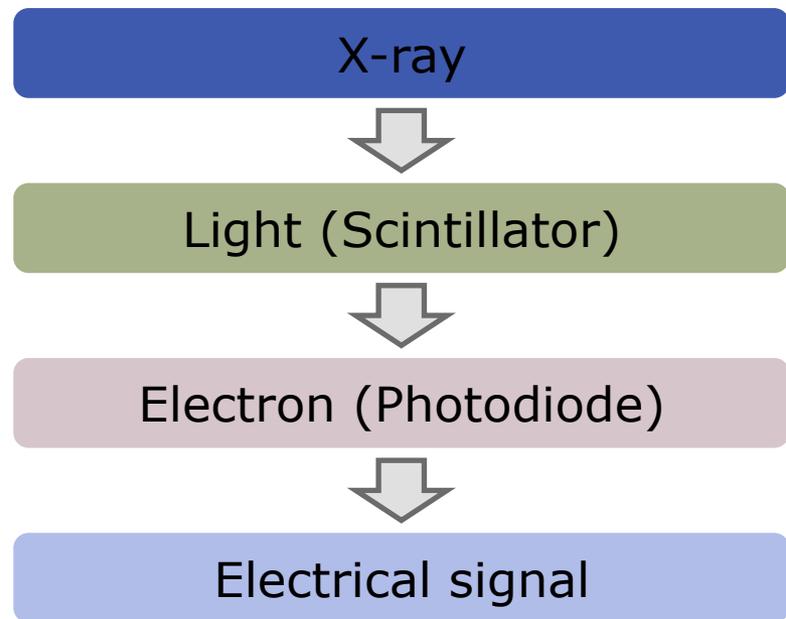
*Pixel pitch  $172 \mu\text{m}$*

# Superiority of direct detection

## Direct detection

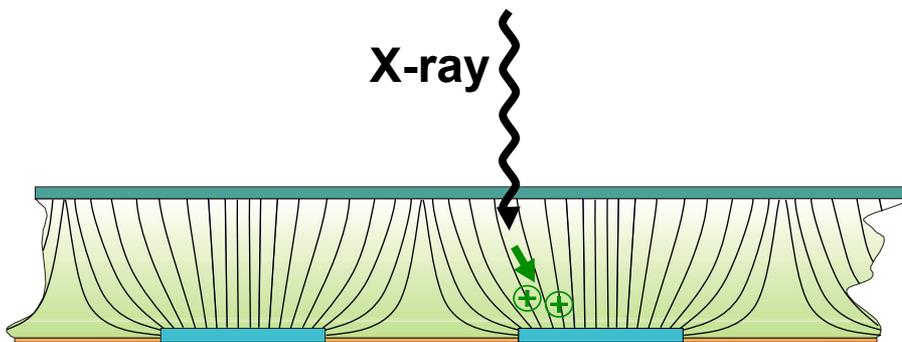


## Indirect detection



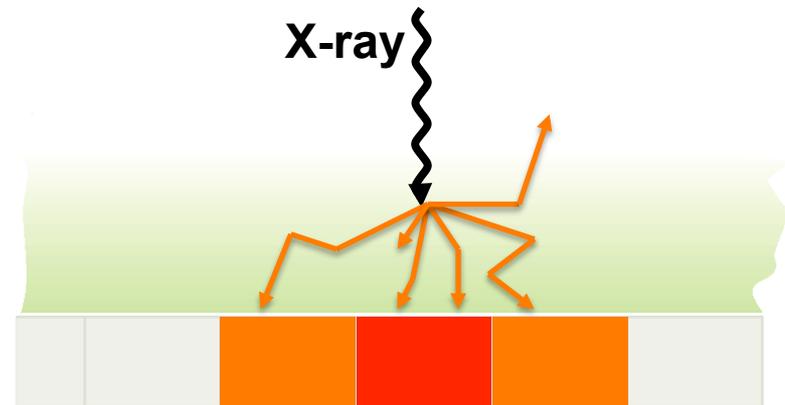
# Superiority of direct detection

## Direct detection



- Charge captured in electric field
- => **All photons counted**
- => **Signal remains in pixel**

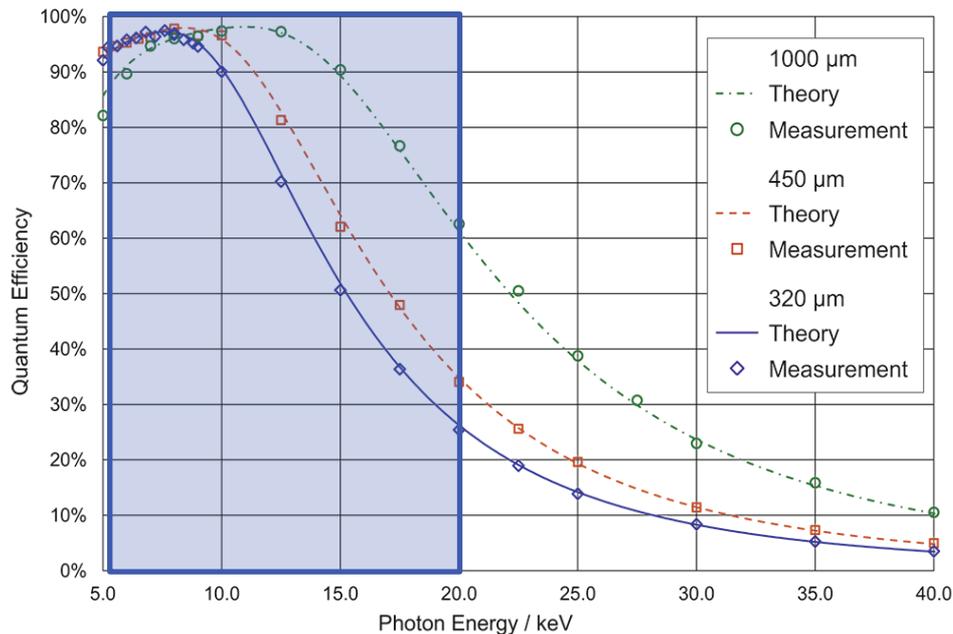
## Indirect detection



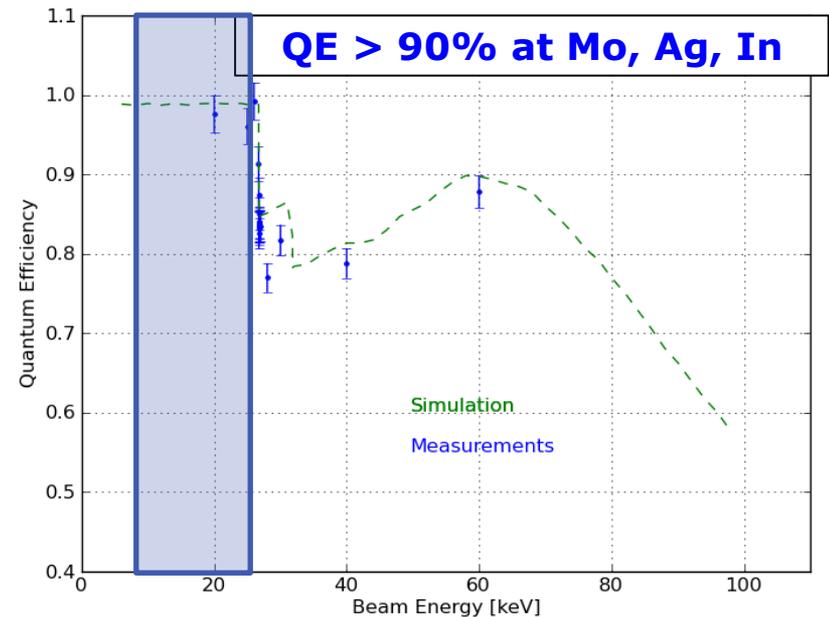
- Secondary radiation scattered in scintillator
- => **Signal spread across pixels**
- => **Light partially lost**

# Silicon and CdTe sensors

High quantum efficiency across a wide energy range



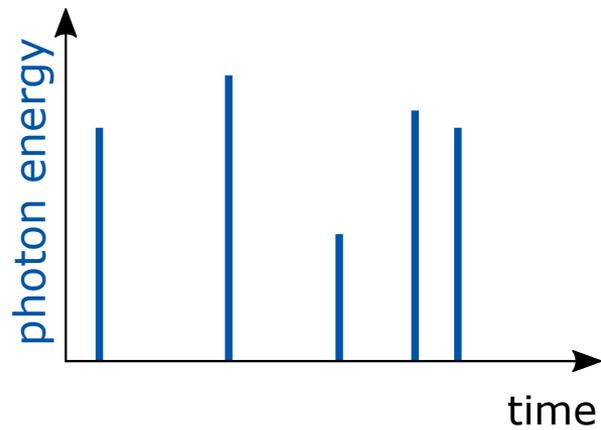
Silicon for low energies



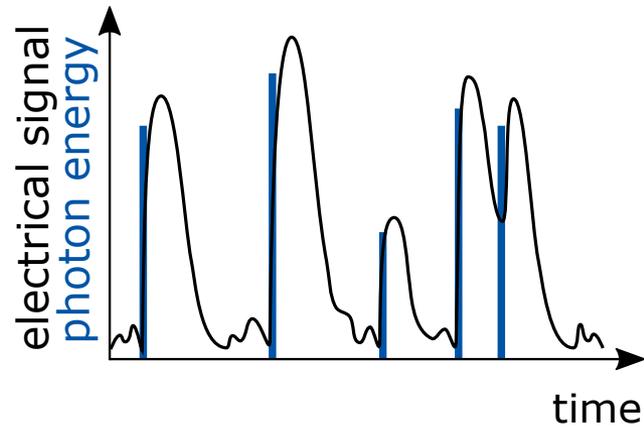
CdTe for high energies

# Photon counting – one by one

– Photons absorbed in sensor pixel

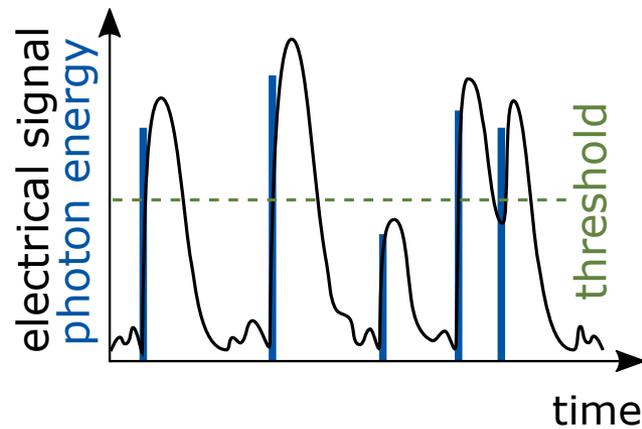


# Photon counting – one by one



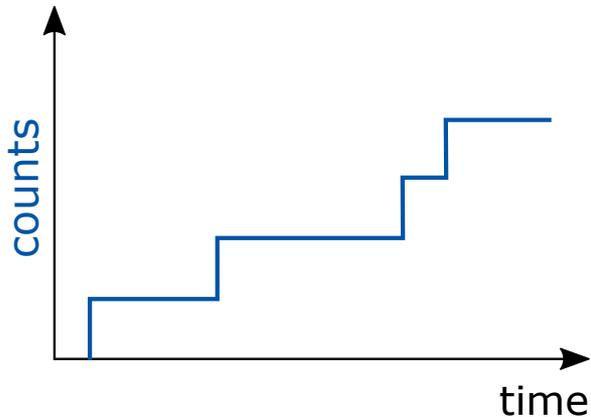
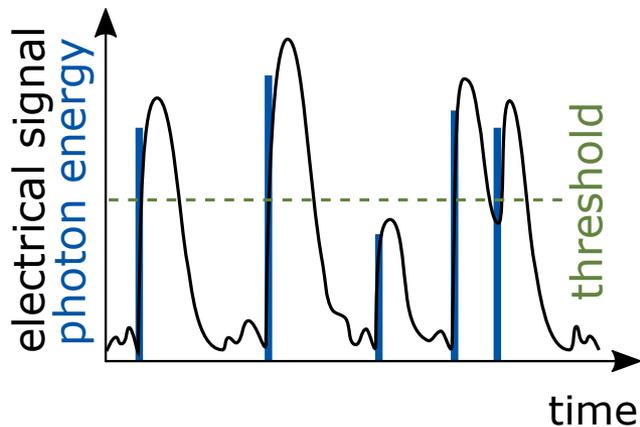
- Photons absorbed in sensor pixel
- Charge pulse proportional to energy

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

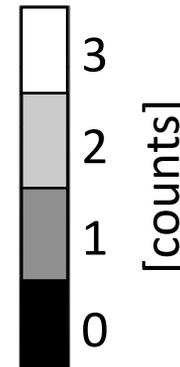
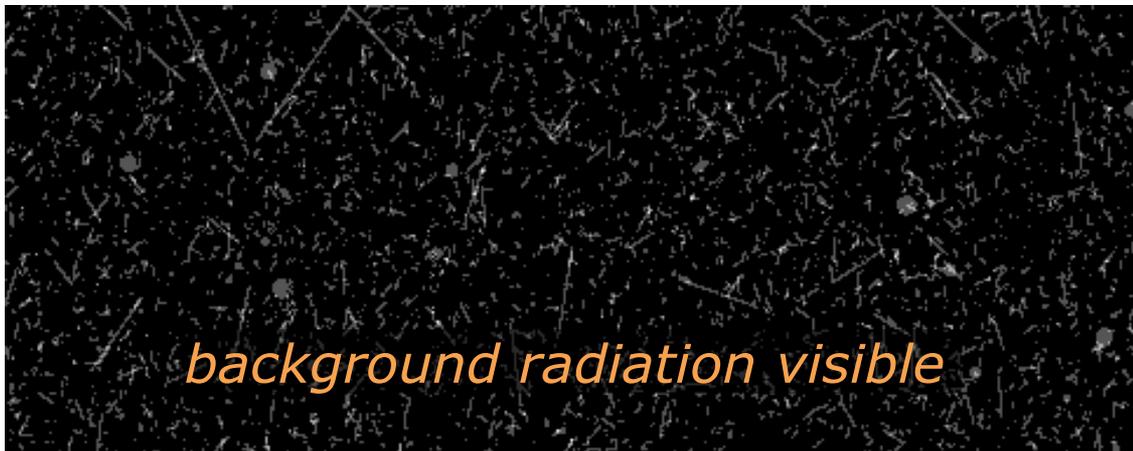
# Noise-free detection

100 ms



**No readout noise**  
**No dark signal**

1 hour

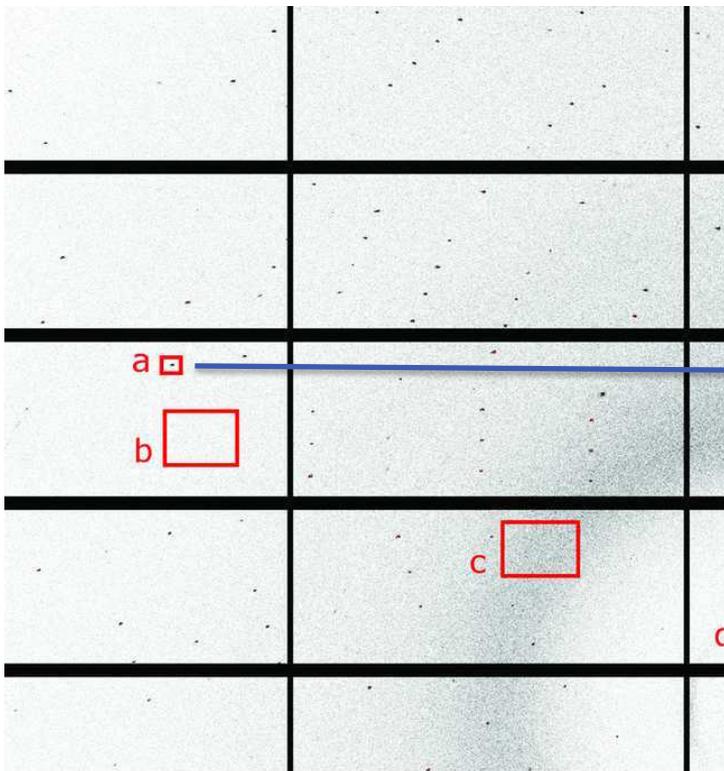


100K detector

# Negligible background for max. $I/\sigma(I)$

## Protein crystallography in vacuum

Thaumatin @ I23.



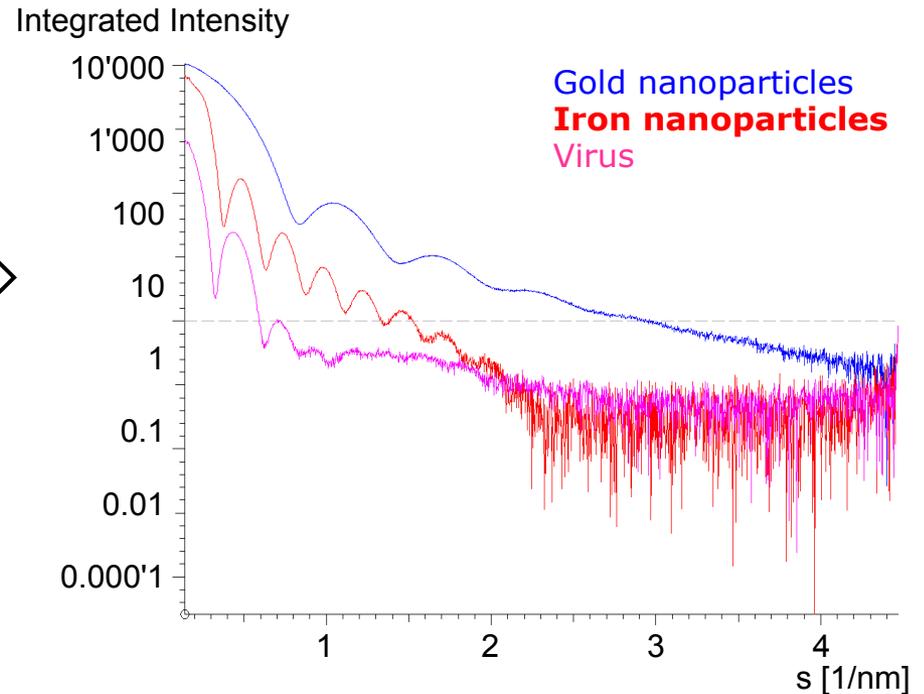
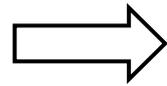
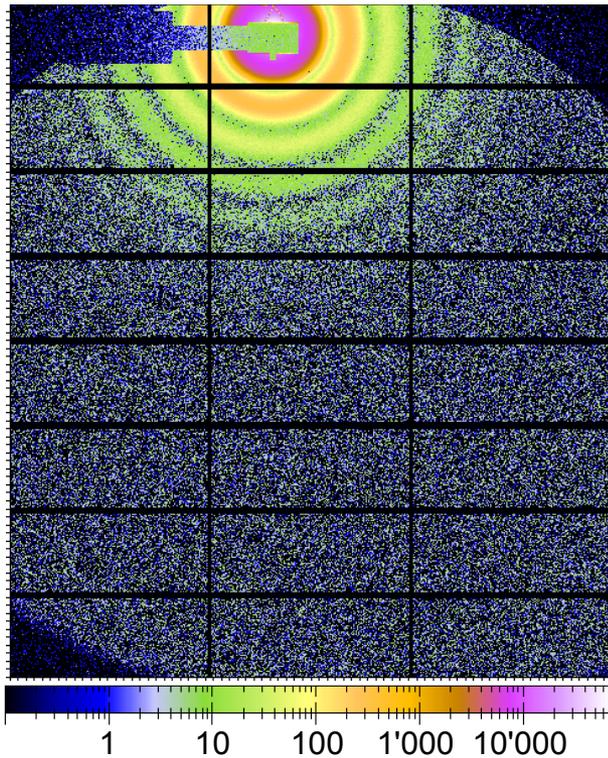
0	1	1	2	2	1	2	1	2	3	0	2	1	0	2	3	0	1	1	2
2	1	1	1	0	2	0	0	1	0	0	1	1	2	2	1	0	0	2	0
2	2	1	1	1	0	1	0	3	0	0	1	2	1	0	0	1	0	1	2
3	0	0	1	0	0	0	0	2	2	2	1	0	1	0	2	0	0	1	1
2	0	3	1	0	0	1	0	2	0	0	1	2	1	1	1	1	0	2	3
0	0	1	1	0	1	0	1	0	0	1	0	2	0	1	0	1	0	2	1
0	0	1	0	1	2	0	1	1	1	0	0	1	1	0	2	2	0	2	1
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2	1	1	0	2	2	1	6	33	147	359	119	2	2	2	0	0	0	2	1
1	5	0	0	3	1	2	8	36	30	35	31	1	1	1	2	0	2	1	0
1	1	1	0	1	1	3	2	1	4	2	4	1	0	0	2	1	1	1	0
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0	3	1	2	0	0	1	1	0	1	2	1								1
0	0	0	0	0	2	0	3	1	1	0	1	0	0	0	0	2	0	1	0

Wagner et al. (2016)

# Best SAXS data from HPC detectors

Low noise background for correct intensities at large angles

High dynamic range for contrast



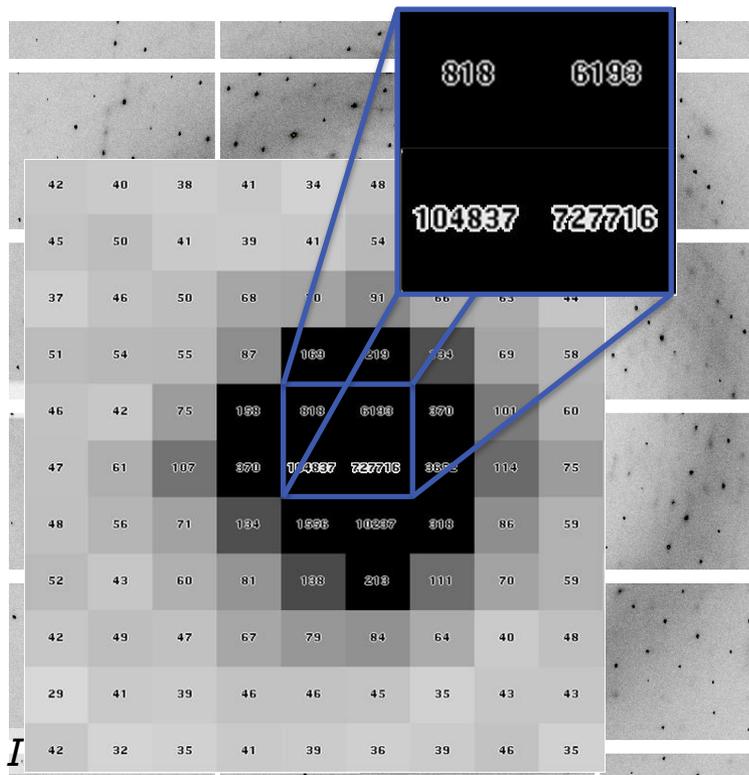
Manfred Rössle (BioSAXS @ PETRA III)

# Dynamic range for strong reflections

## HPC detectors

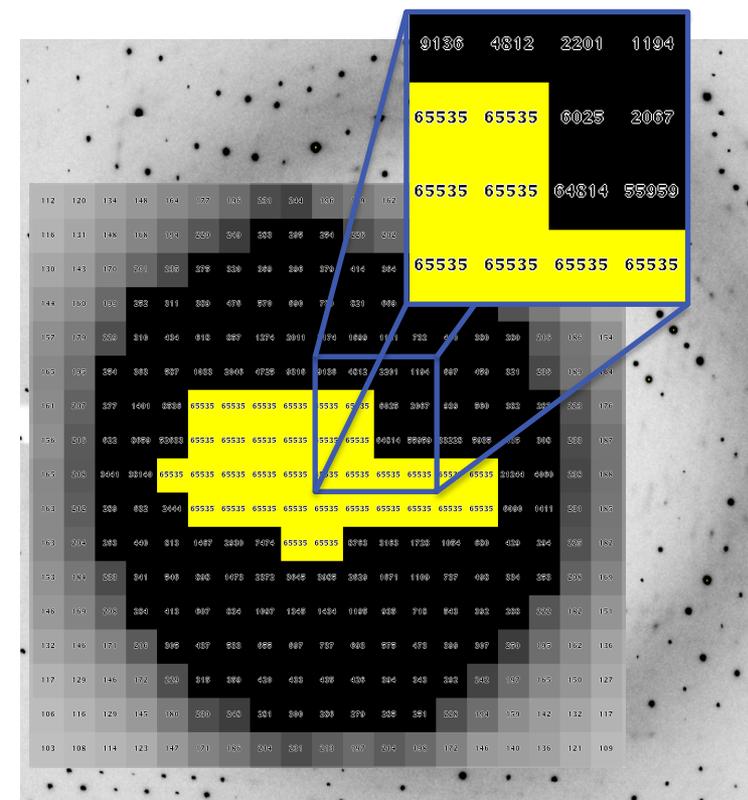
PILATUS @ 20 bit (1 million counts)

EIGER @ 32 bits (4 billion counts)



## Scintillator-based detectors

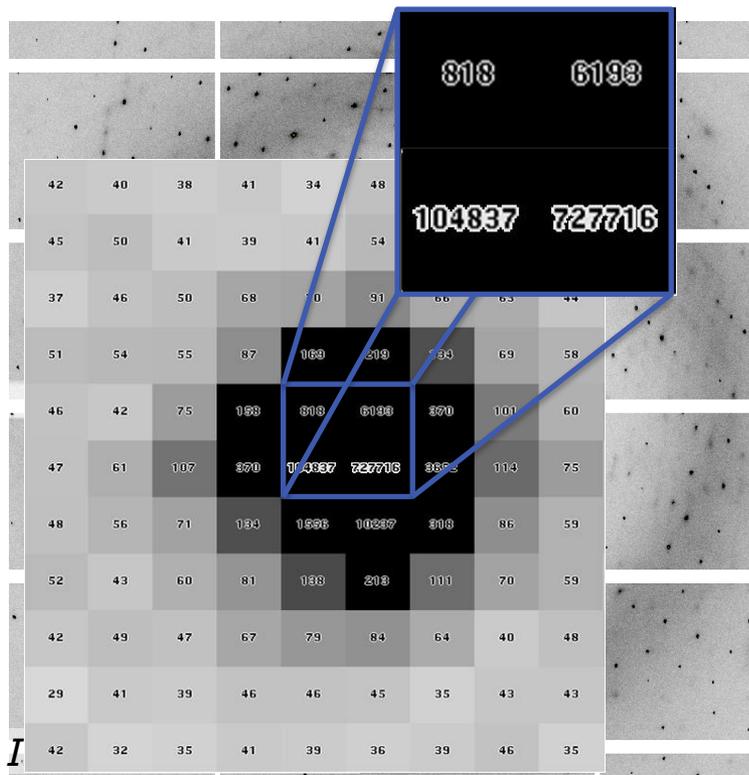
CCD @ 16 bits (65 thousand ADU)



# Excellent spatial resolution

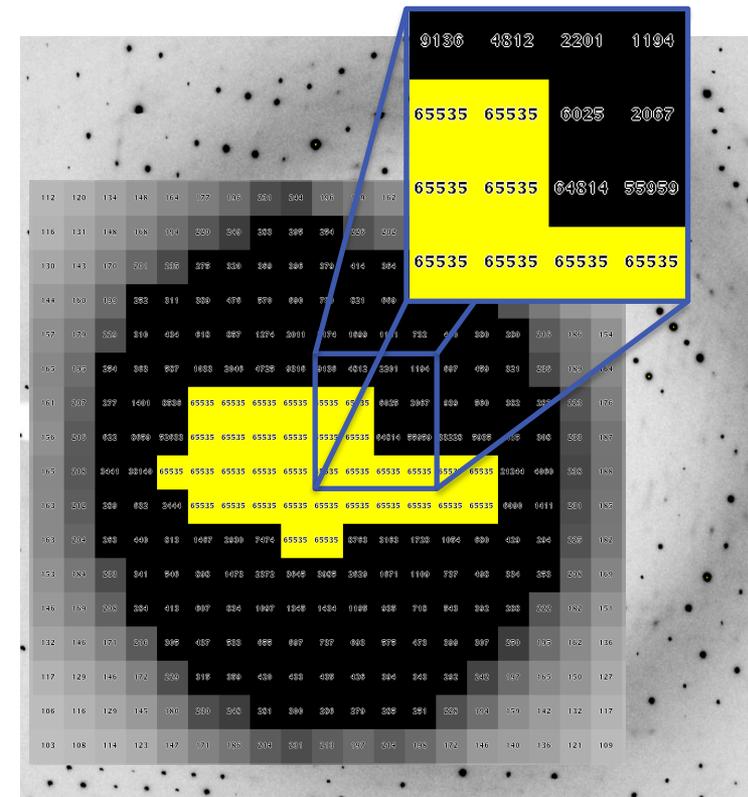
## HPC detectors

Sharp point spread function



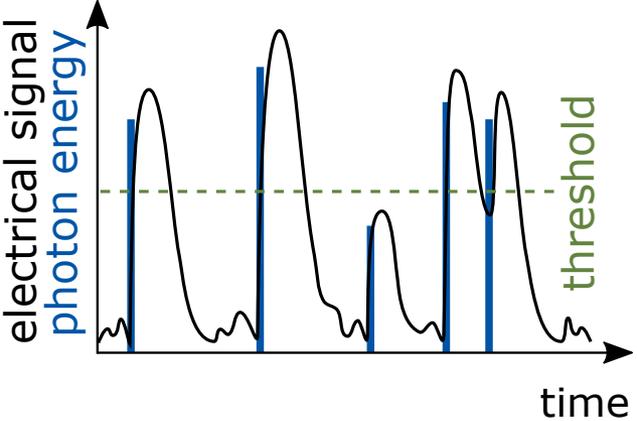
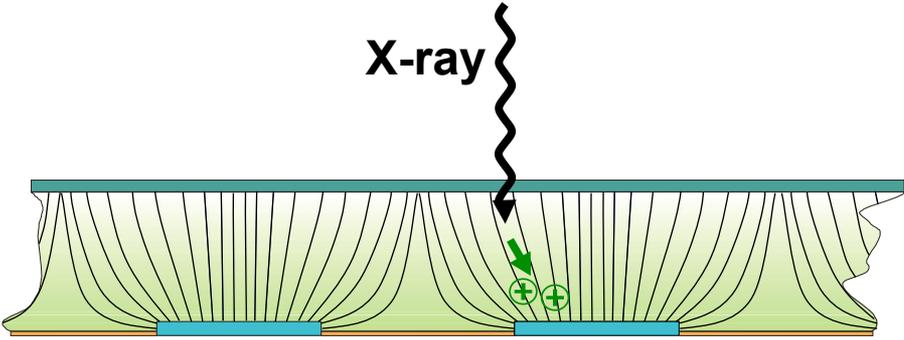
## Scintillator-based detectors

Visible light dilutes signal



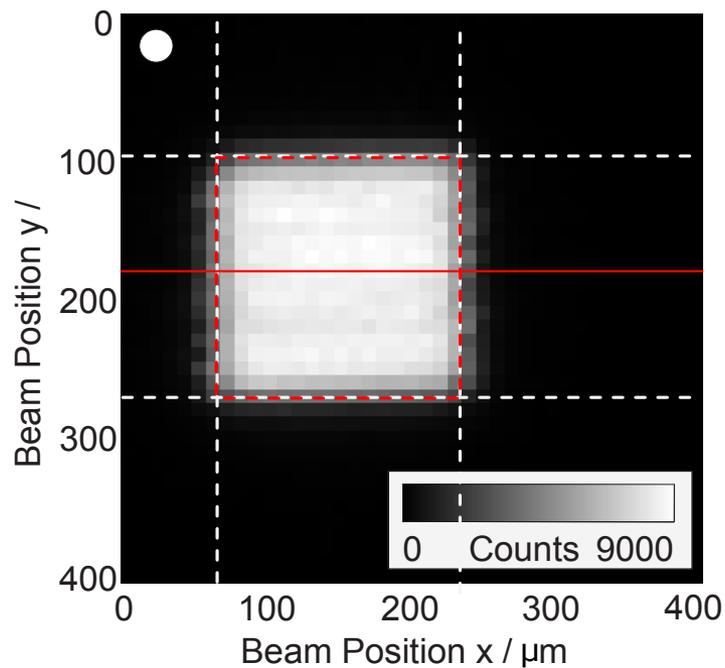
# Excellent spatial resolution

Direct detection and threshold for sharp point spread

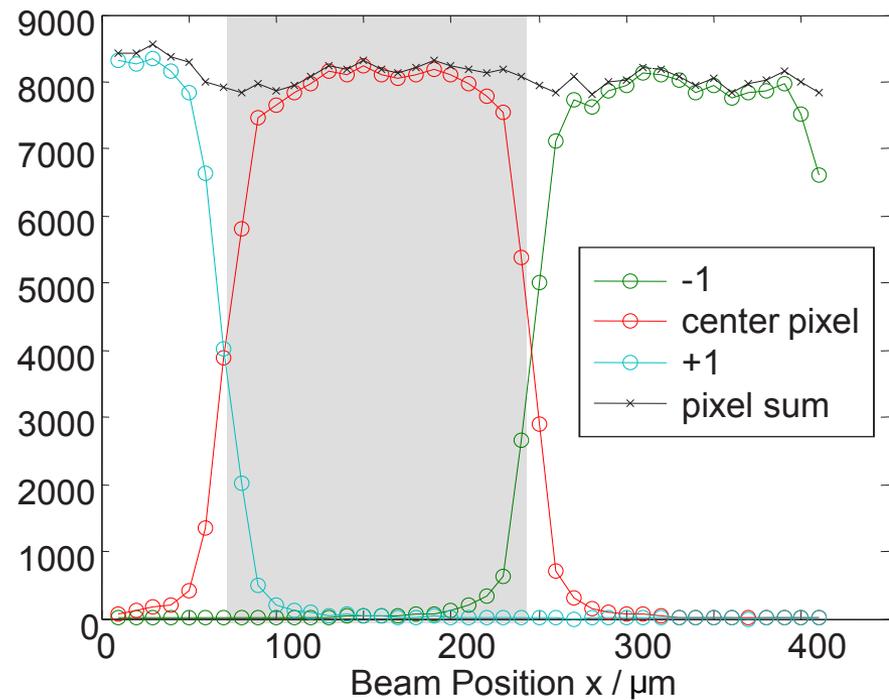


# Spatial resolution PILATUS3

All signal contained with pixel

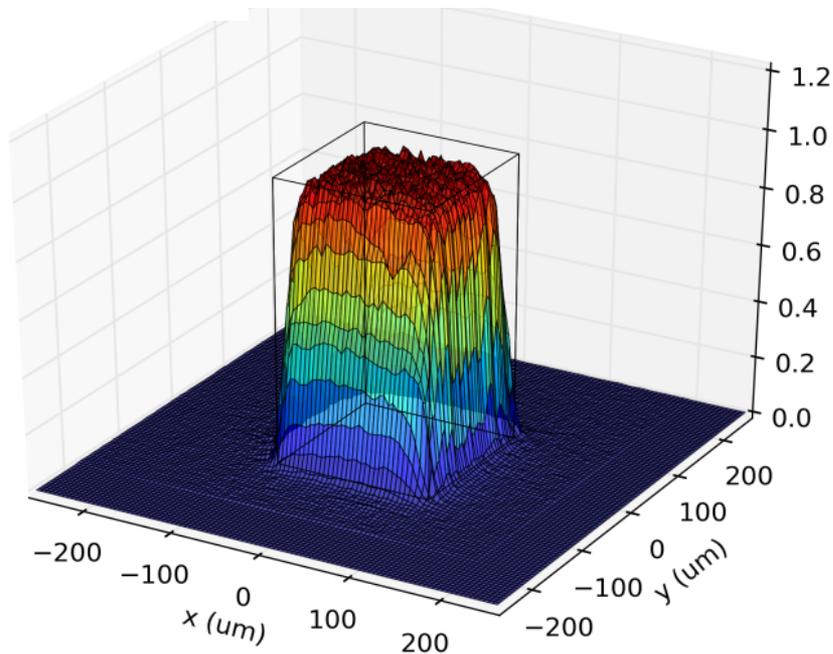


20  $\mu\text{m}$  pencil beam, 10  $\mu\text{m}$  rastering

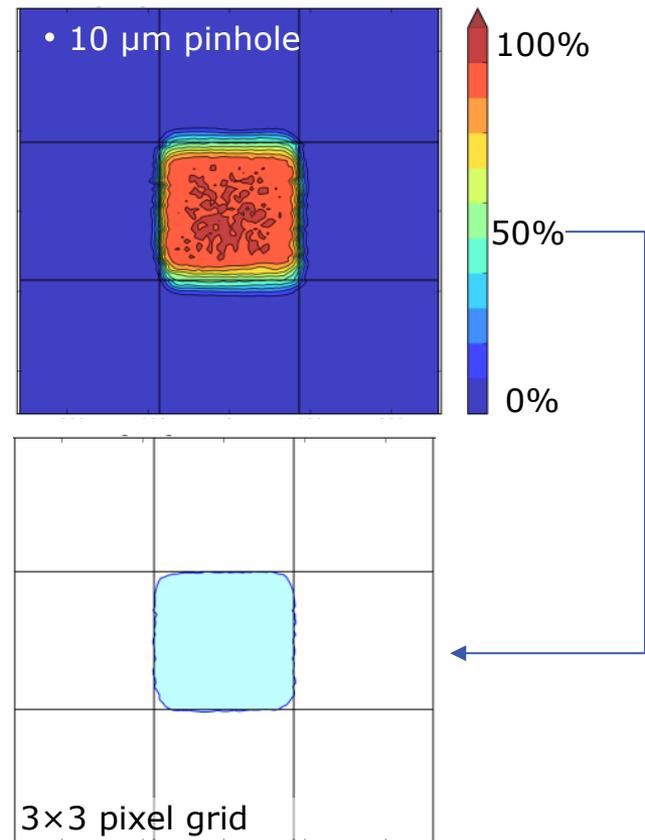


# Spatial resolution PILATUS3 CdTe

For CdTe as for silicon



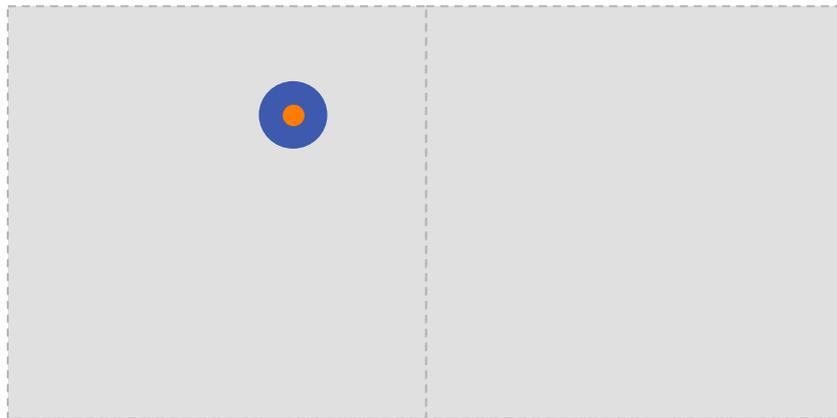
10 µm pinhole at Ag K $\alpha$  wavelength



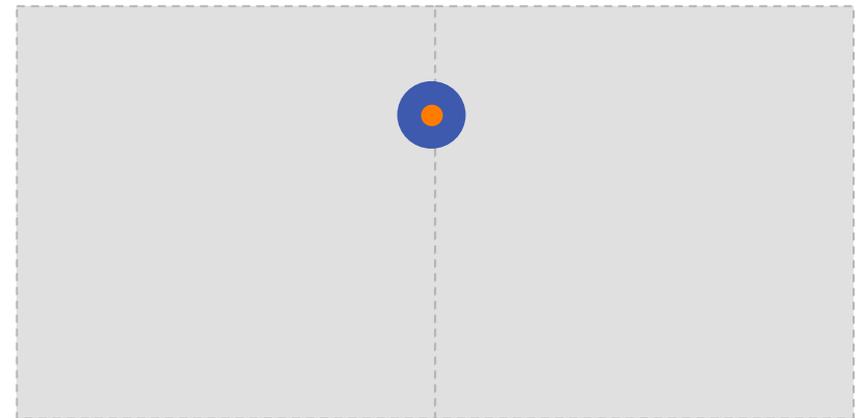
# Threshold at half photon energy

*All signal counted within on pixel*

*Electronic noise + fluorescence filtered*



1 photon  
100% signal  
1 count



1 photon  
60% signal  
1 count

40% signal  
-

# MYTHEN2 – 1D detectors for XRPD



*MYTHEN2 R  
for laboratories*

*MYTHEN2 1D – 640 strips  
MYTHEN2 1K – 1280 strips*



*MYTHEN2 X  
for synchrotrons*

*50  $\mu$ m strips  
up to 4 detectors per DCS*

# PILATUS with Si sensor

*PILATUS3 R  
for laboratory*

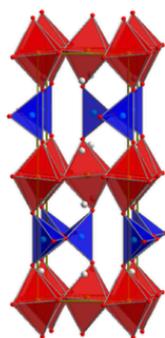


*PILATUS3 S/X  
for synchrotrons*

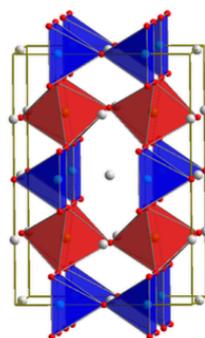


# Electrochemical oxidation of $\text{SrFeO}_{2.5}$

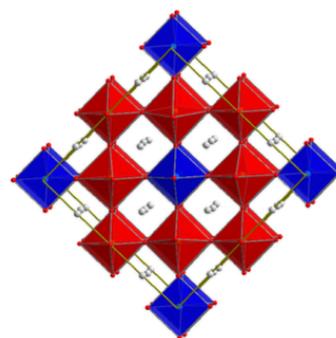
$\text{Fe}^{3+}$   
 $\text{SrFeO}_{2.5}$



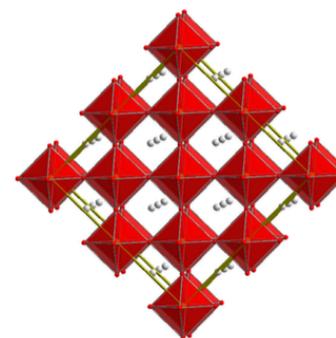
$\text{Fe}^{3+}/\text{Fe}^{4+}$   
 $\text{SrFeO}_{2.75}$



$\text{Fe}^{4+}/\text{Fe}^{3+}$   
 $\text{SrFeO}_{2.875}$

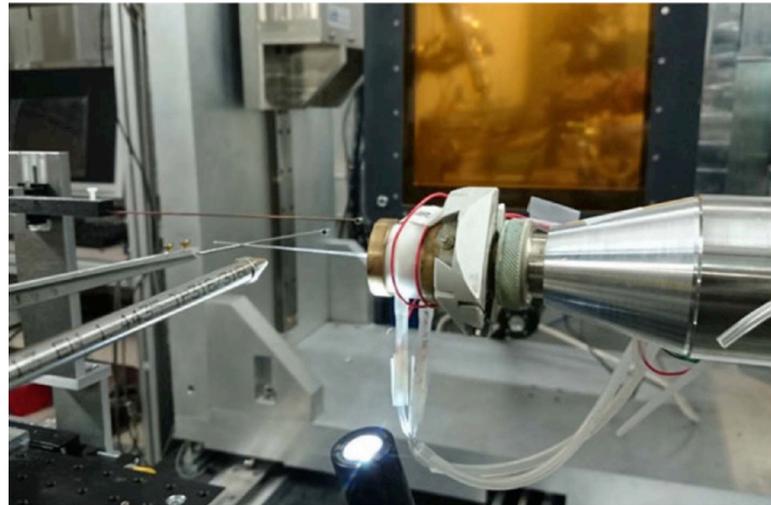


$\text{Fe}^{4+}$   
 $\text{SrFeO}_3$



*Thanks to Werner Paulus  
(Université de Montpellier)*

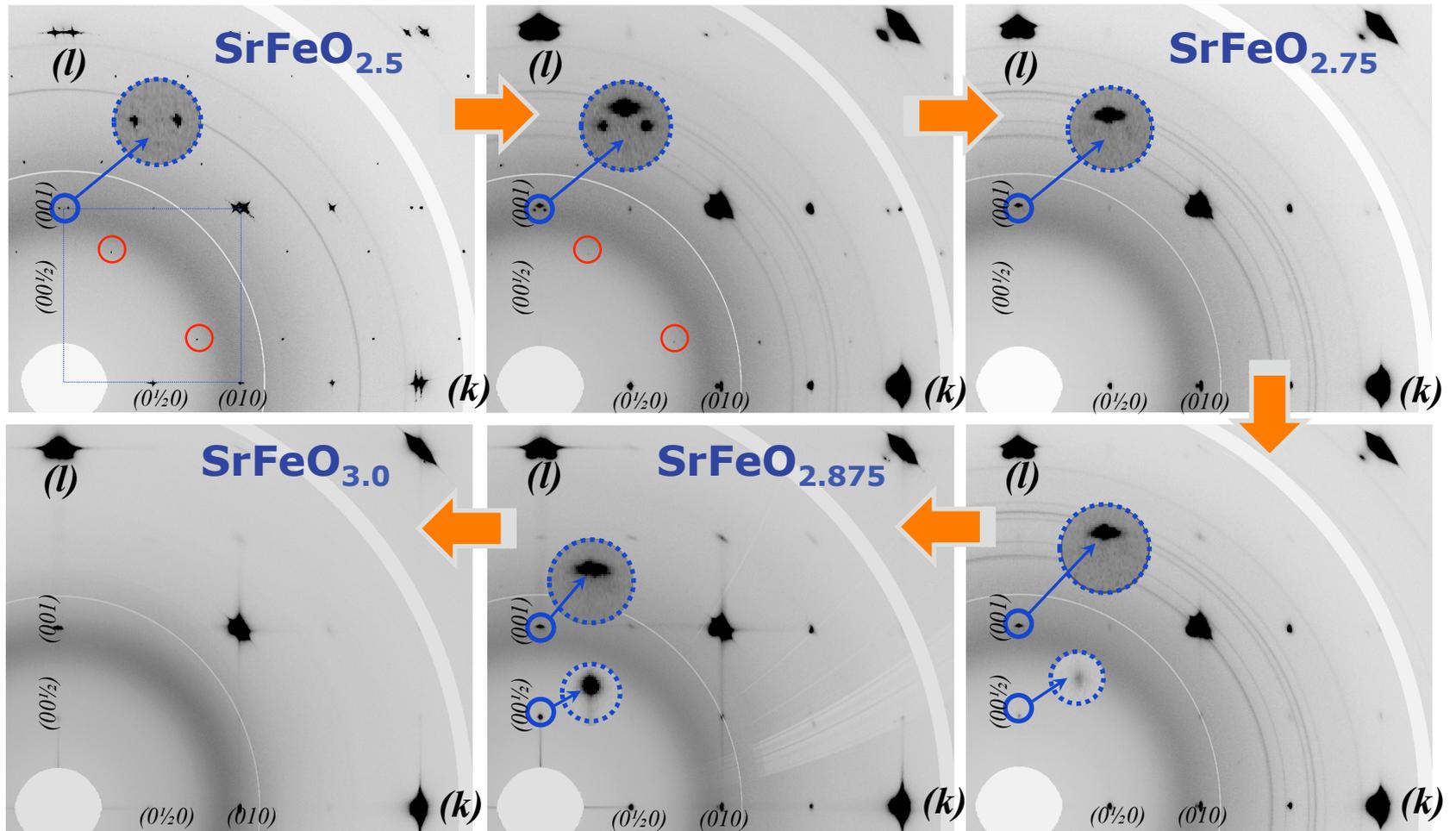
# *Electrochemical cell at BM01*



*Swiss-Norwegian beamline (BM01) at ESRF*

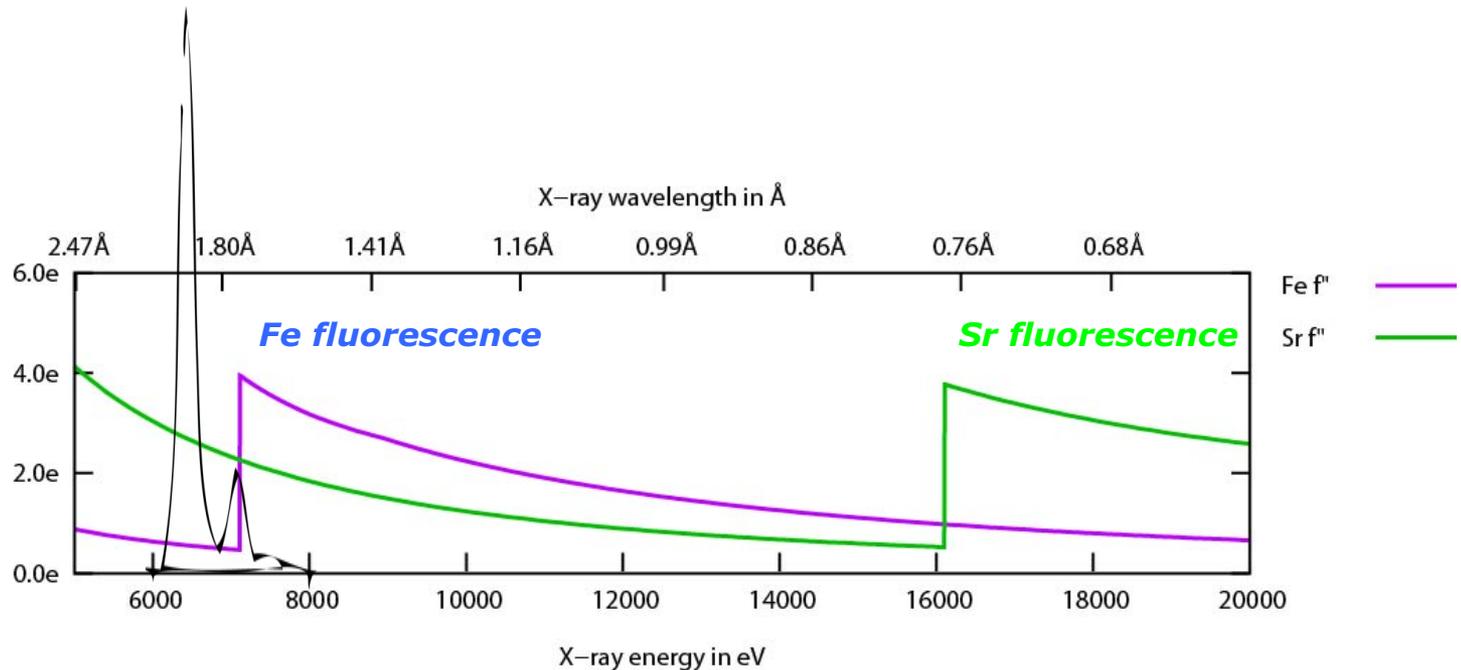
*also STOE Stadivari Mo  
with PILATUS 200K*

# Evolution of reflections in $0kl$ plane



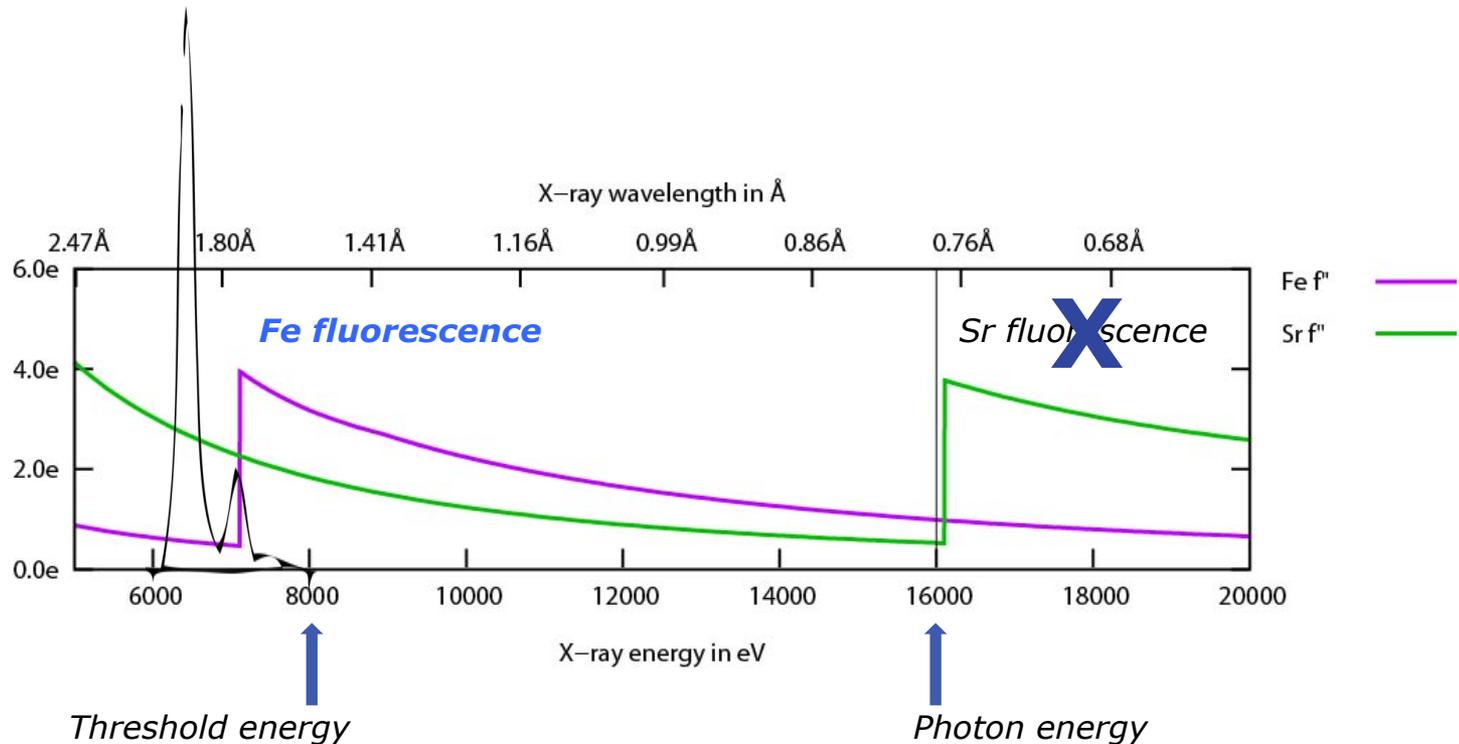
$t = 5$  days

# Threshold suppresses fluorescence



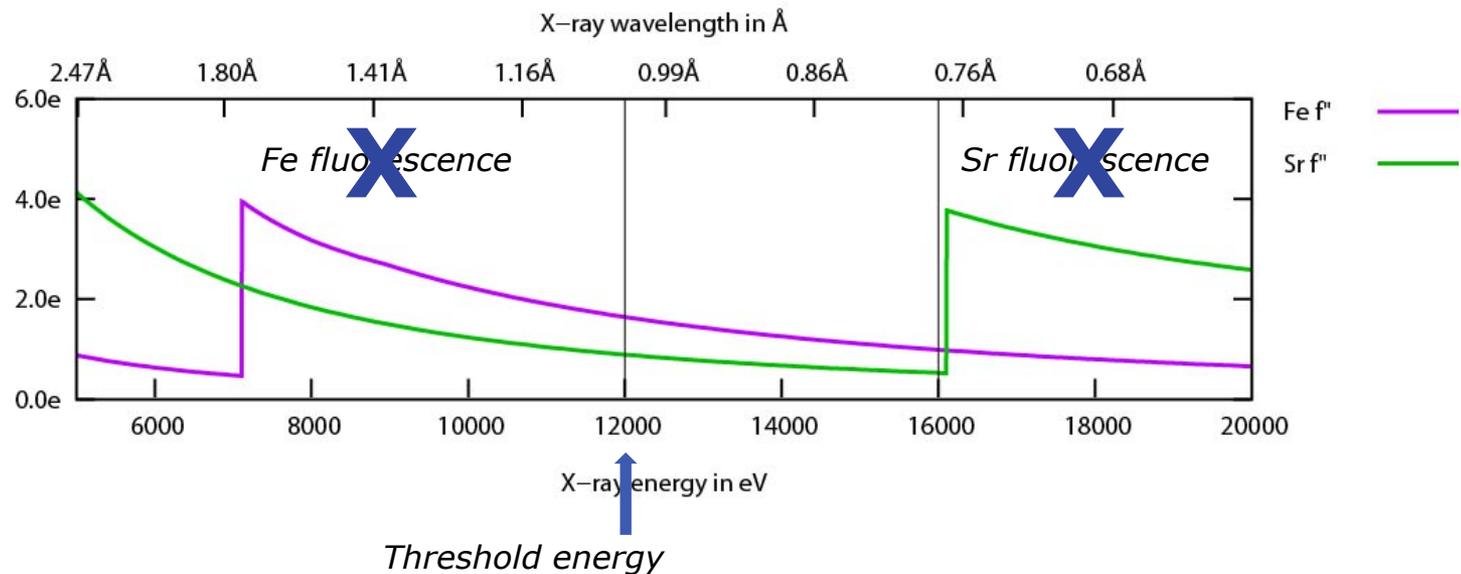
taken from <http://skuld.bmsc.washington.edu/scatter/>

# Threshold suppresses fluorescence



taken from <http://skuld.bmsc.washington.edu/scatter/>

# Threshold suppresses fluorescence



taken from <http://skuld.bmsc.washington.edu/scatter/>

# PILATUS3 CdTe detectors

*PILATUS3 R CdTe  
for laboratories*

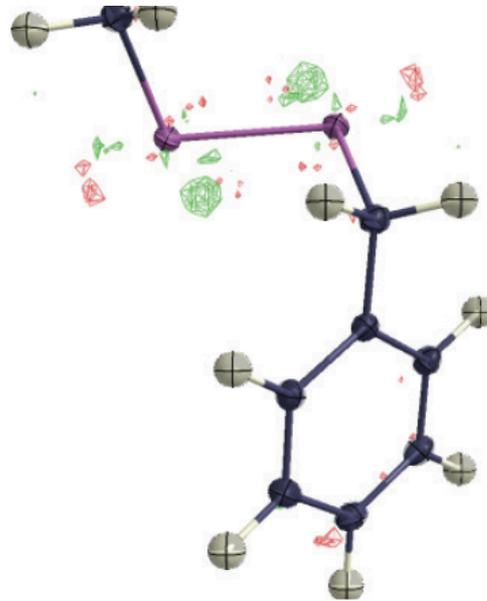


*PILATUS3 X CdTe  
for synchrotrons*



*25 x 28 cm<sup>2</sup>*

# *Charge density studies*

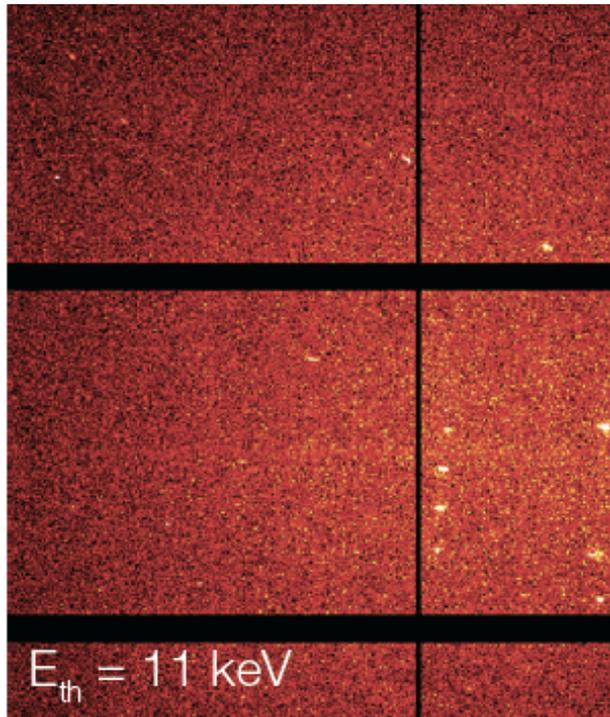


*Thanks to Dietmar Stahlke  
(Universität Göttingen)*

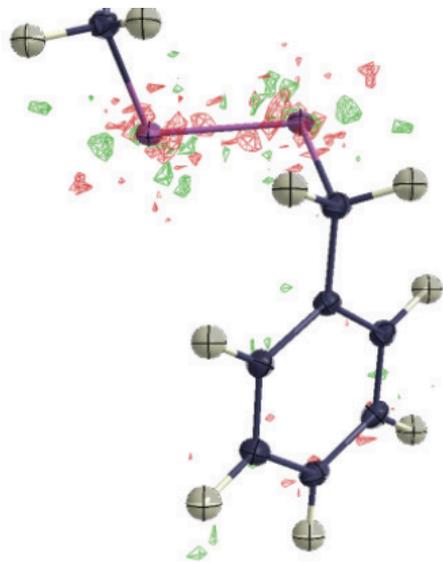
# Fluorescence suppression

## *Dibenzyl diselenide*

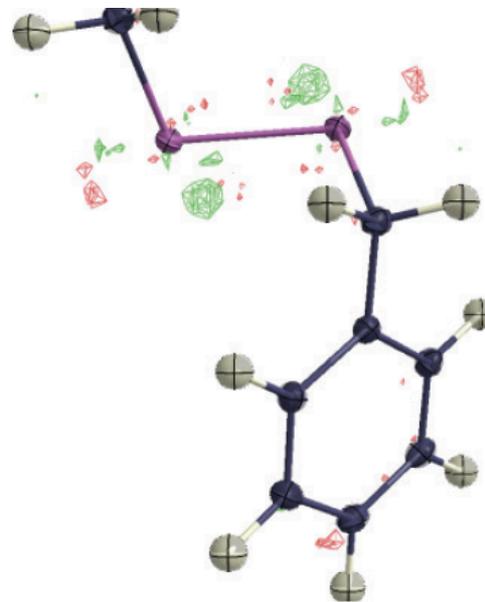
- Ag source
- Se fluorescence between 11.2 and 12.5 keV
- Fluorescence background suppression with 13.5 keV threshold energy



# Dibenzyl di-Se: Residual density



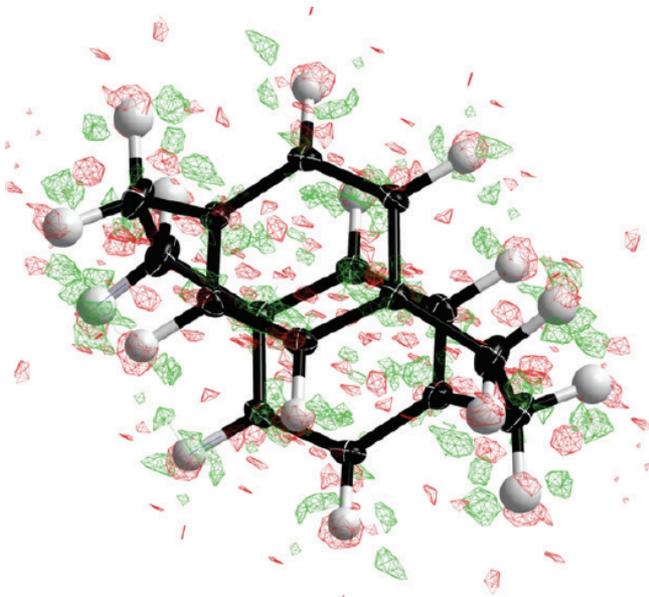
CCD



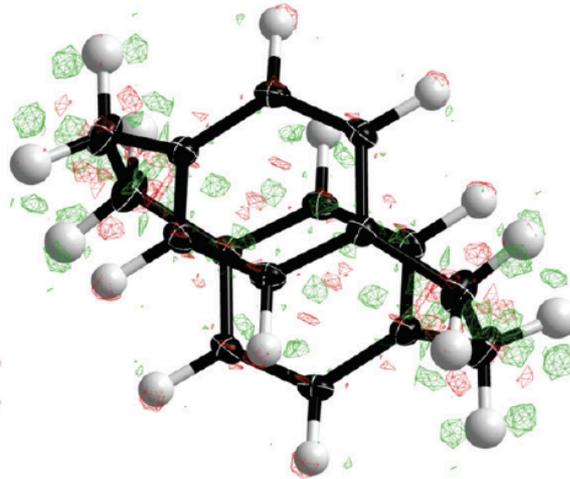
PILATUS3 R CdTe

- *isolevels at*  
 $\pm 0.11 e \text{ \AA}^{-3}$

# Paracyclophane: Residual density



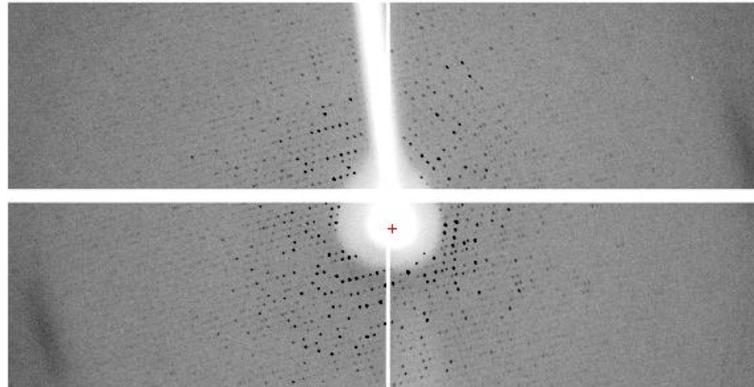
CCD



PILATUS3 R CdTe

- isolevels at  
 $\pm 0.047 \text{ e \AA}^{-3}$

# *Structure of 70S ribosome*



*Thanks to Jean Cavarelli and Alastair McEwen  
(IGBMC Illkirch)*

# EIGER detectors

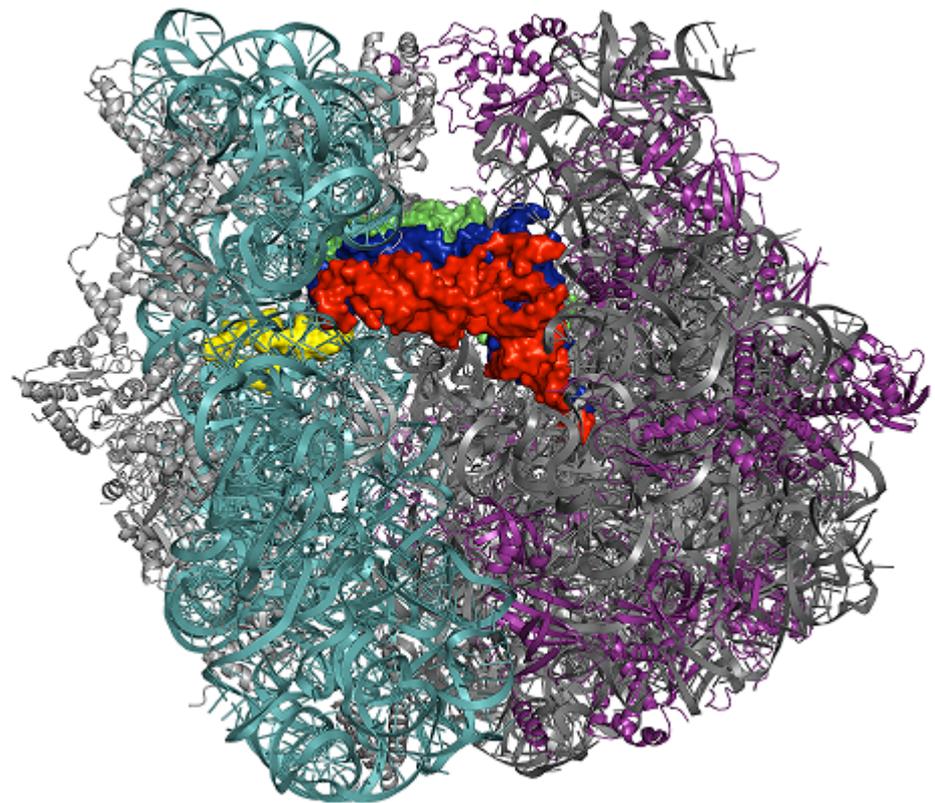
*EIGER R  
for laboratories*



*EIGER X for  
synchrotrons*

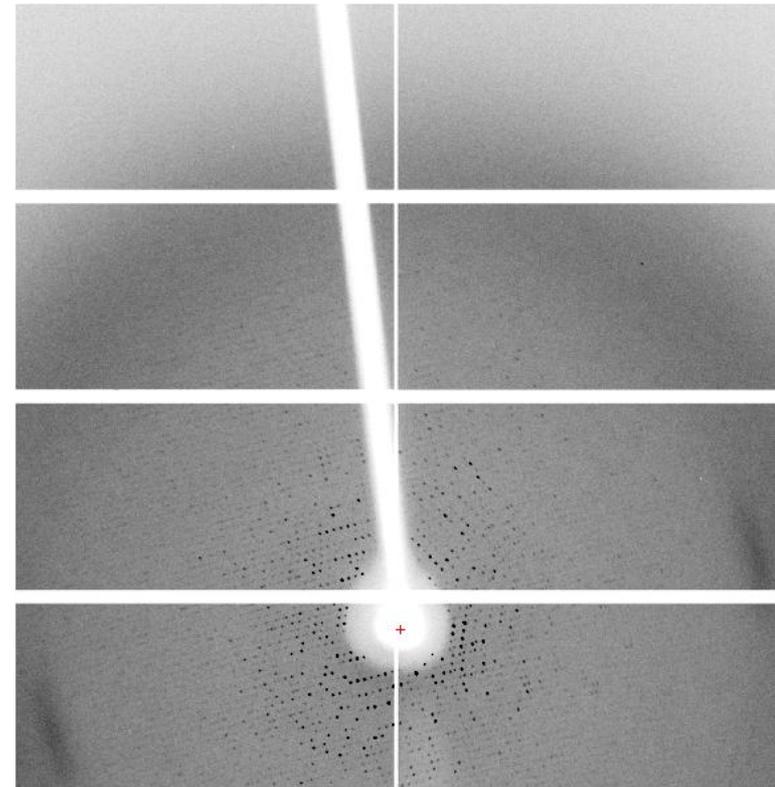
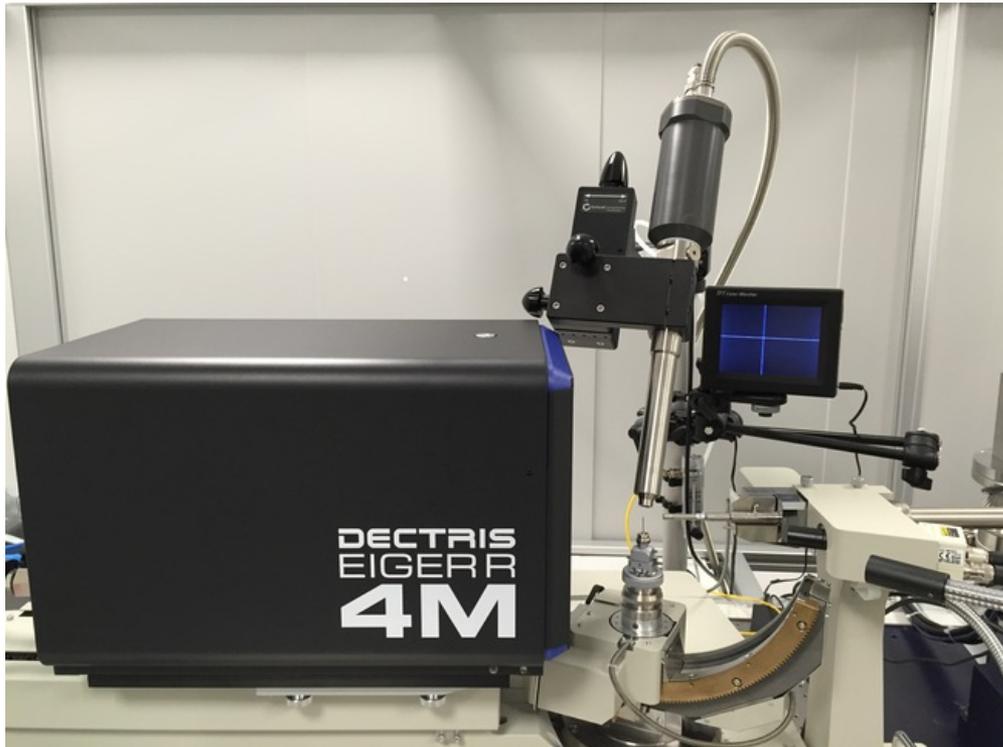
# *Thermus thermophilus* 70S ribosome

- 70S ribosome
- 30 nt mRNA-containing Shine-Delgarno sequence
- $tRNA^{fmet}$
- Paromomycin
- $P 2_12_12_1$
- 209 Å; 449 Å; 620 Å
- Two copies in AU (4.6 MD)
- Diffracts to  $\sim 3$  Å @ SLS/SOLEIL



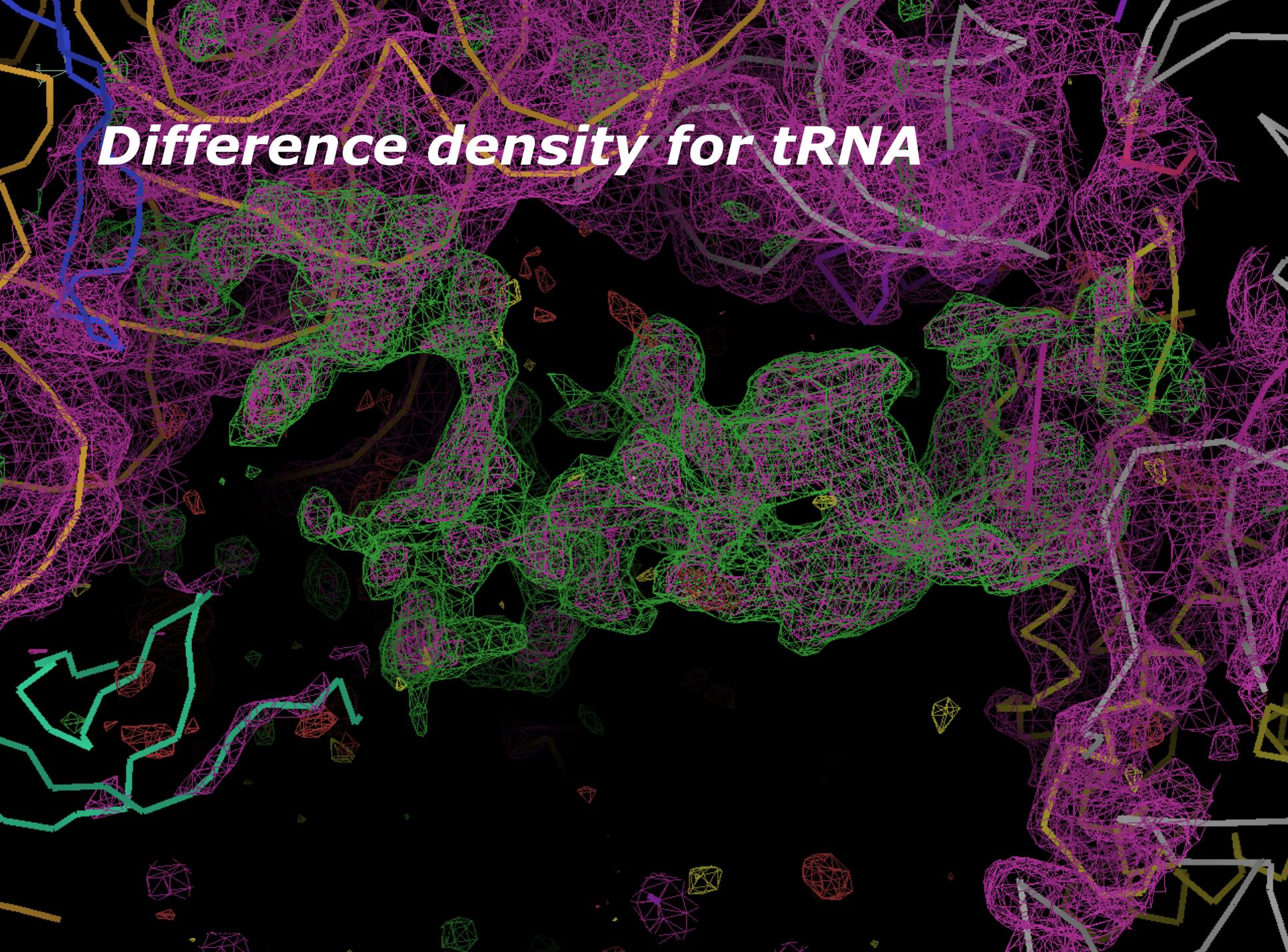
Jenner et al. (2010)

# *Excellent data, clear spot separation*

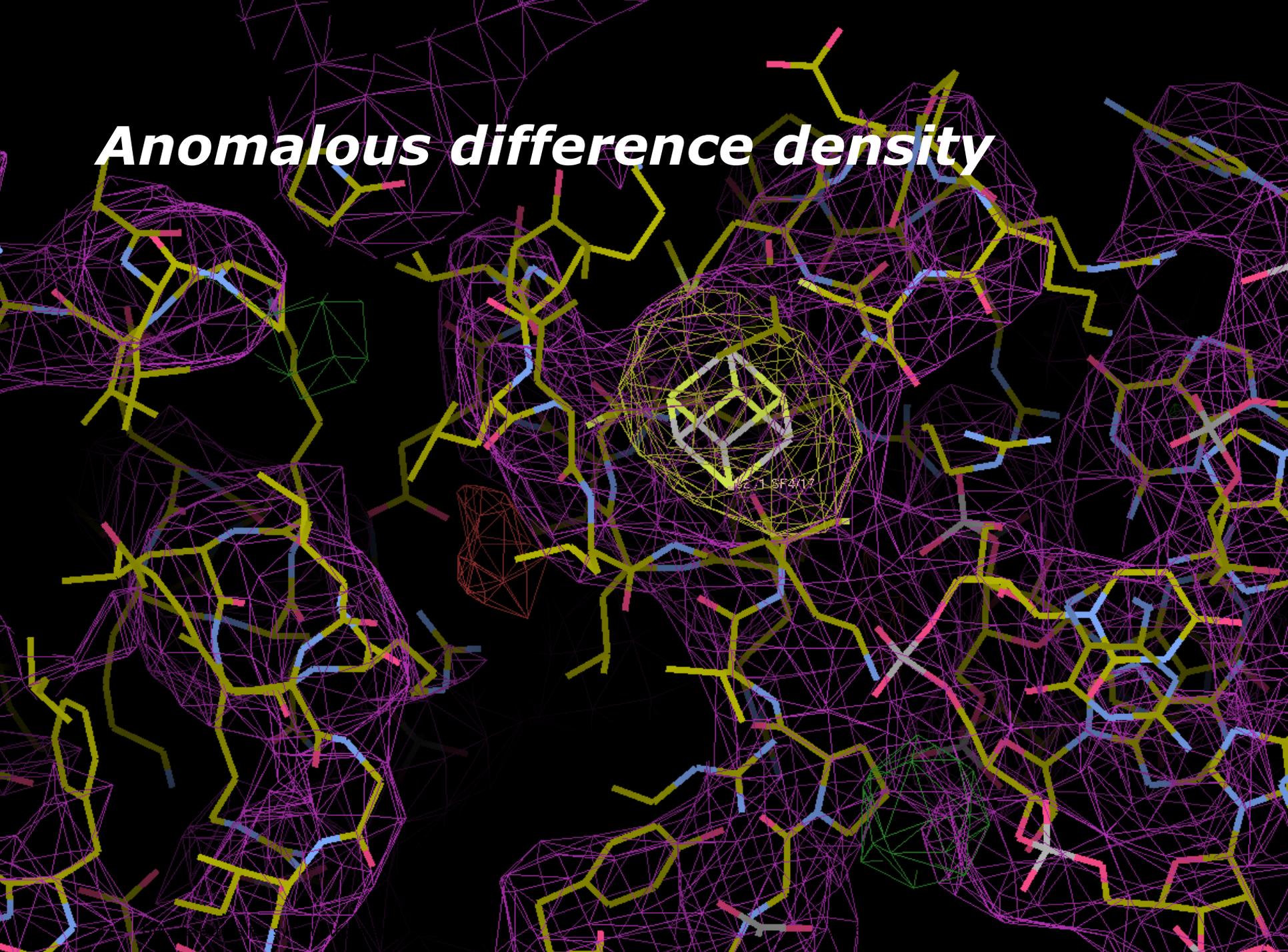


*20% beam (1.8 mrad)*

# ***Difference density for tRNA***



# *Anomalous difference density*



# ***Conclusions***

***Direct detection for high spatial resolution***

***Photon counting for speed***

***Adjustable threshold to cut fluorescence***

***CdTe sensors for high X-ray energies***

**DECTRIS<sup>®</sup>**

*detecting the future*

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***Thank you for  
your attention!***

***[www.dectris.com](http://www.dectris.com)***

***[andreas.foerster@dectris.com](mailto:andreas.foerster@dectris.com)***

**DECTRIS Ltd.**  
5405 Baden-Dättwil  
Switzerland  
**[www.dectris.com](http://www.dectris.com)**