DECTRIS®

detecting the future

Large-area CdTe pixel detectors for high-energy X-ray applications

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PPXRD-14, Fort Myers, Florida, 6.06.-9.06.2016

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High-energy X-rays?

1. (free) Definition

- Laboratory: > 8 keV (Cu-source)
- Synchrotron: > 60 keV
- Detector: > 40 keV

2. Properties

- Higher penetration depth

3. Use (in material analysis)

- Imaging techniques
- Scattering (and diffraction)

4. Technical aspects

- Laboratory tube: energy; flux
- Detector

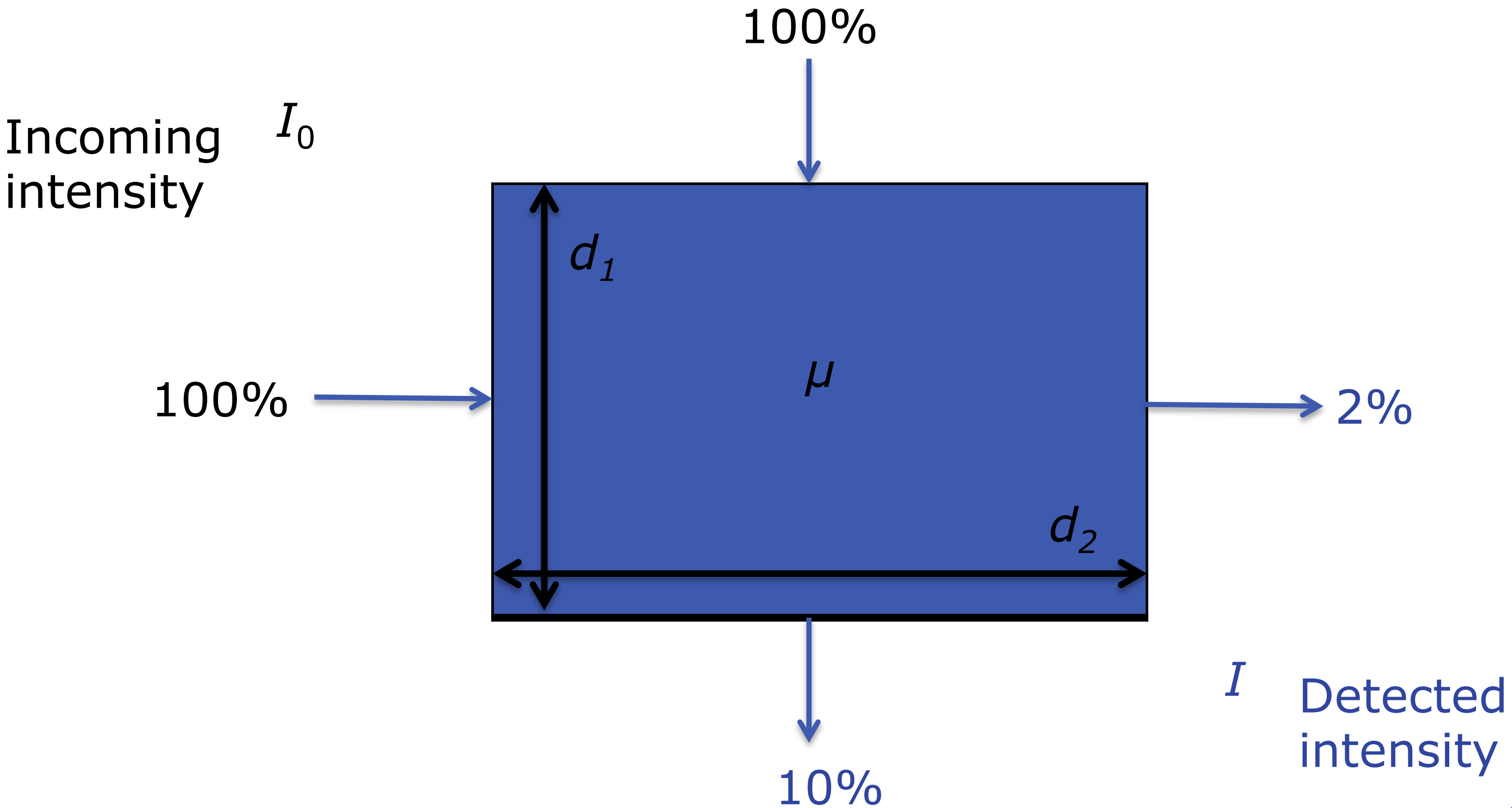
High-energy X-rays in pharmaceutical science

1. Computed Tomography (CT) techniques
2. Real-time/in situ X-Ray Powder Diffraction (XRPD)
3. Pair Distribution Function (PDF) technique

High-energy X-rays in pharmaceutical science

1. Computed Tomography (CT) techniques
2. Real-time/in situ X-Ray Powder Diffraction (XRPD)
3. Pair Distribution Function (PDF) technique
4. XRPD-CT
   - Spatially resolved chemical insight (crystalline materials)
5. PDF-CT
   - Spatially resolved chemical insight (nanocrystaline and amorphous materials)

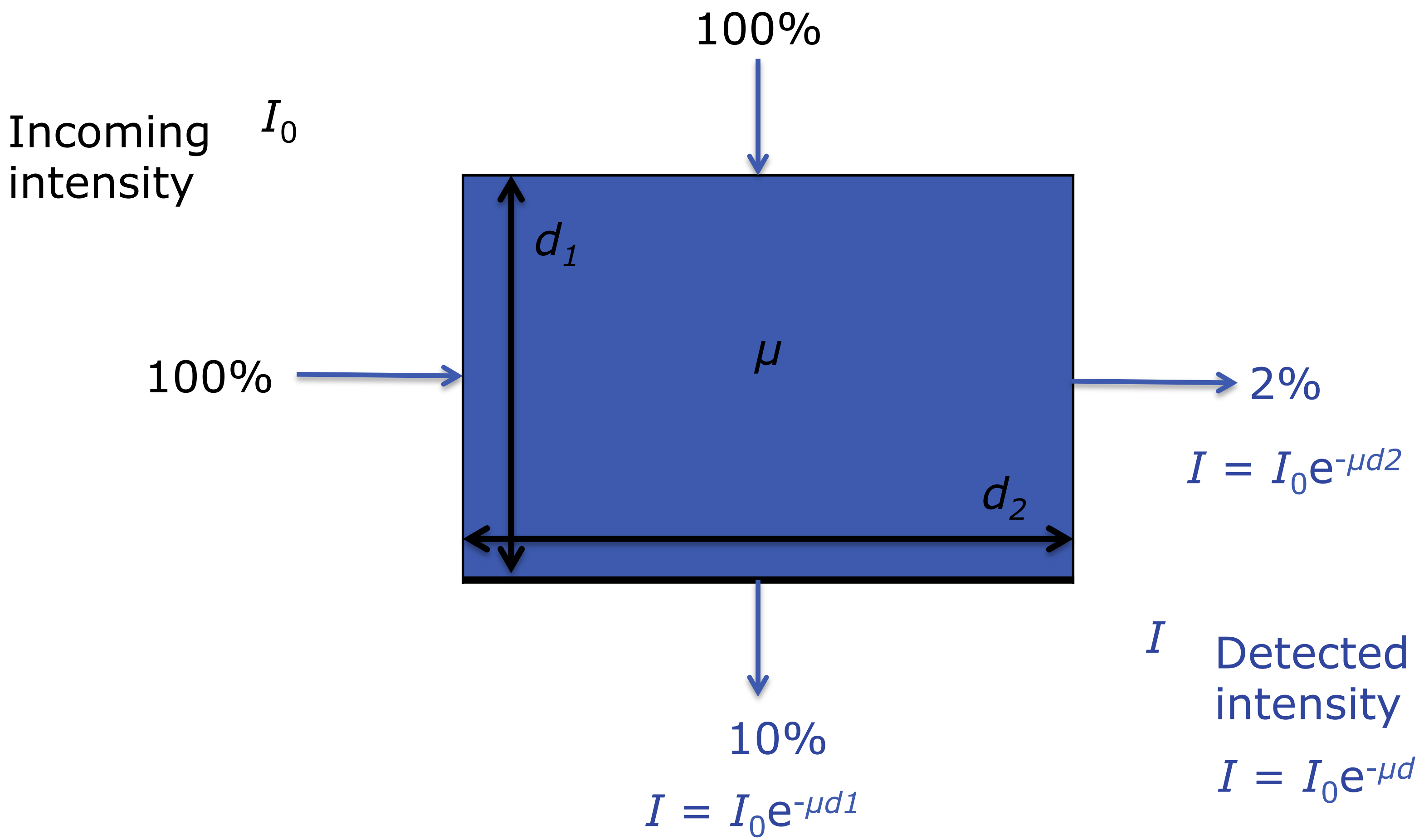
CT principle



$I = I_0 e^{-\mu d}$

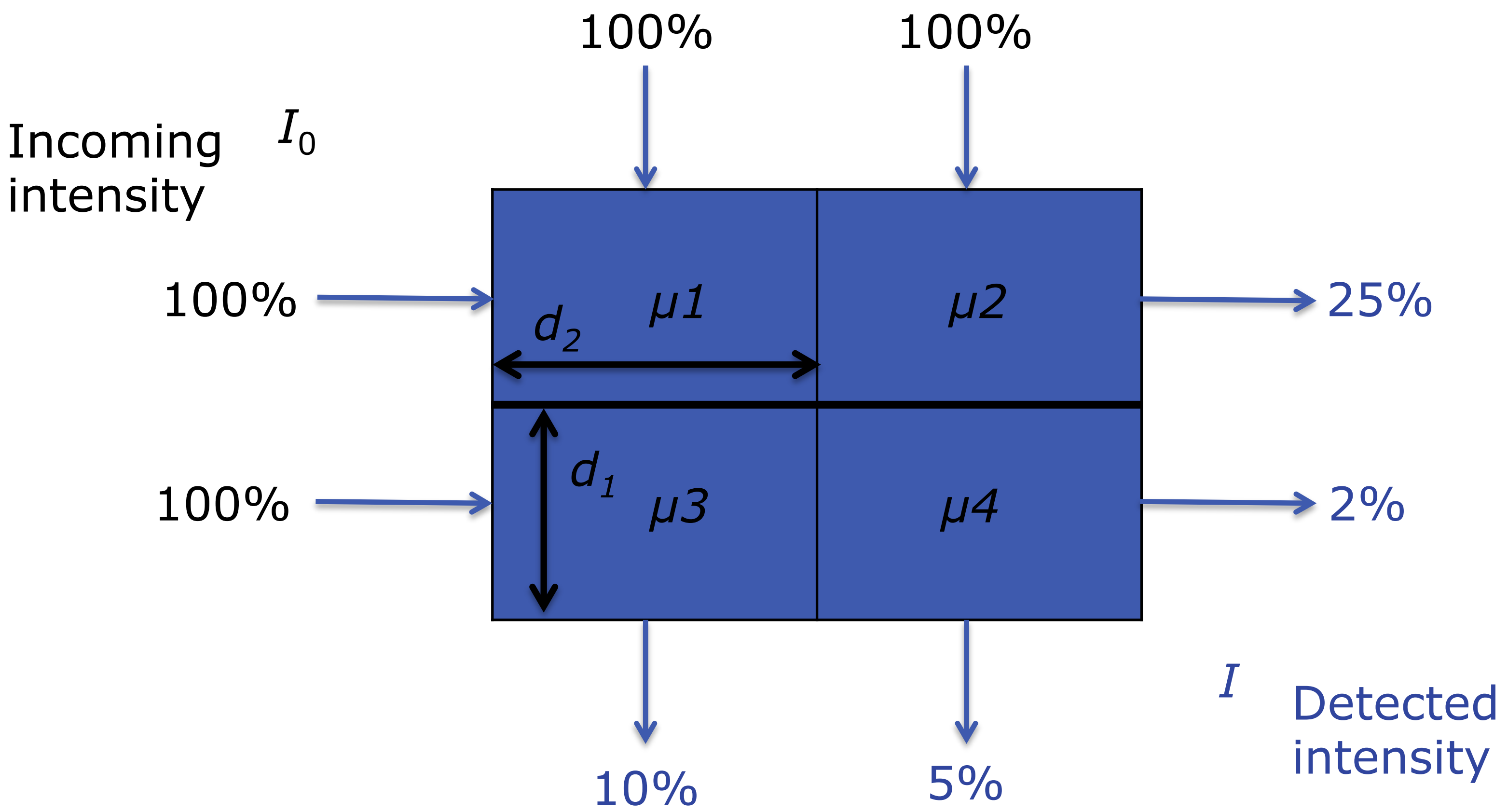
Simplified visualization of Lambeert-Beer law on attenuation of light caused by the properties of a material which the light is travelling through.

CT principle



Simplified visualization of Lambeert-Beer law on attenuation of light caused by the properties of a material which the light is travelling through.

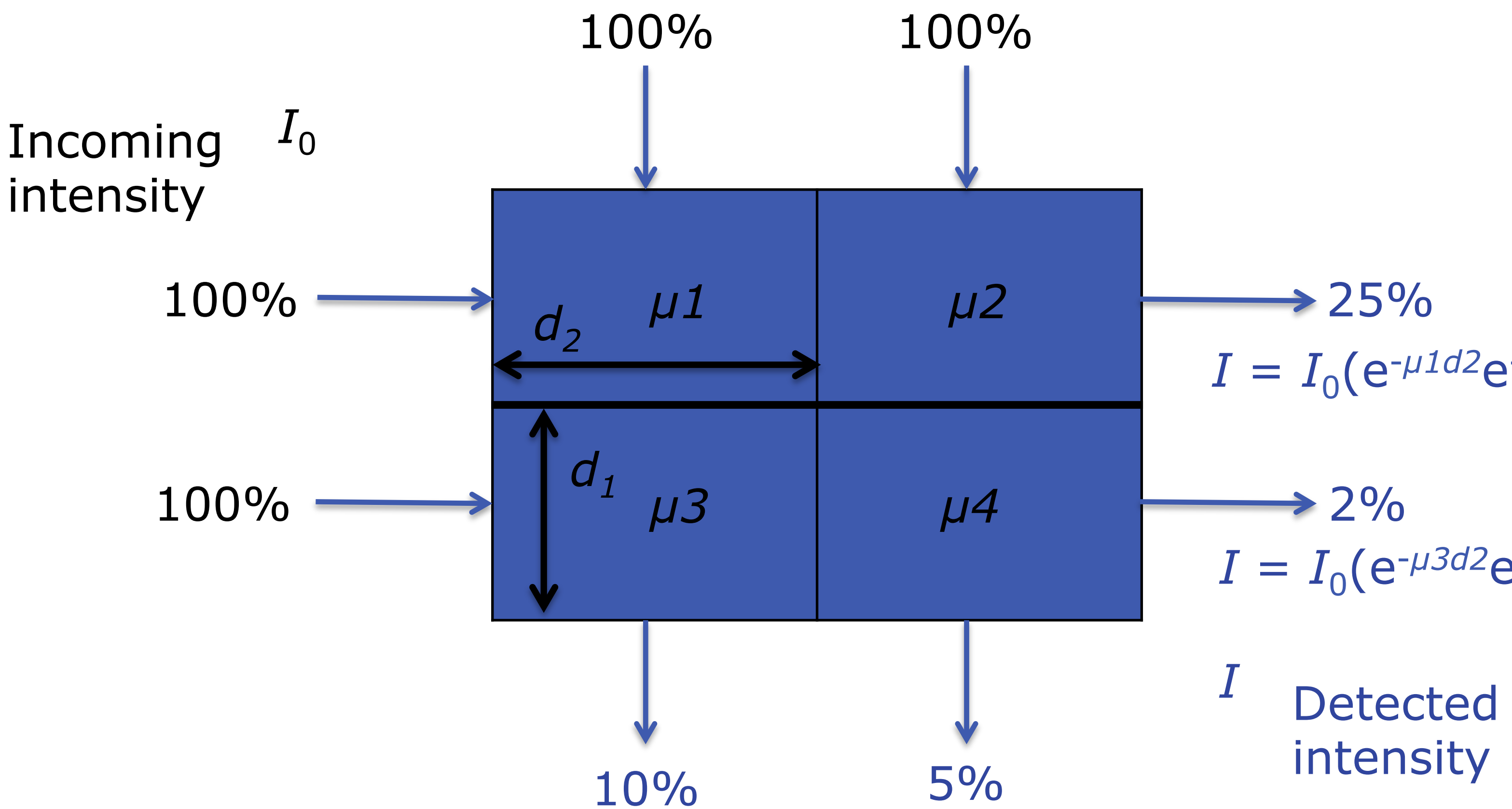
CT principle



$I = I_0(e^{-\mu 1 d1} e^{-\mu 2 d2} \ldots)$

Expansion of Lambeert-Beer law on to objects that comprise four different Materials with different absorption coefficients.

CT principle



$$I = I_0(e^{-\mu 1 d2} e^{-\mu 2 d2})$$

$$I = I_0(e^{-\mu 3 d2} e^{-\mu 4 d2})$$

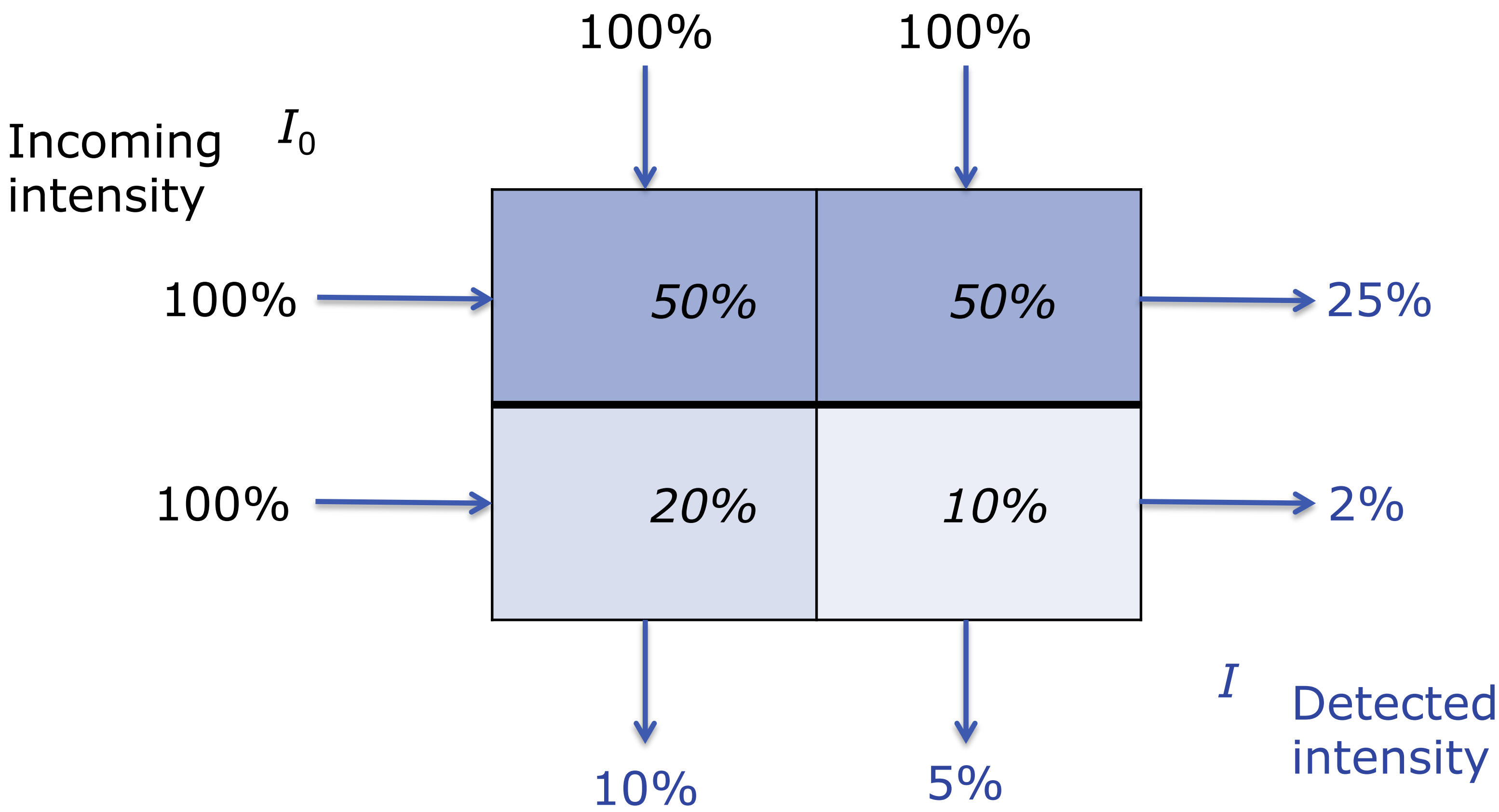
$$I = I_0(e^{-\mu 1 d1} e^{-\mu 3 d1})$$

$$I = I_0(e^{-\mu 2 d1} e^{-\mu 4 d1})$$

$$I = I_0(e^{-\mu 1 d1} e^{-\mu 2 d2} \ldots)$$

Expansion of Lambeert-Beer law on to objects that comprise four different Materials with different absorption coefficients.

CT principle



$I = I_0(e^{-\mu 1 d1} e^{-\mu 2 d2} \ldots)$

Expansion of Lambeert-Beer law on to objects that comprise four different Materials with different absorption coefficients.

CT for investigation of porosity of granules

1. **Objective**
   - *Analyze size, shape and distribution of pores within granules because pores influence dry strength, dissolution and disintegration in liquid medium*
2. **Standard method(s)**
   - *Mercury porosimetry*
   - *Gas adsorption*
3. **New method**
   - *CT*

Farber, L., Tardos, G., Michaels, J.N. (2003) *Powder Technol*. **132**, 57-63.

CT for investigation of porosity of granules

1. **Measurement**
   - *Scanning time 1 hour*
2. **Result**
   - *10% uncertainty due to relatively low signal-to-noise ratio*

Farber, L., Tardos, G., Michaels, J.N. (2003) *Powder Technol*. **132**, 57-63.

CT for investigations of density variations in tablets

1. **Objective**
   - *Analyze density variations in a tablet*
2. **Standard method(s)**
   - *Differential matching*
   - *Hardness tests*
   - *X-ray shadow of lead grids placed in a compact*
3. **New method(s)**
   - *CT*

Farber, L., Tardos, G., Michaels, J.N. (2003) *Powder Technol*. **132**, 57-63.

CT for investigations of density variations in tablets

1. Measurement

- *Multiple steps due to artifacts of a detector*

2. Result

- *Density plot of two tablets with 3D information about the spatial distribution of the components*

Sinka, I.C., Burch, S.F., Tweed, J.H., Cunningham, J.C. (2004) *Int. J. Pharm*. **271(1-2)**, 215-224.

In situ XRPD

1. Aim

- *Investigate processes in situ (in operando): solid-state reaction(s) during milling*
- *X-rays that are able to penetrate the mill*

2. Method

- *High Energy X-Ray Powder Diffraction used on a specifically designed mill*

3. Results

- *Time-resolved structural information of reactants, impurities and (meta)stable product(s)*

Halasz, I. *et al*. (2013) *Nature Protocols* **8(9)**, 1717-1729.

PDF for nanopharmaceuticals

1. Aim

- *PDF for analysis of amorphous and nanocrystalline compounds presents a probability of finding pairs of atoms at a real-space distance in a probed volume.*

2. Method

- *X-ray (total) scattering experiments, performed at energies usually higher than 60 keV*
- *Exposure time 1-10 s using 2D detectors*

3. Results

- *Information about non-crystalline phase(s)*

Billinge, S.J.L. *et al*. (2004) *Chem. Comm*. 749-760
Billinge, S.J.L. (2015) *Nanomedicine* **10**, 2473-2475.

PDF for nanopharmaceuticals

1. Method for

- *API/Excipient/Formulation control and identification*
- *Stability studies*
- *Particle size control*
- *Quantification (residual crystalline sample)*

2. Improvements

- *Synchrotrons*
- *2D detectors*

3. Challenges

- *Collecting wide solid angle fast (detector size)*
- *Collecting PDF data in laboratory (Mo, Ag source)*
- *Radiation damage*

Thakral, S. Et al. (2016) *Adv. Drug Deliv. Rev.* **100**, 183-193.
Billinge, S.J.L. (2015) *Nanomedicine* **10**, 2473-2475.

XRD-CT

1. Aim

- *Spatial identification of crystalline compounds present in a sample*
- *Crystallization and preferred crystal orientation in a stirred reactor (in situ)*

2. Method

- *Synchrotron-based tomographic energy dispersive diffraction imaging (TEDDI)*

3. Limitations

- *Crystallization rates limited by the speed of data acquisition (30 s per point)*

Jacques, S.D.M. (2005) *Cryst. Growth Des.* **5(2)**, 395-397.

PDF-CT

1. Aim

- *Spatial identification of non-crystalline compounds present in a sample*

2. Method

- *X-ray (total) scattering measurements combined with computed tomography*

Simon, D.M. (2013) *Nat. Commun.* **4**, 2536.

High-energy applications: Problems

1. **Tubes**

2. **Algorithms**

3. **Detectors (specifically for in-house measurements)**

   - *Noise*
   - *Speed*
   - *Efficiency*
   - *Size*
   - *Availability*

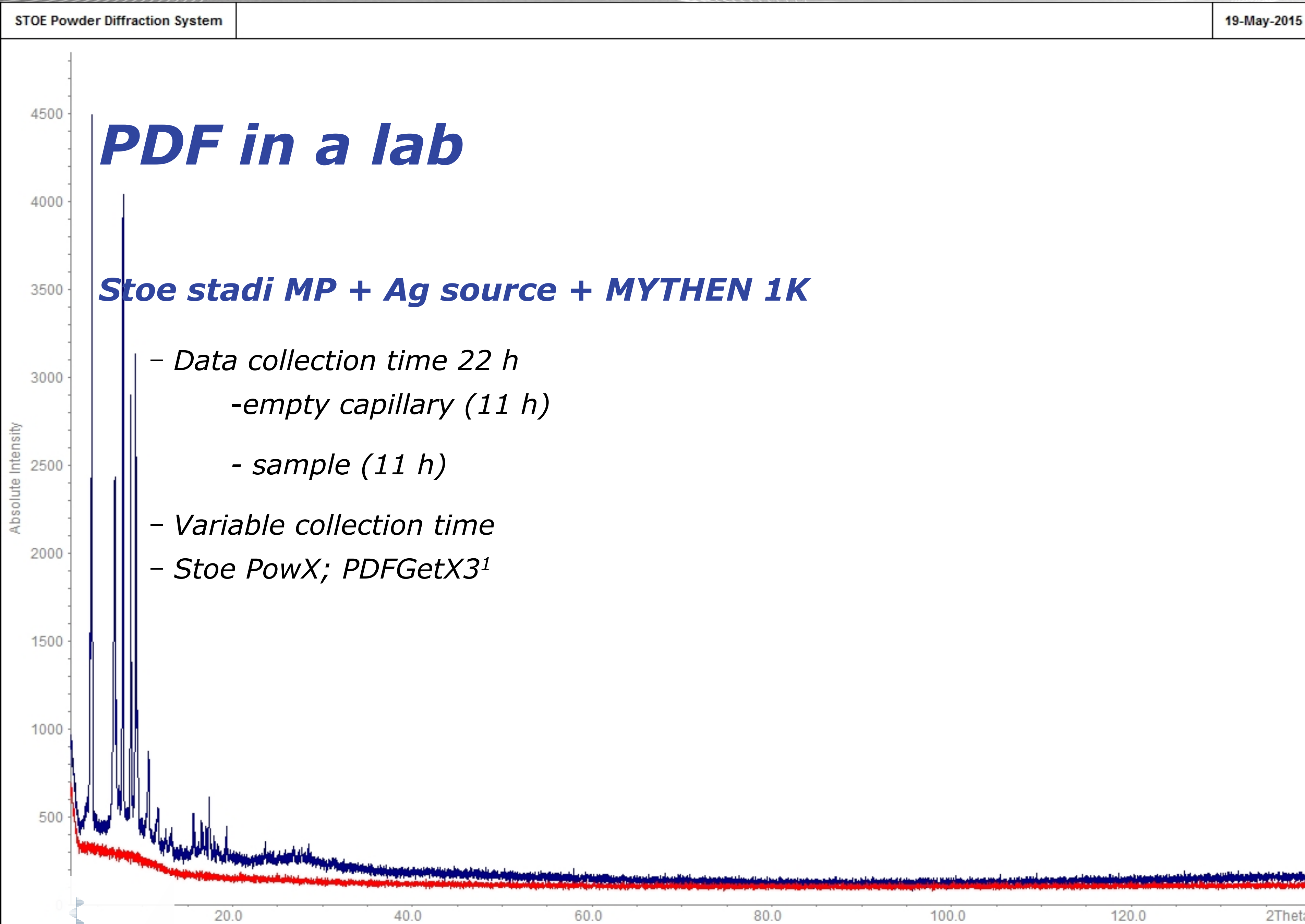
High-energy applications: Problems

1. **Tubes ✔**
2. **Algorithms ✔**
3. **Detectors (specifically for in-house measurements) ✔**
   - *Noise*
   - *Speed*
   - *Efficiency*
   - *Size*
   - *Availability*

PDF in a lab

Stoe stadi MP + Ag source + MYTHEN 1K

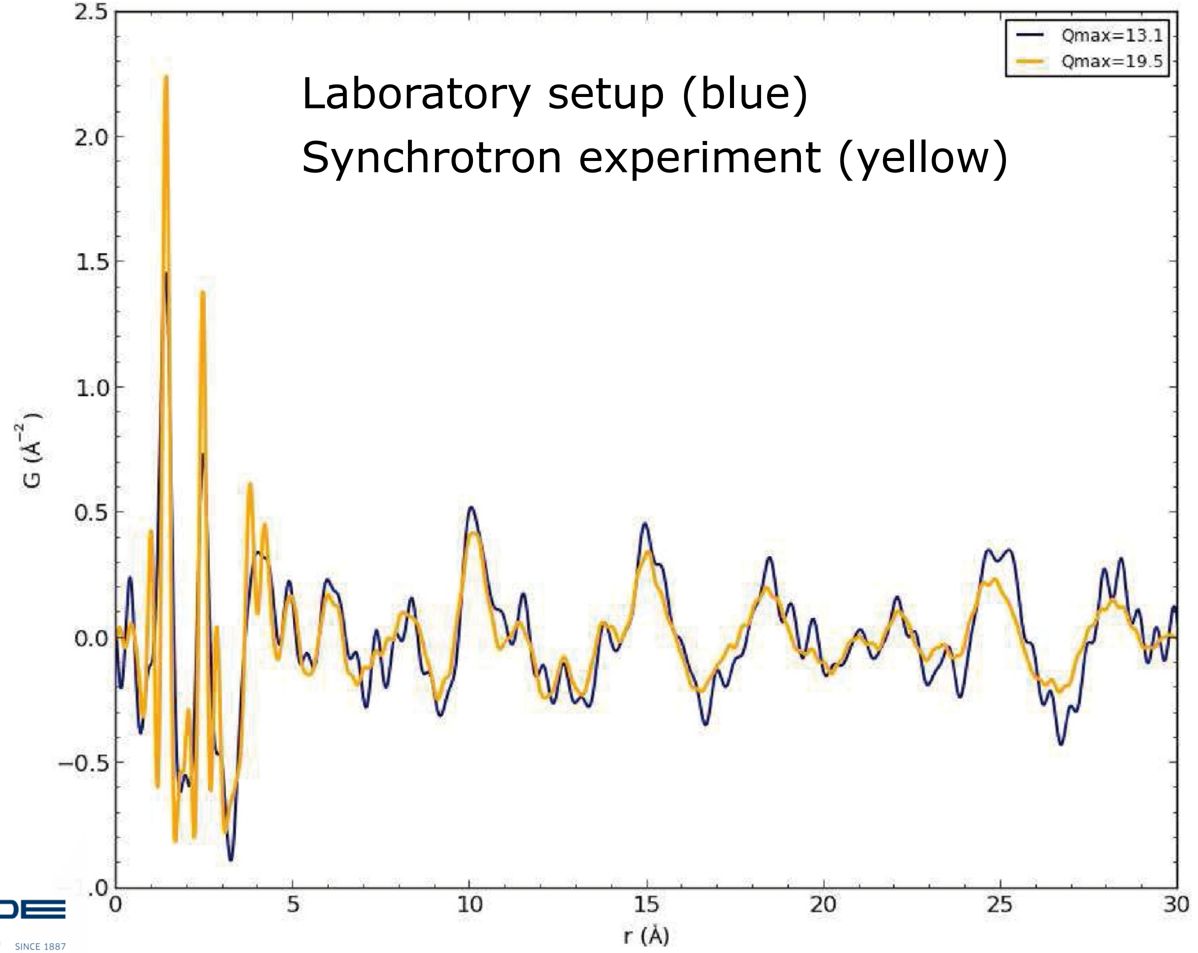
- Data collection time 22 h
  - -empty capillary (11 h)
  - - sample (11 h)
- Variable collection time
- Stoe PowX; PDFGetX3[1]



[1] Juhas, P., Davis, T., Farrow, C.L. and Billinge, S .J. L., *J. Appl. Cryst*. **2013**, *46*, 560-566.

PDF in a lab

Stoe stadi MP + Ag source + MYTHEN 1K



High-energy applications: Problems

1. **Tubes ✔**

2. **Algorithms ✔**

3. **Detectors (specifically for in-house measurements) ✔?**

   - *Noise*
   - *Speed*
   - *Efficiency*
   - *Size*
   - *Availability*

Detectors

1. Noise

- Hybrid Photon Counting (HPC) technology

2. Speed

- Special design of the readout chip
- Firmware

3. Efficiency

- Sensor material responsive to high-energies

4. Size

- Growth/processing of sensor material

5. Availability

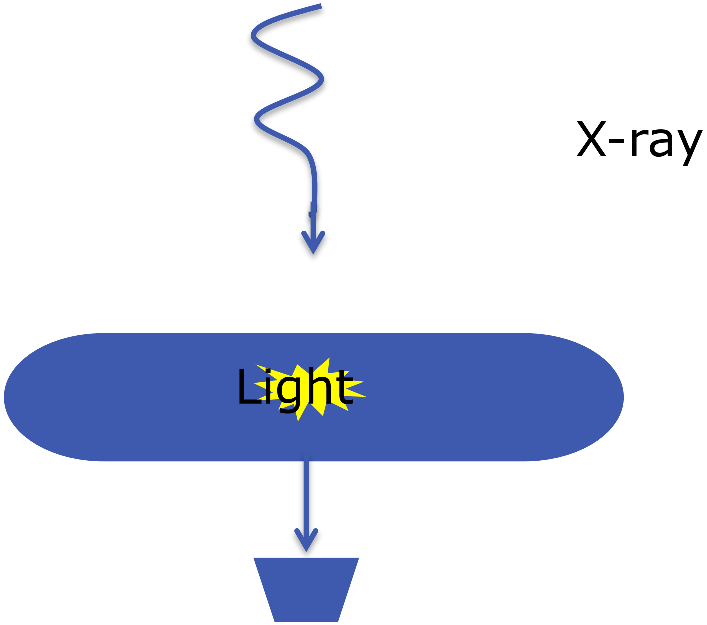
- Price
- Calibration(s)

PILATUS3 CdTe

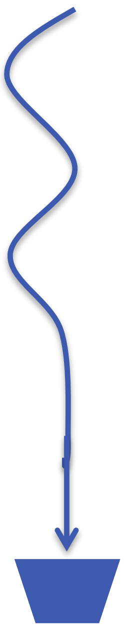
Loeliger, T. *et al*., Proc. IEEE Nuclear Science Symposium and Medical Imaging Conference (NSS/MIC), 2012, 610.

PILATUS3 CdTe

Hybrid Photon Counting (HPC) = Direct detection of X-rays



Indirect detection
Traditional detectors

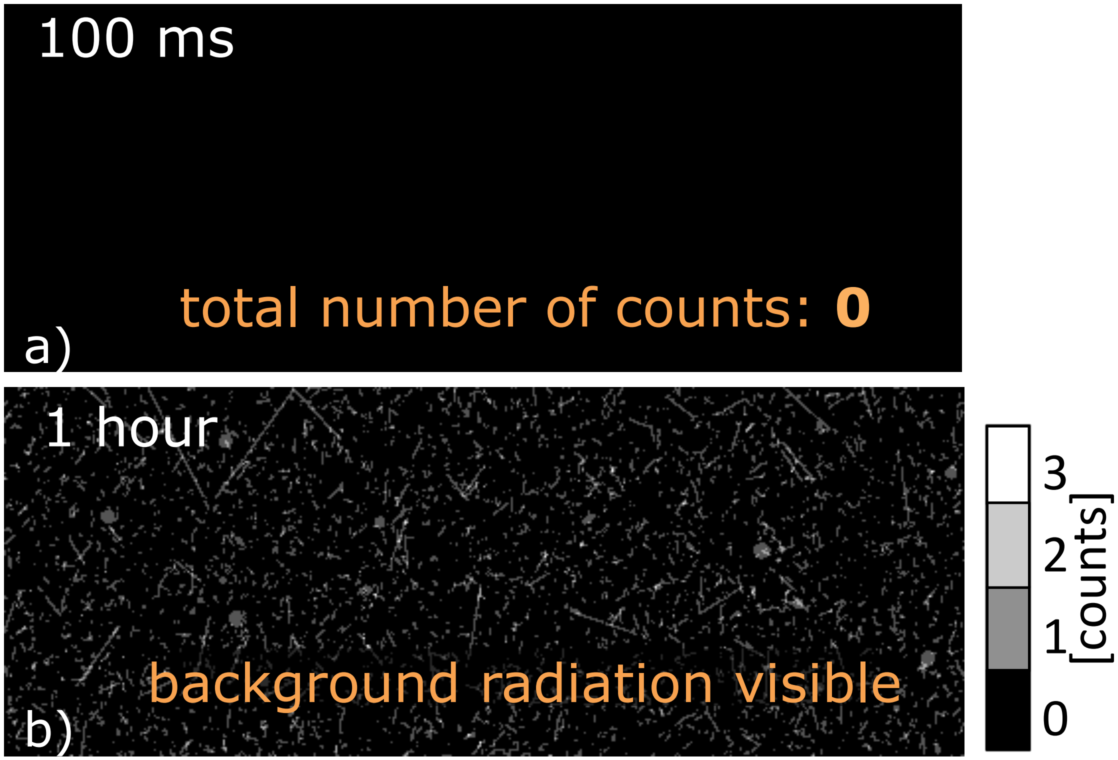


Direct detection
HPC detectors

- noise-free performance

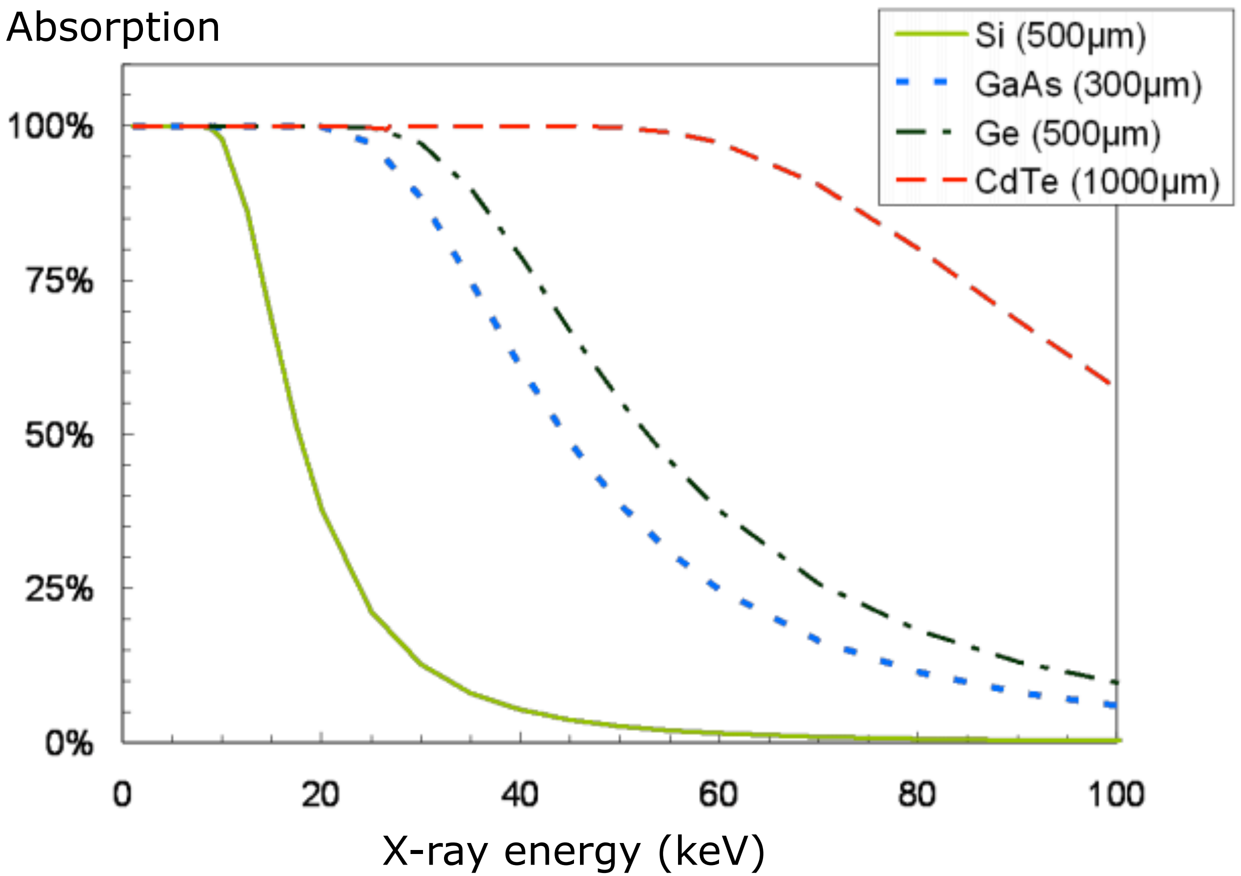
PILATUS3 CdTe

Hybrid Photon Counting (HPC) = no-noise performance



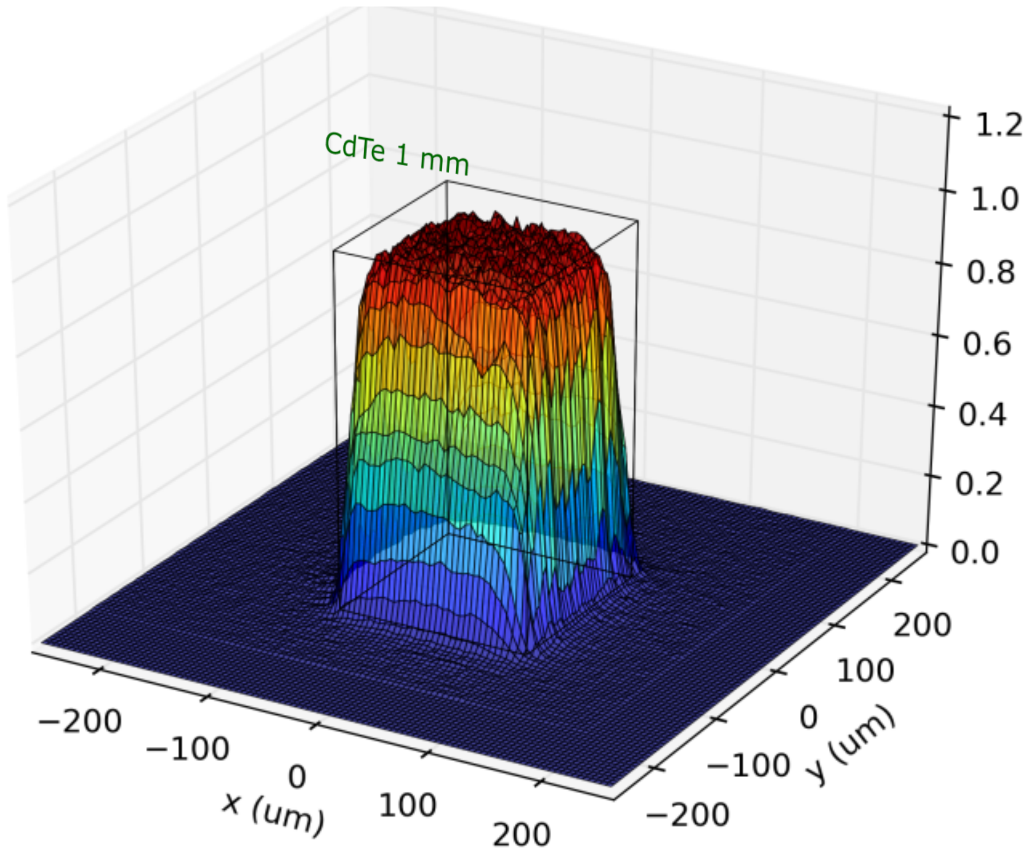
Images collected with PILATUS 100K detector using exposure time 100 ms and b) 1h.
Both images are free of readout noise and dark current.

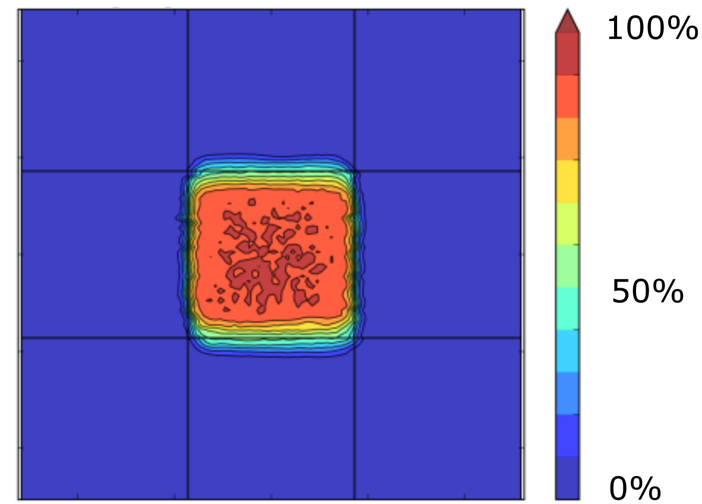
PILATUS3 CdTe



Absorption of different material sensors calculated for energy range 0-100 keV.

PILATUS3 CdTe





Pixel response scanned using a 10 μm diameter pinhole.

PILATUS3 CdTe

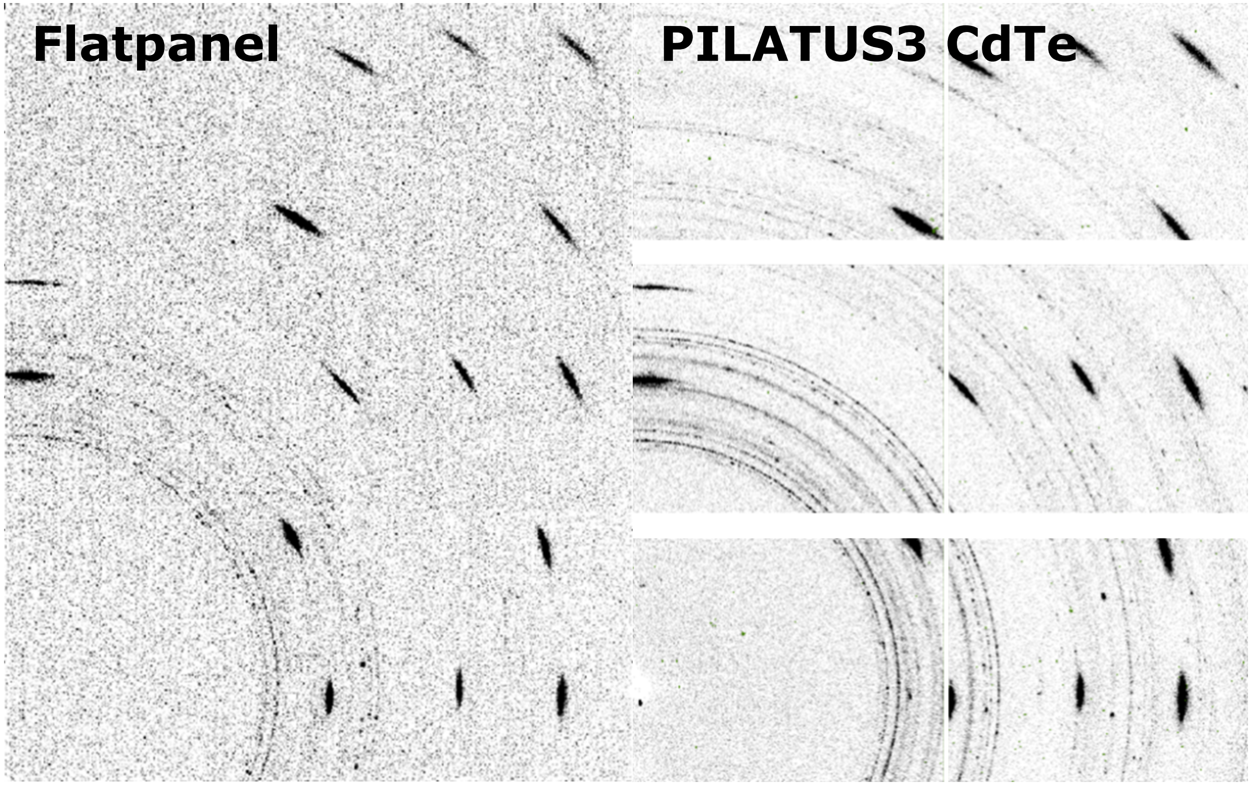
Tests at synchrotron sources:

XRPD

PDF

XRPD-CT

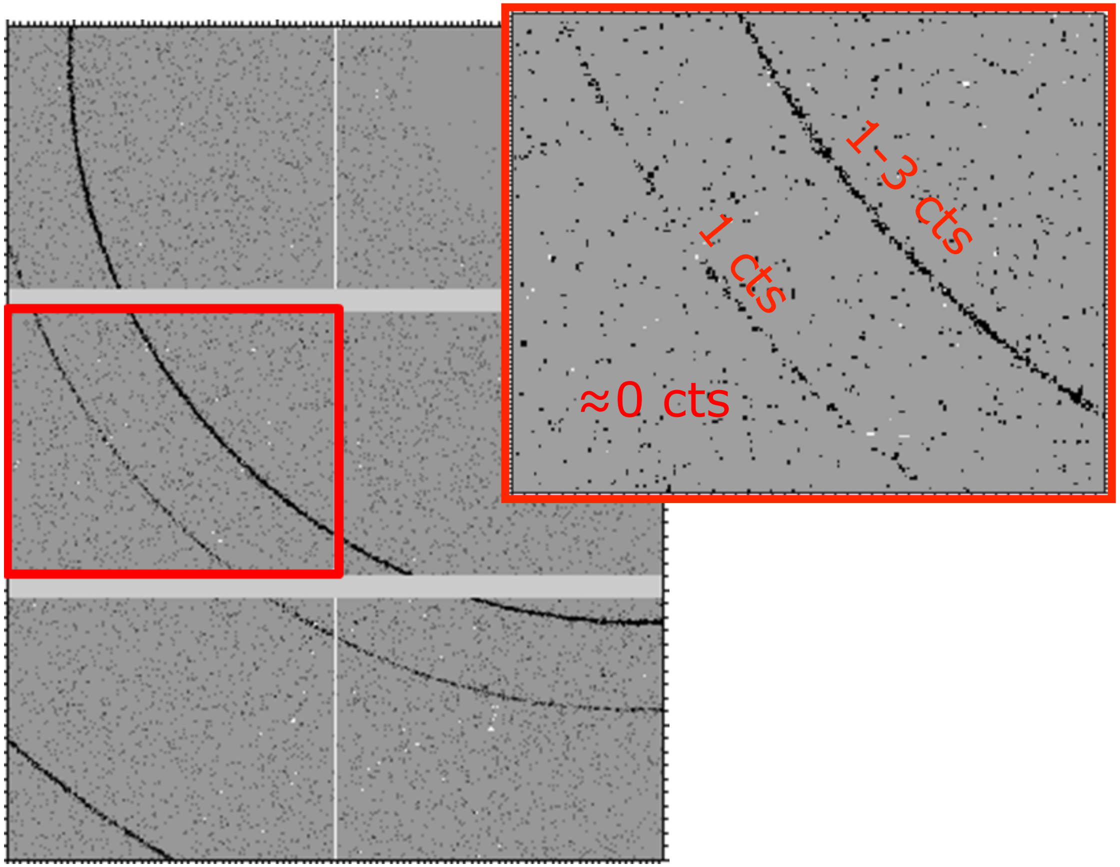
XRPD (ESRF, ID15)



Two XRPD patterns of a superconducting filament containing Nb_3Sn powder in a tungsten tube (Ø50µm). Both patterns are recorded at 46.3 keV, the same solid angle, the same flux per pixel and the same exposure time (100 ms).

ESRF ID15: M. Di Michiel, G. Vaughan, R. Homs, T. Buslaps Univ. Manchester: S. Jacques

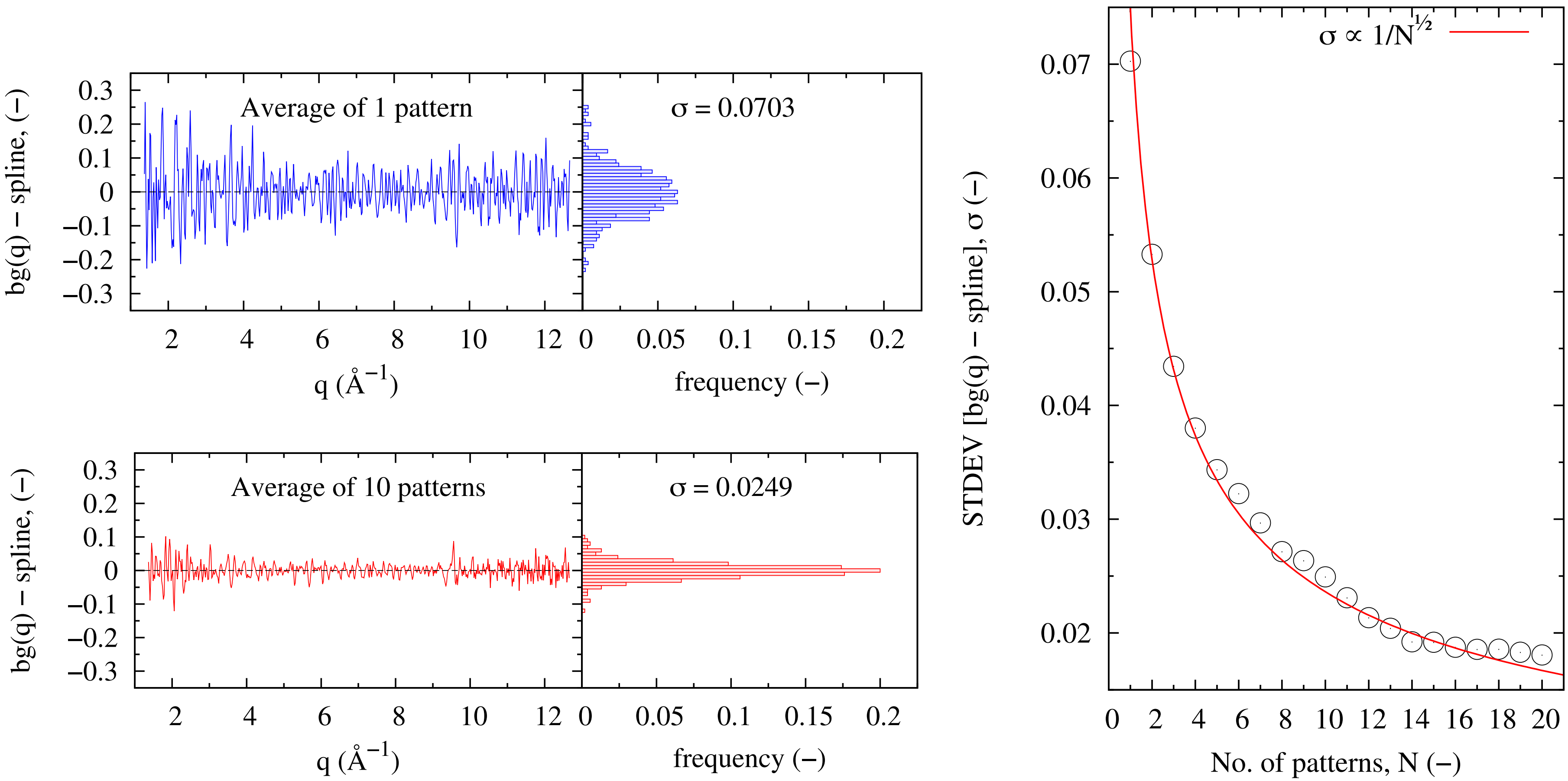
XRPD (DLS, I12)



XRPD pattern of a CeO_2 sample, taken at 55 keV,
Using 0.2 mm slits and exposure time of **0.001** sec.
Background intensity is mainly zero.

Acknowledgment: Michael Drakopoulos, Oxana Magdysyuk (DLS)

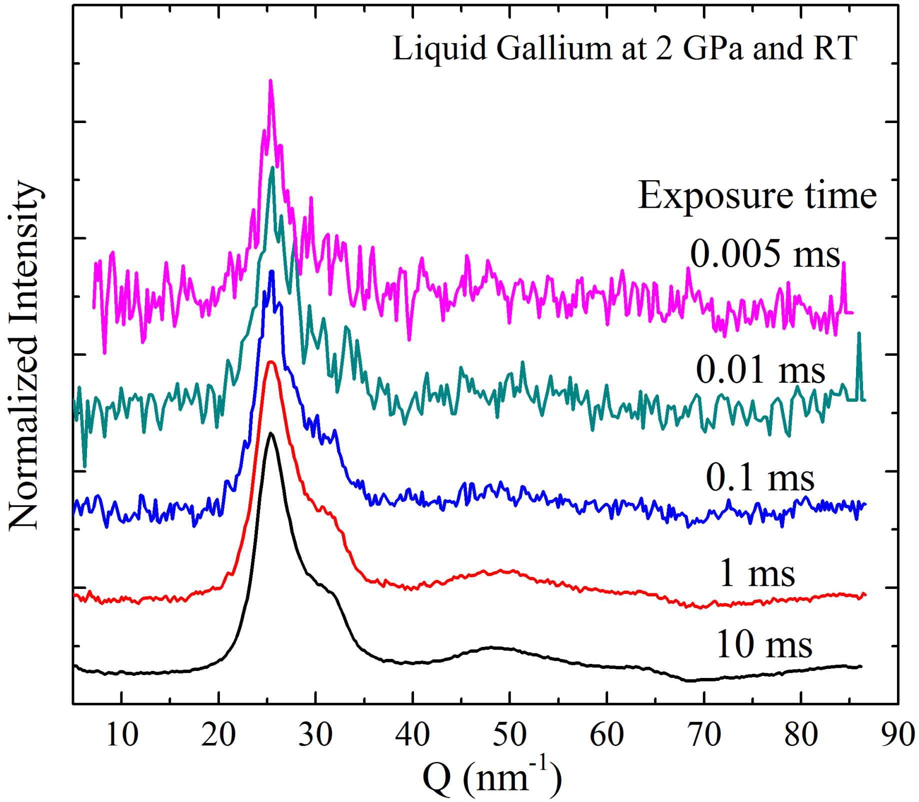
PDF (DESY, P02)



Standard deviation in PDF measurements for data collected at 60 keV. Exposure Time per pattern is 0.1 s.

Bednarcik, J. and Liermann, H.P., poster presentation at DESY Users Meeting, 2016.

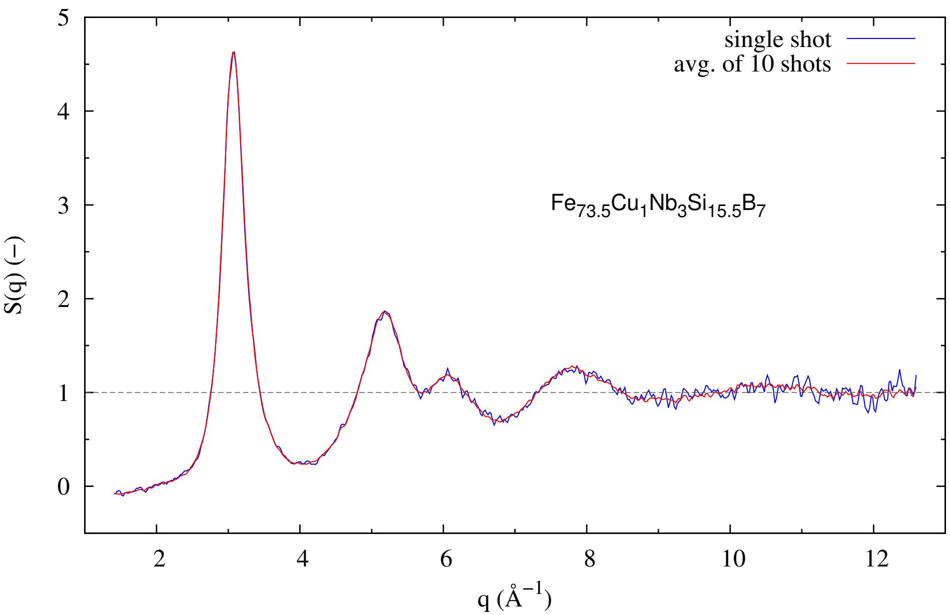
PDF (APS, HPCAT)

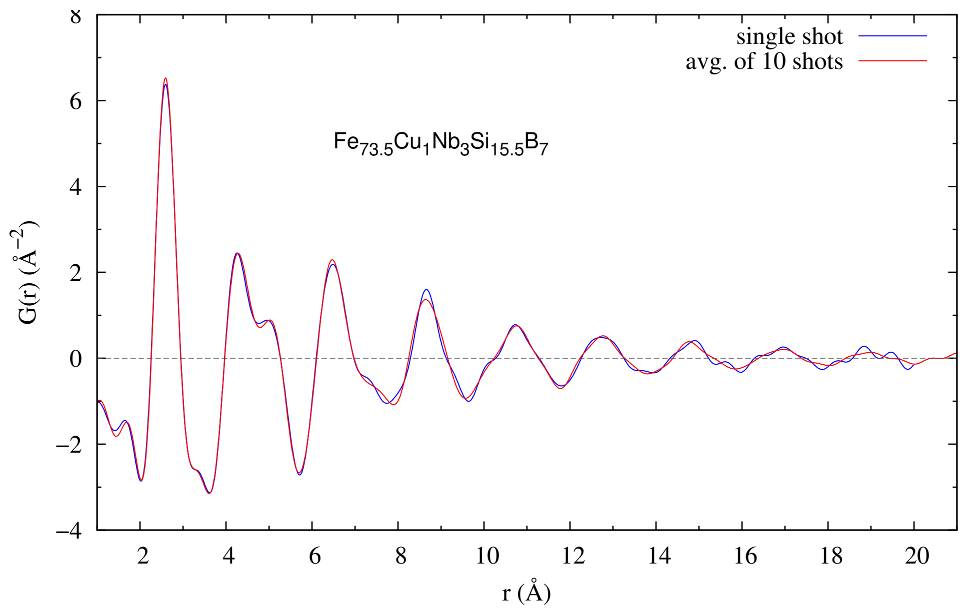


Diffraction patterns of liquid Ga at 2 GPa collected at *E*=30.5 keV.

Acknowledgement: Stanislav Sinogeikin, HPCAT, APS.

PDF (DESY, P02)





To calculate the structure factor the measured intensities I(q) were corrected for only background and Compton scattering.

PDFs for 0.1s and 1s looks very similar. Almost no differences in the nearest neighborhood from 2 Å to 3.8 Å.

Bednarcik, J. and Liermann, H.P., poster presentation at DESY Users Meeting, 2016.

XRPD-CT (ESRF, ID15)

1. Aim

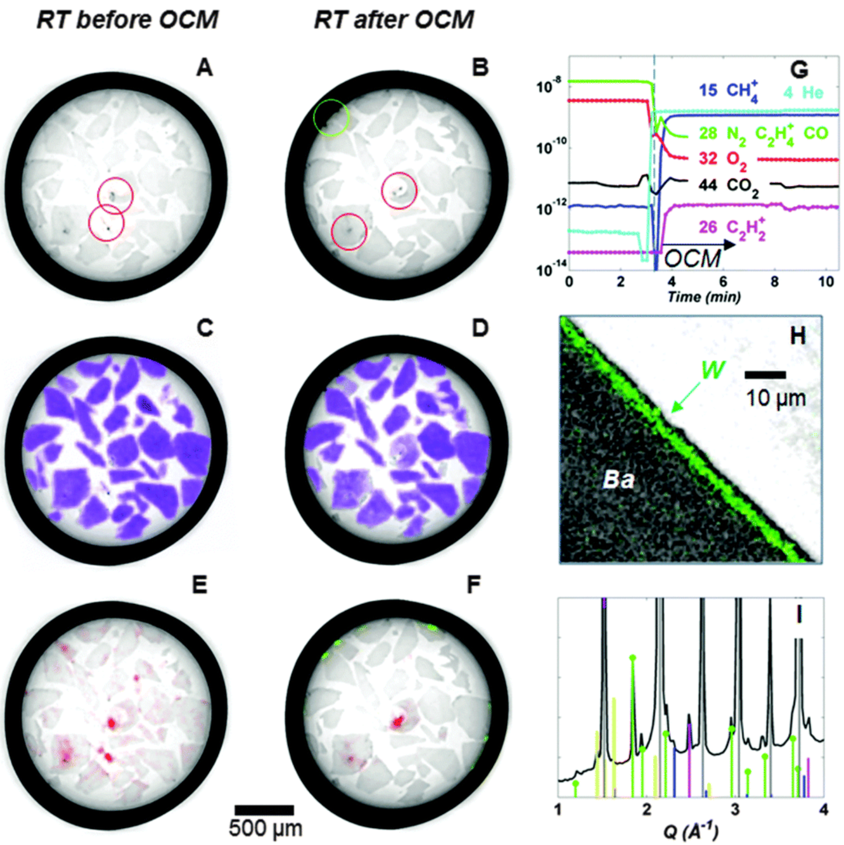
- *Spatially and time-resolved identification of of a catalytic membrane reactor (CMR) during oxidative coupling of methane.*

2. Method

- *XRD-CT*

A. Vamvakeros *et al*., *Chem. Commun*., 2015, 51, 12752

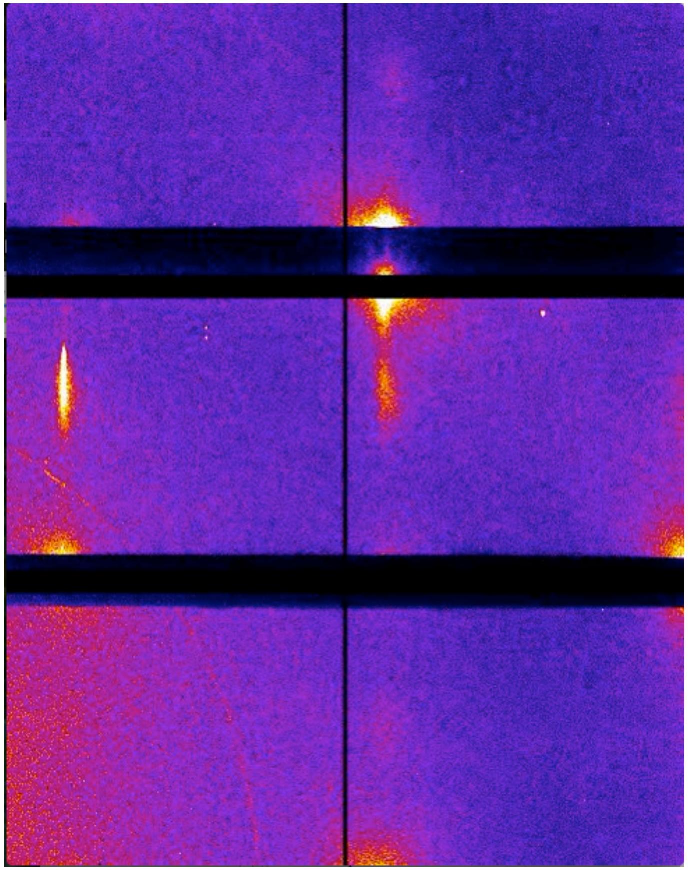
XRPD-CT (ESRF, ID15)



Identification and determination of position of crystobalite (C,D), Na^2WO_4, and Ba_2WO_4 (A, B, E, F) within the catalytic membrane reactor.

A. Vamvakeros *et al*., *Chem. Commun*., 2015, 51, 12752

PILATUS3 CdTe dynamics



Successful tests at several beamlines
And various applications

First PILATUS3 CdTe (2M) installed
At ESRF, ID-15

PILATUS3 CdTe 1M
ordered APS, Sector 13

…

Zhou Hua, Argonne National Laboratory.

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