High-resolution powder diffraction beamline ID22

Andy Fitch
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Fitch *et al.*, *J. Synchrotron Rad.* 30, 1003–1012, (2023)

STREAMLINE

STREAMLINE has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 870313

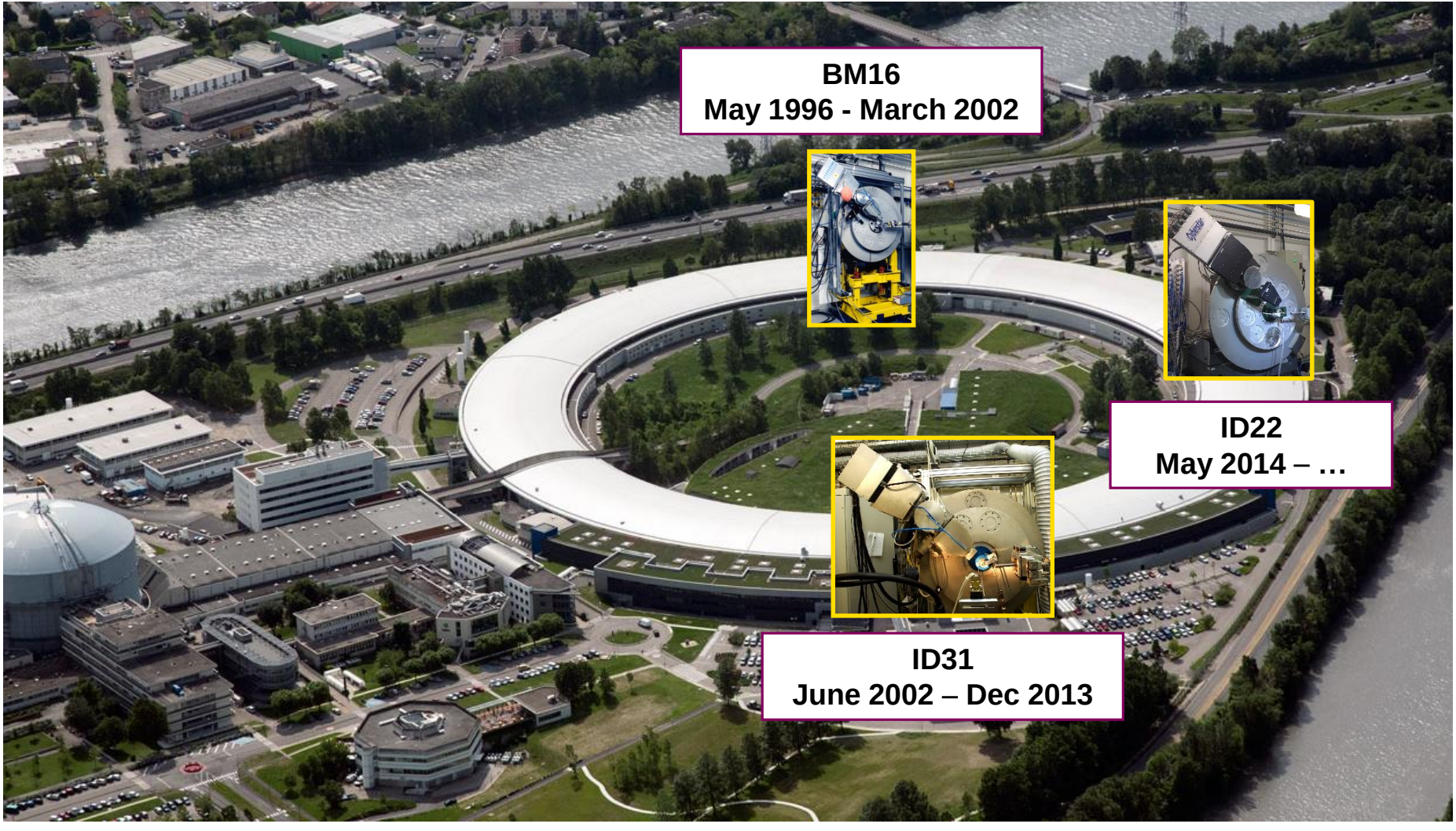
Synchrotron radiation and powder diffraction

High intensity, collimation and λ tunability

$\Downarrow$

- High angular resolution, i.e. narrow peak widths
- Rapid data collection / good statistics
- Highly monochromatic X-rays so well-defined instrumental peak shape (no $\alpha_1\alpha_2$ doublets, etc)
- λ tunable: measure at absorption edges, or well away; optimise for the experiment
- High energies for increased Q range, PDF, or penetrate through absorbing samples or sample environments

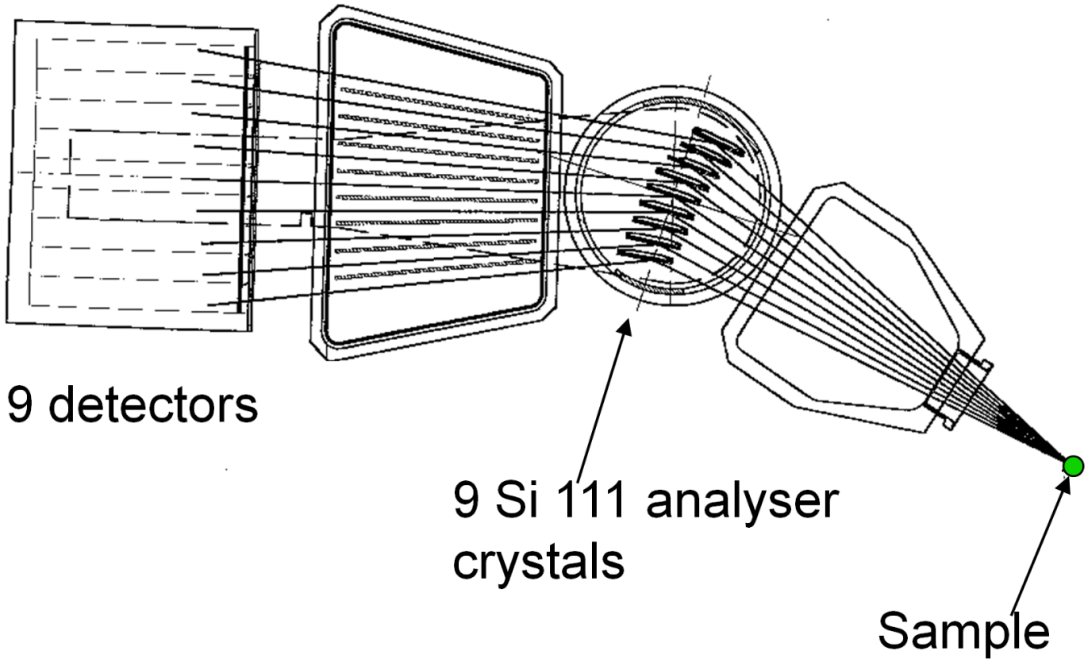
ESRF's high resolution powder diffraction beamline



Improvements to ID22 during EBS upgrade

- EBS storage ring
  Brighter source ⇒ two-fold increase in flux
- Change of undulators (⇒ 2.5 m long in-vacuum u26)
  ⇒ two-fold increase in flux above 60 keV
- New multi-analyser stage (9 ⇒ 13 Si 111 crystals)
- Installation of Eiger pixel detector behind multi-analyser stage
- Automatic correction for axial divergence
  ⇒ Narrower and more-symmetric peaks
  ⇒ Improved statistical quality at high angles
- BLISS has replaced SPEC for beamline control.

Nine-channel multi-analyser stage



J.-L. Hodeau, M. Anne, P. Bordet, A. Prat, Institut Néel, Grenoble.
Hodeau *et al*. *Proceedings SPIE*, **3448,** 353-361, (1998)

Multi-analyser upgrade

Jan 2021 July 2021

⇒

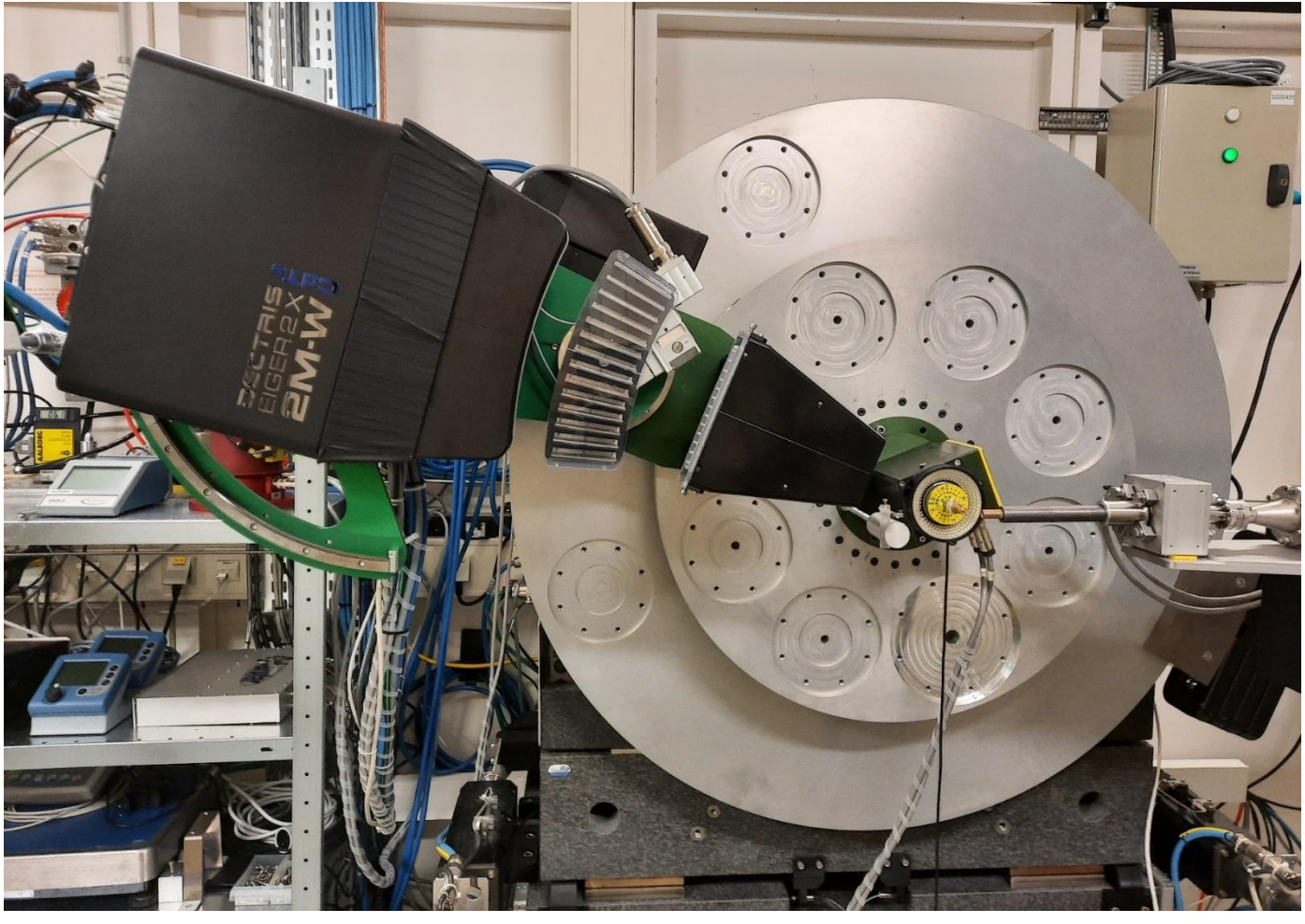
⇒

9 analyser + 9 scintillation crystals counters

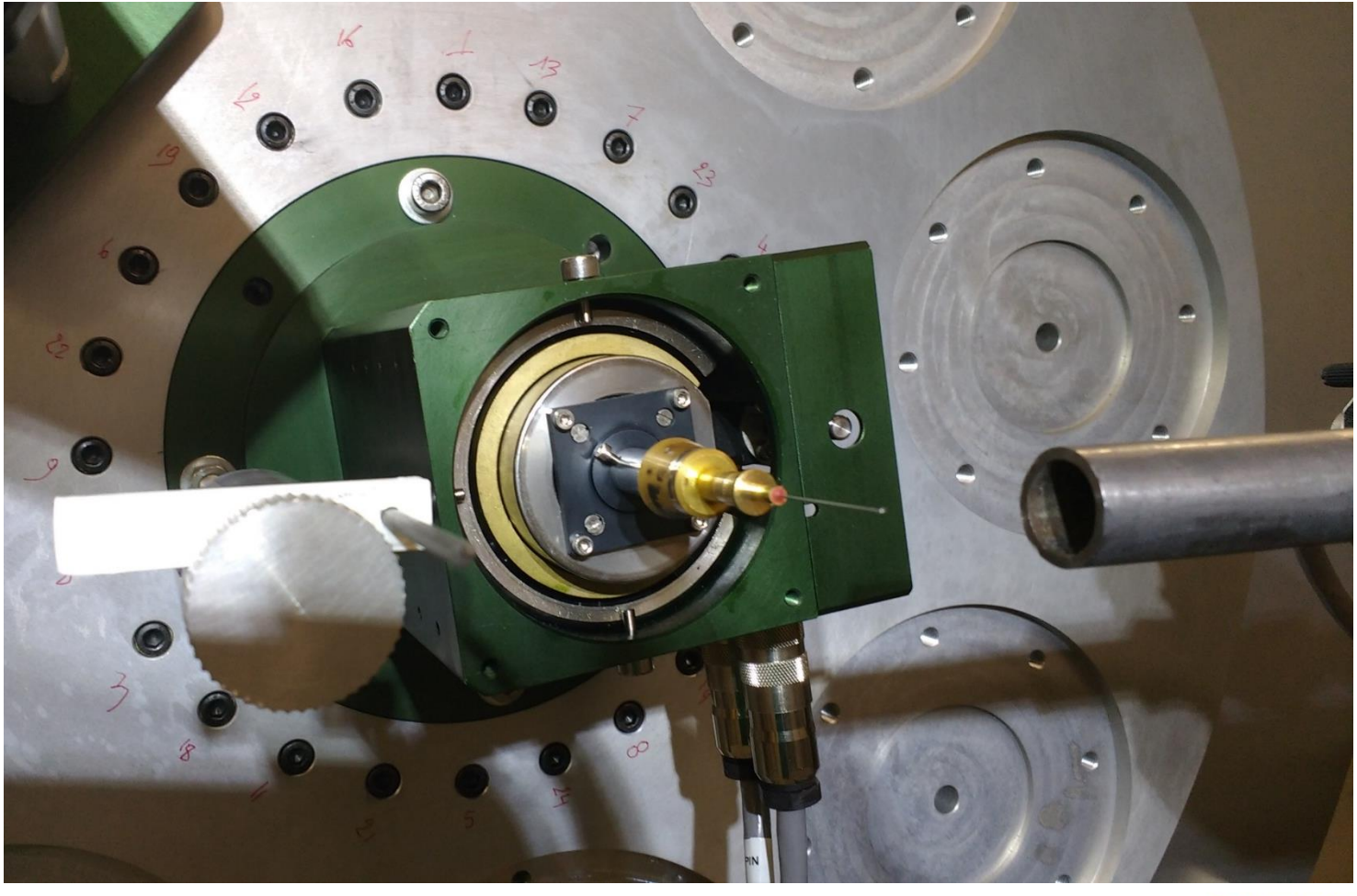
9 analyser + Eiger 2M crystals

13 analyser + Eiger 2M crystals

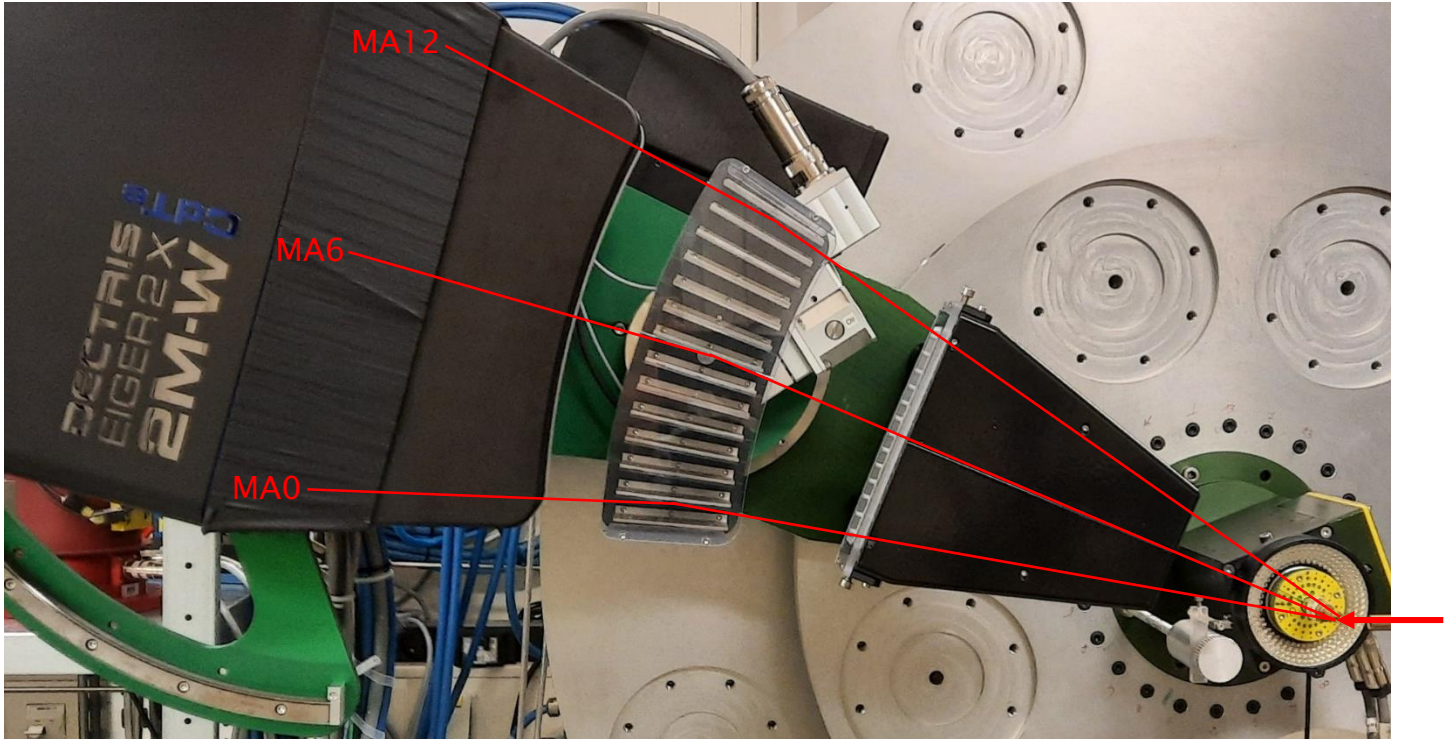
ID22 Powder Diffractometer



Capillary sample spinner



13-channel Si 111 multi-analyser stage + Eiger2 X 2M-W CdTe

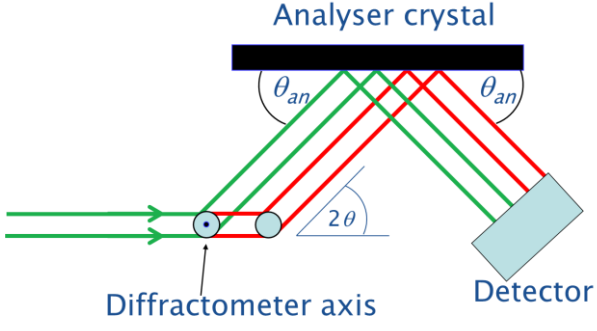


Original 9-channel version conceived by J.-L. Hodeau, M. Anne, P. Bordet, A. Prat, Institut Néel, Grenoble. Hodeau *et al*. *Proceedings SPIE*, **3448,** 353-361, (1998)

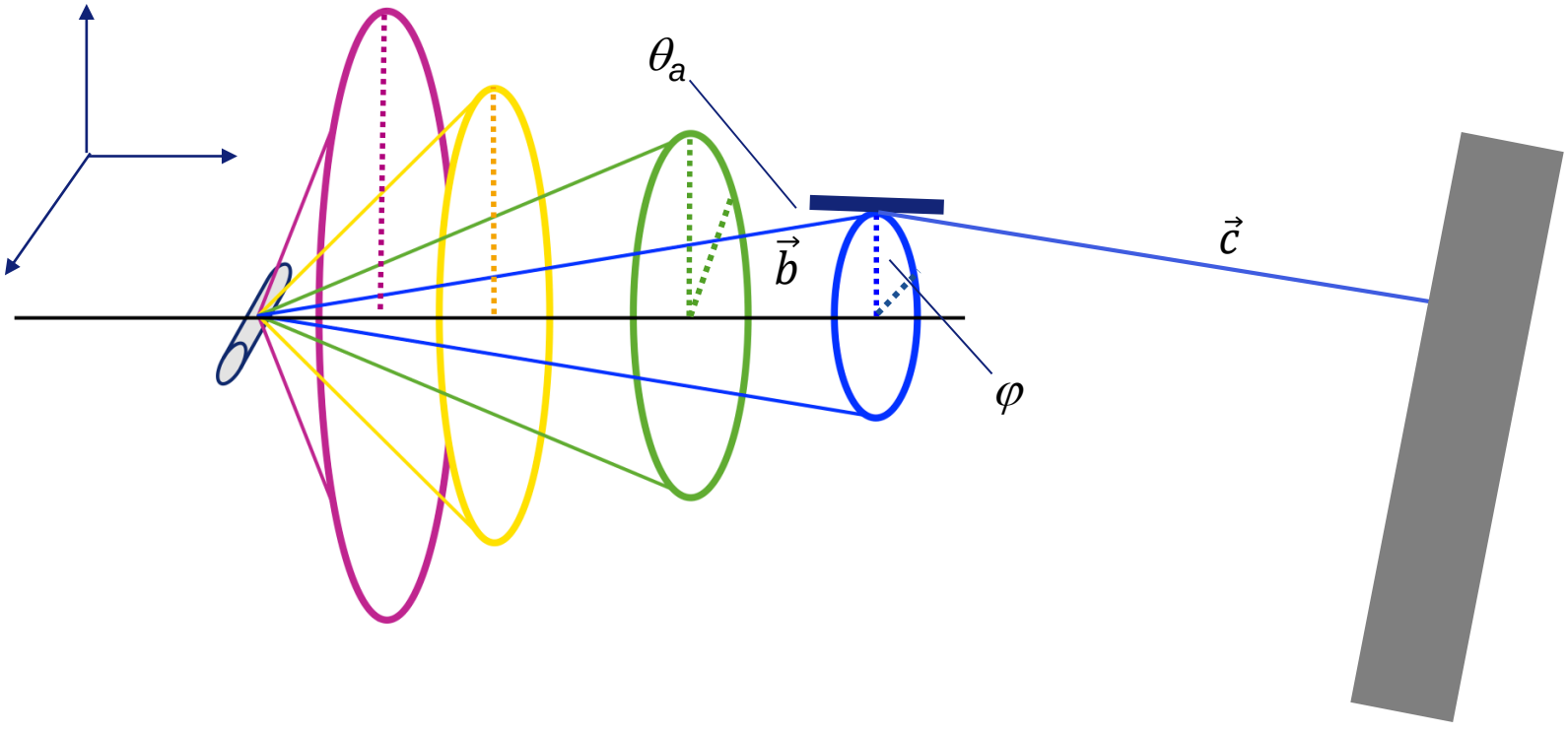
Analyser crystal

Stringently defines a true 2θ *angle* rather than infers 2θ from the *position* of a slit or pixel of a PSD.

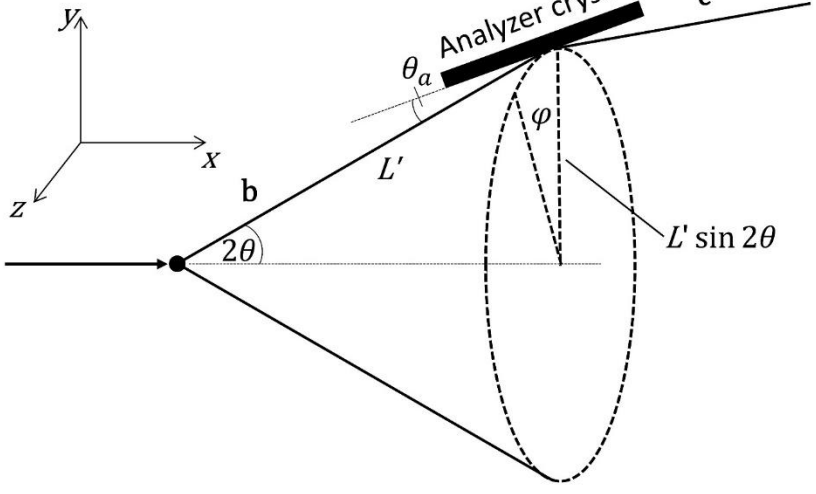
- Narrow (sample-limited) peaks with accurate positions
- Peak positions insensitive to displacement-type aberrations, sample misalignment, specimen transparency, size / shape / surface effects, etc.
- Peak widths independent of any $\theta/2\theta$ parafocusing condition
- Supresses fluorescence, Compton, parasitic scatter.
- But it needs to be scanned, so is not as fast as a PSD.

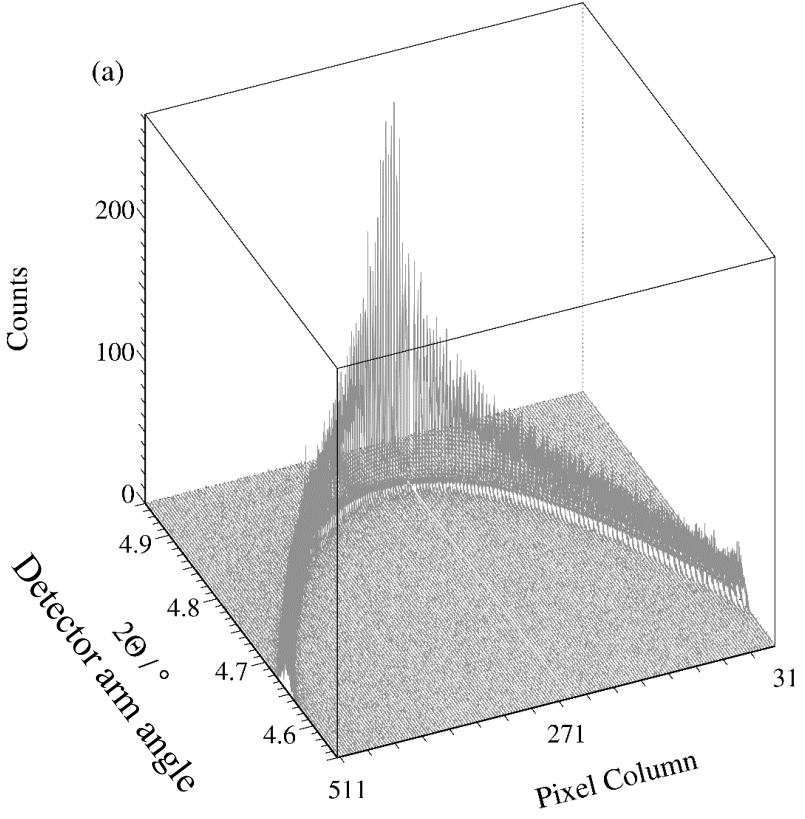


Debye Scherrer cones

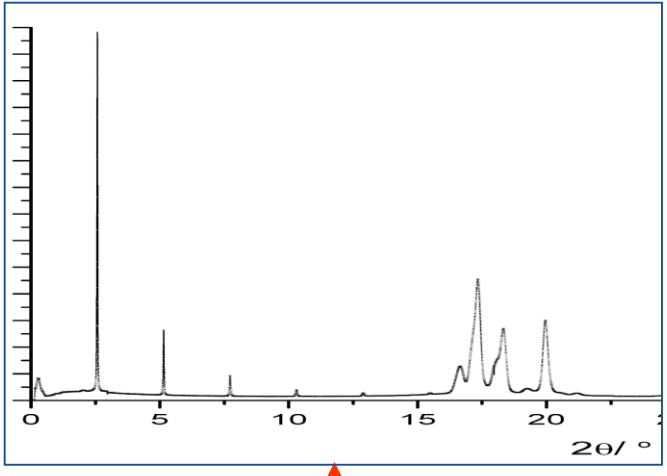


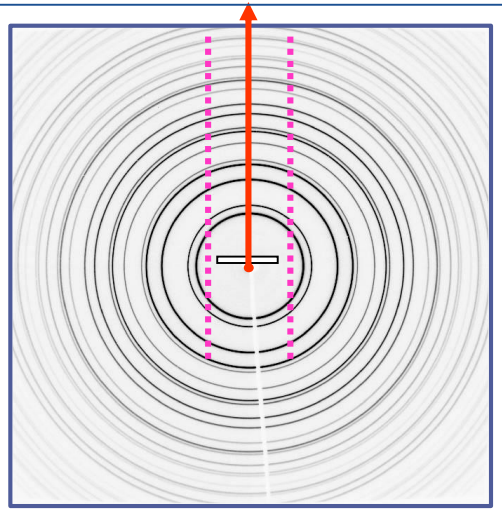
Debye Scherrer cones

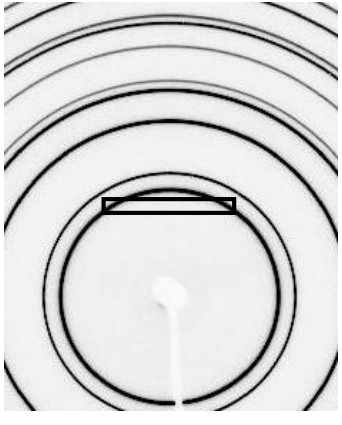


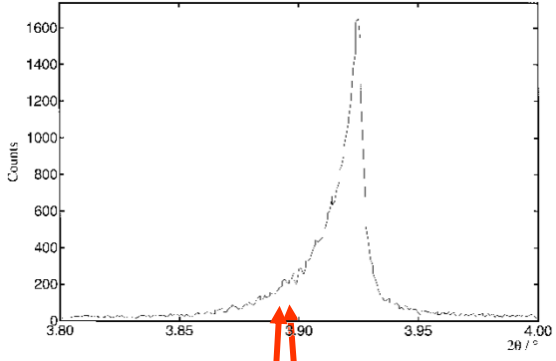


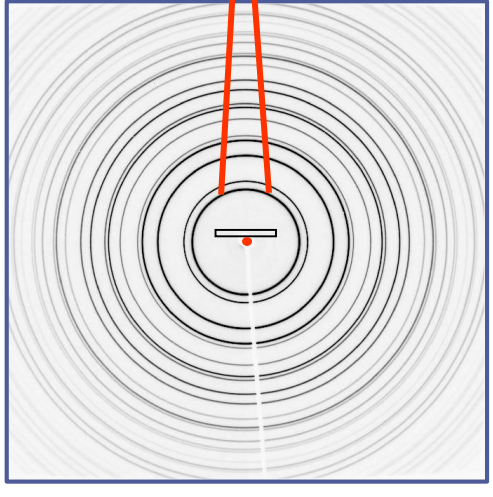
Axial divergence ⇒ asymmetry











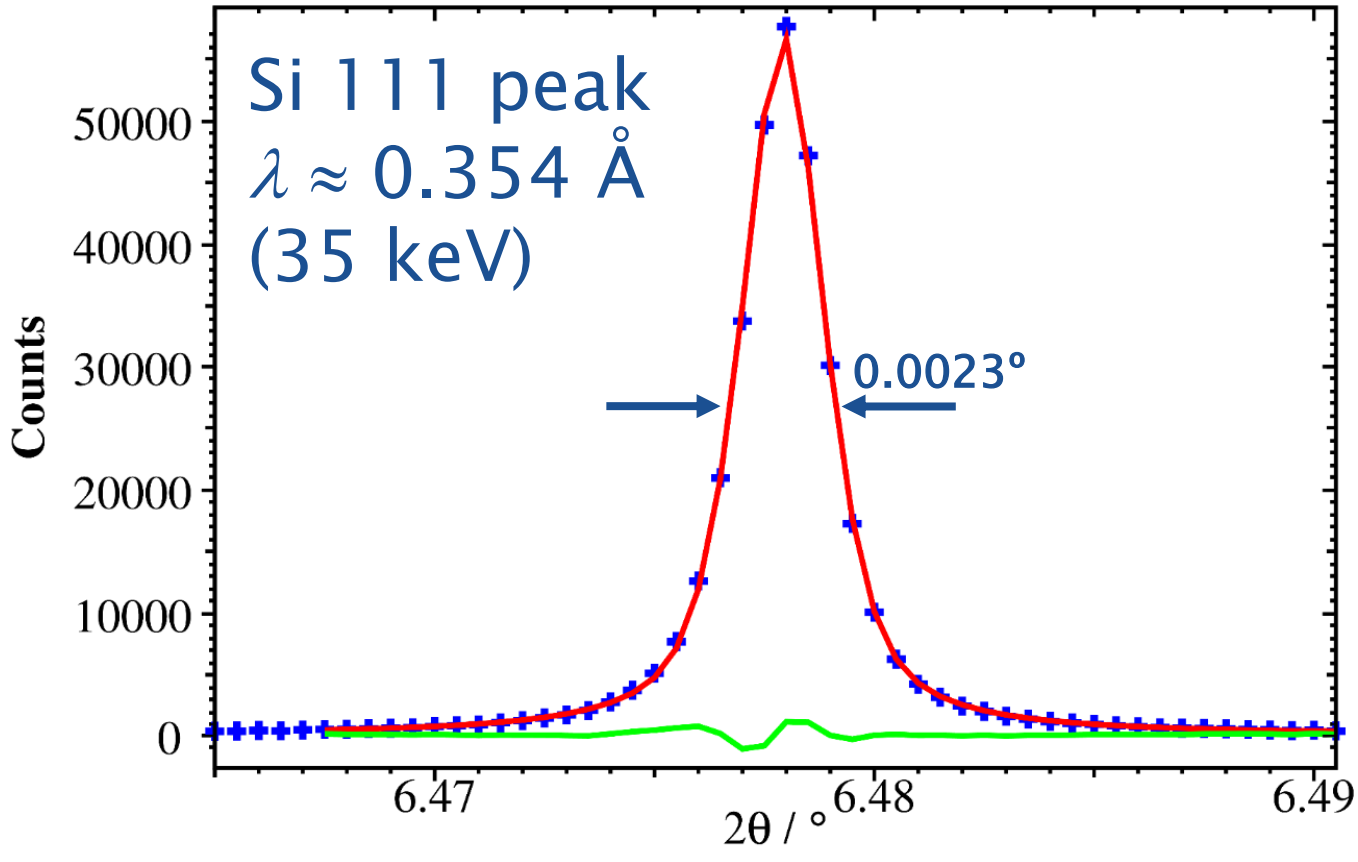
Analyser crystal + 2D Eiger pixel detector

Allows elimination of low-angle peak asymmetry due to axial divergence by exploiting the axial (horizontal) resolution of the detector.

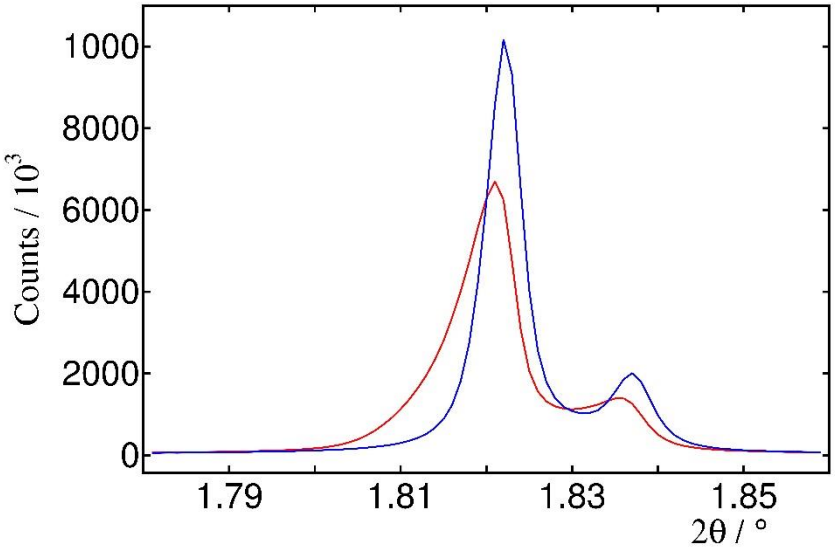
- Improves peak shapes which become more symmetric
- Reduces peak widths, so improves angular resolution
- Exploit the full width of the detector (38 mm) at higher 2θ angles, so improves the statistical quality of the data

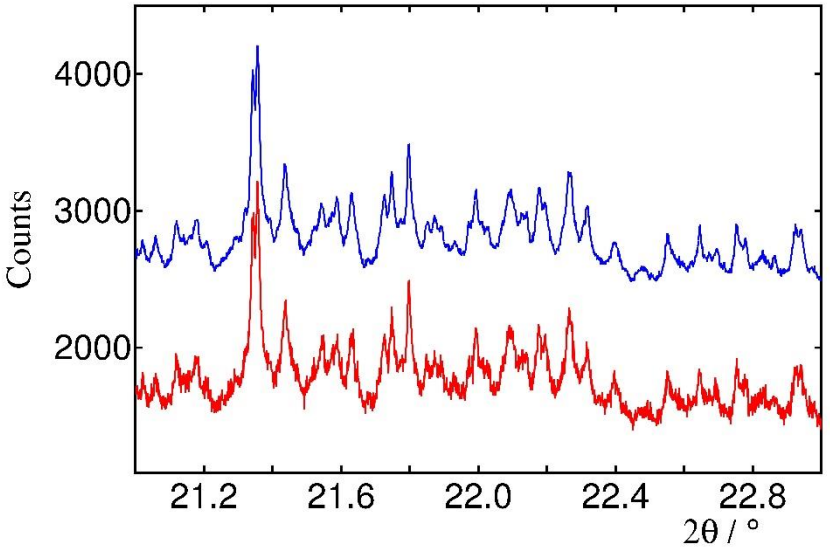
Dejoie *et al*. *J. Appl. Cryst*. 51, 1721-1733 (2018); Fitch & Dejoie, *J. Appl. Cryst.* 54, 1088-1099 (2021)

High angular resolution, symmetric peak



Zeolite ZSM-5

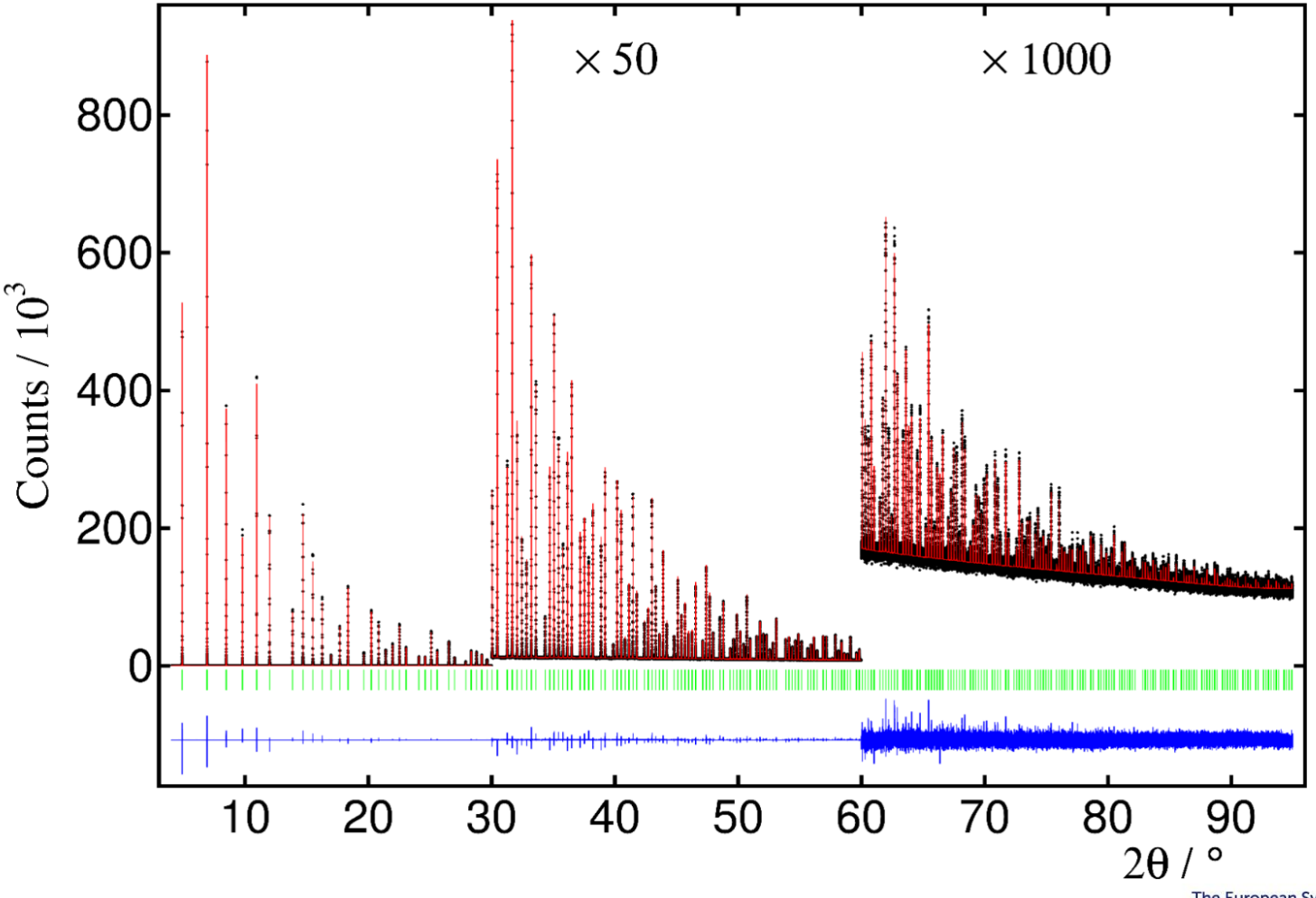




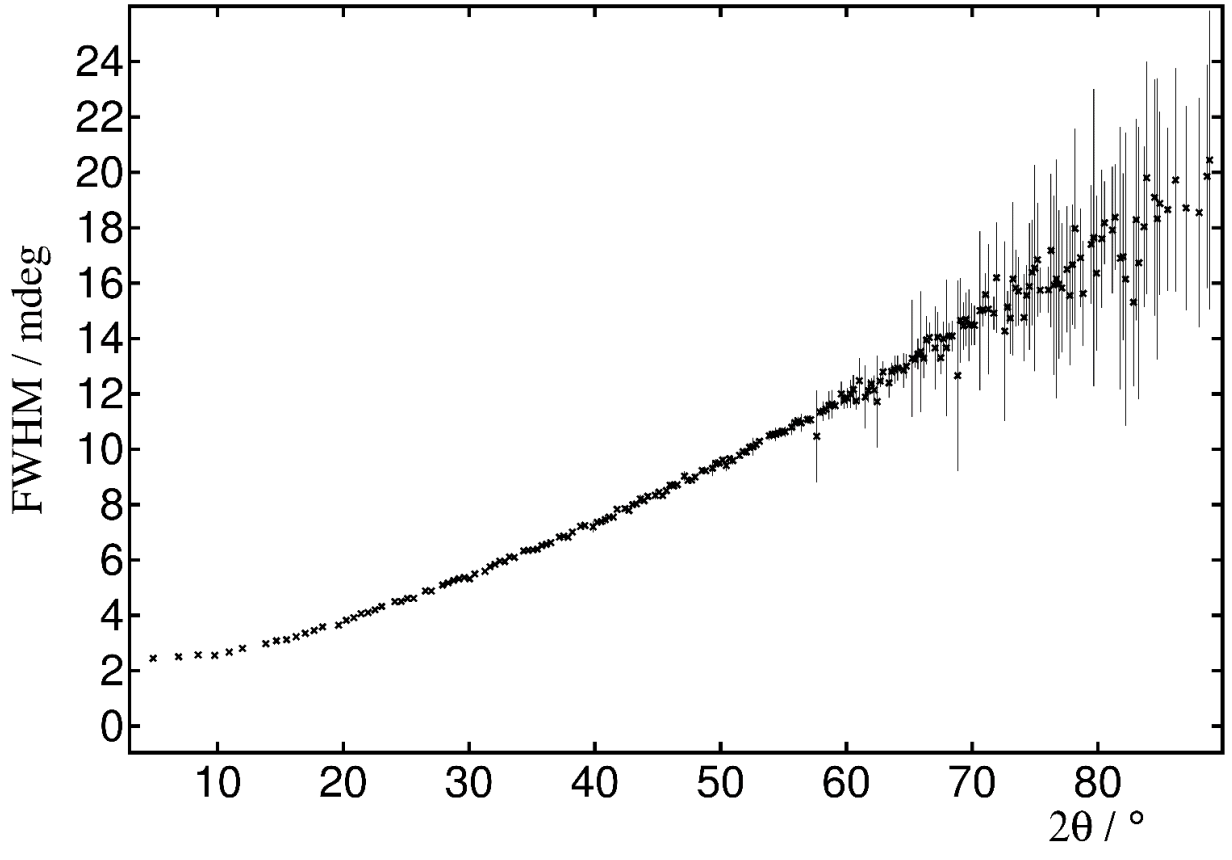
Uncorrected
Corrected

Uncorrected
Corrected, (offset by 1000)

Rietveld fit to LaB_6 at 35 keV

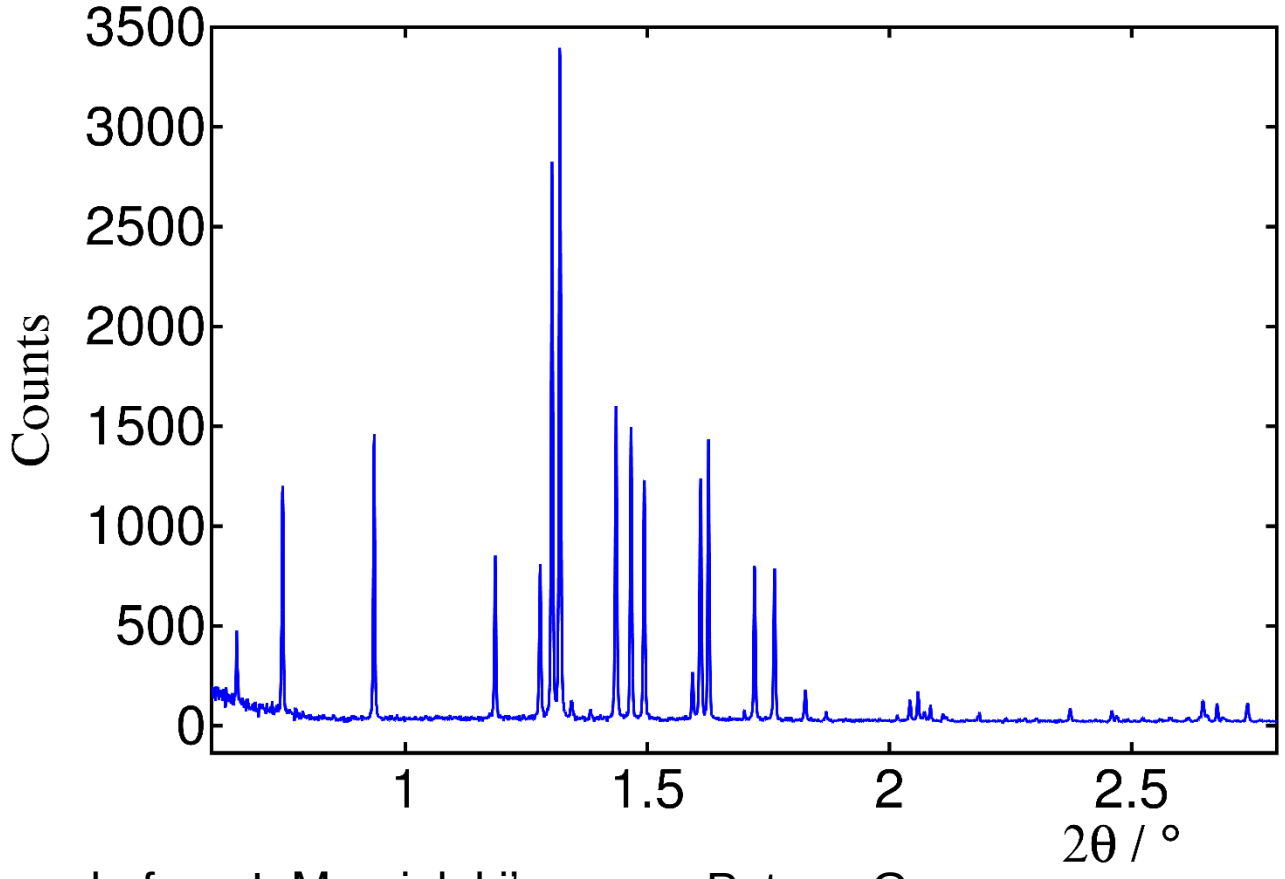


FWHM of LaB_6 at 35 keV



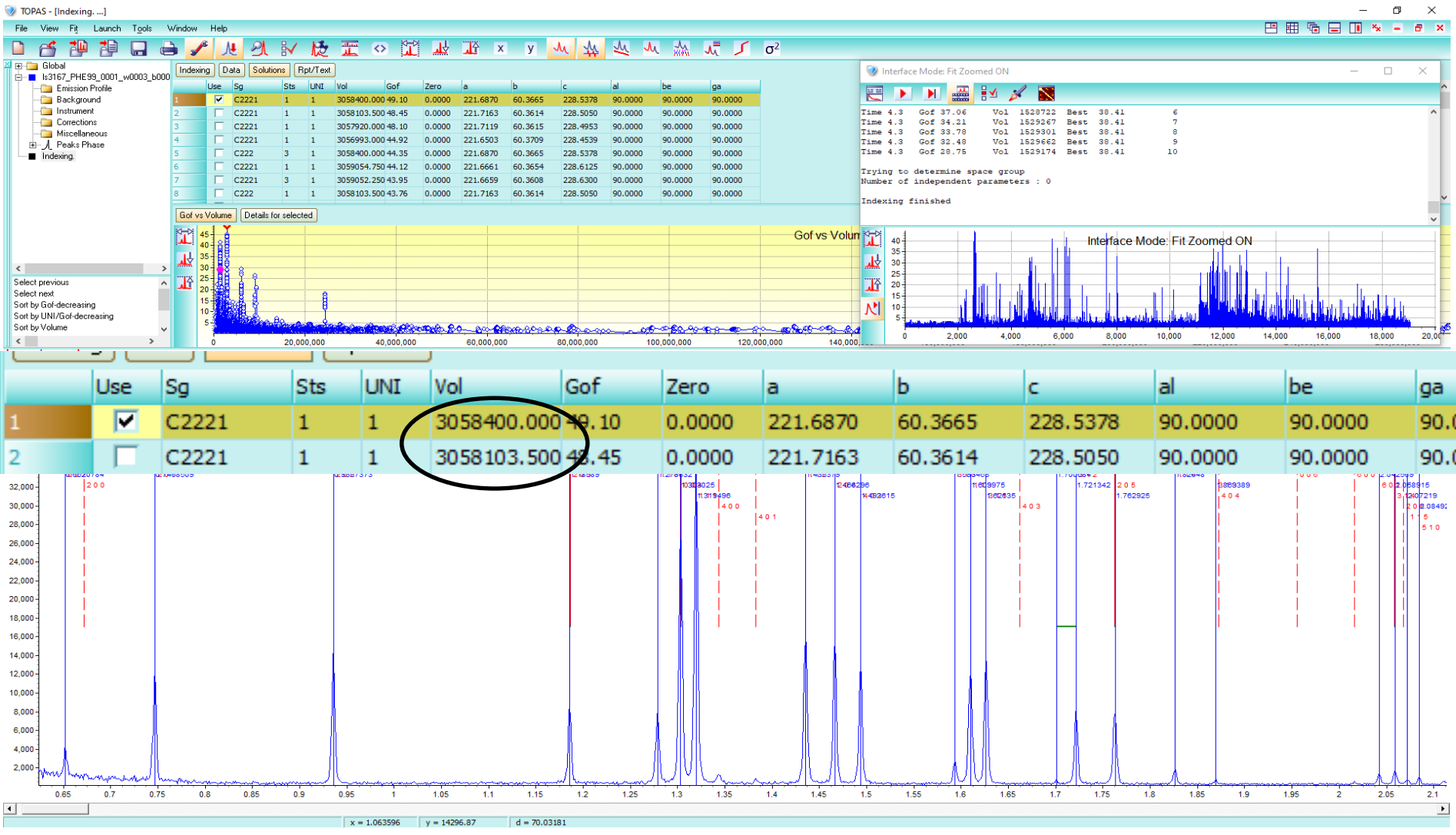
Polymorph of insulin (λ = 1.3 Å)

2θ corrected



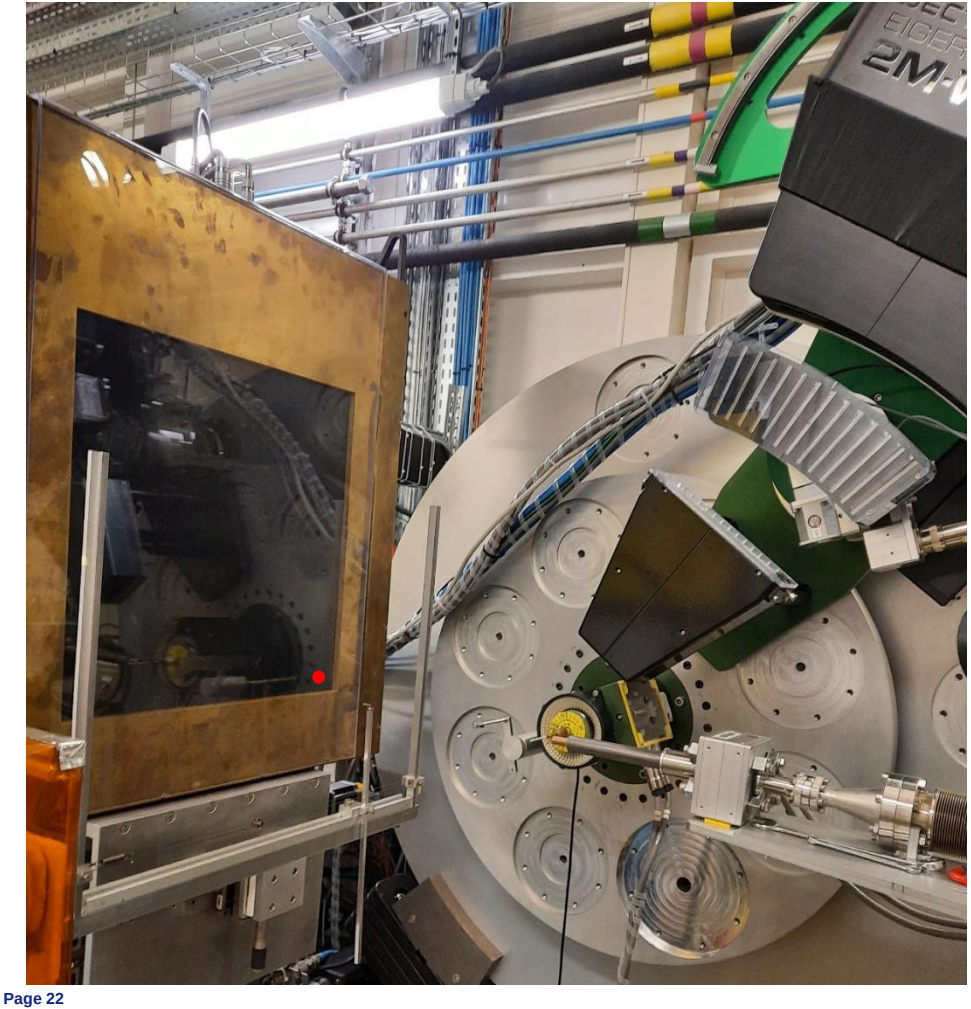
Insulin sample from I. Margiolaki's group, Patras, Greece

Polymorph of insulin (λ = 1.3 Å)

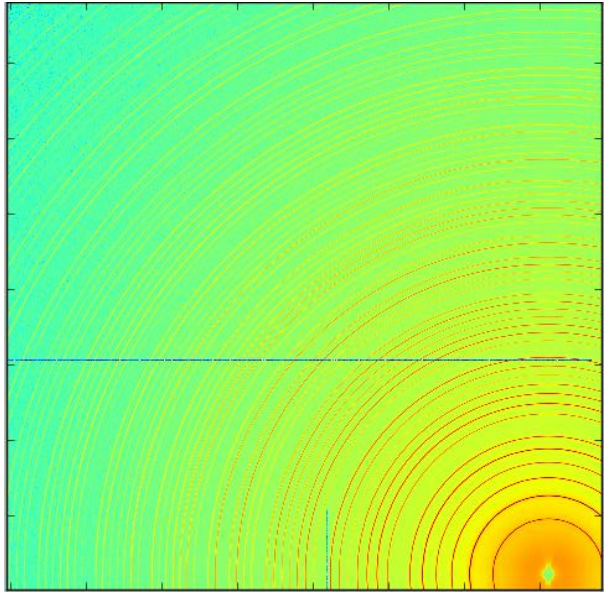


	Use	Sg	Sts	UNI	Vol	Gof	Zero	a	b	c	al	be	ga
1	☑	C2221	1	1	3058400.000	49.10	0.0000	221.6870	60.3665	228.5378	90.0000	90.0000	90.0
2	☐	C2221	1	1	3058103.500	48.45	0.0000	221.7163	60.3614	228.5050	90.0000	90.0000	90.0

Large 2d medical-imaging detector



41 × 41 cm² Perkin Elmer XRD 1611 medical-imaging detector for measurements up to 75 keV.

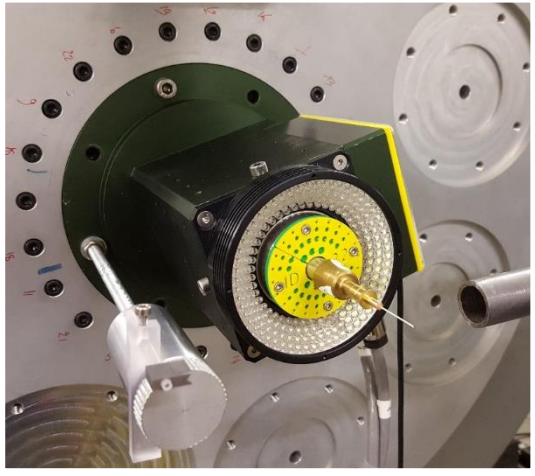


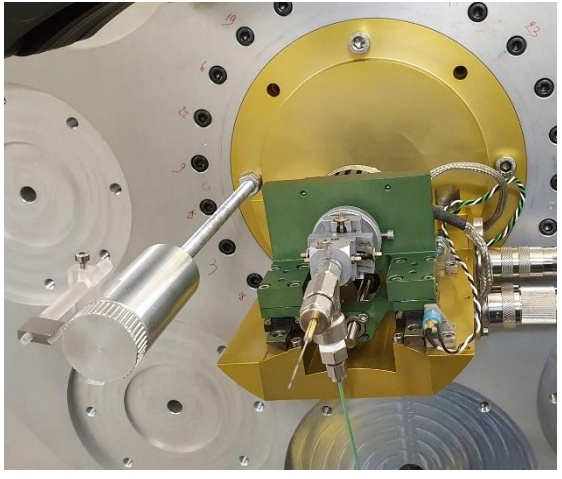
Particular attributes of ID22

- Very high 2θ resolution
- High energies (6–75 keV)
- Standard high-resolution operation = 35 keV (0.354 Å)
  $\Rightarrow$ penetrate through absorbing samples
  $\Rightarrow$ spinning capillary samples for all powder specimens
  $\Rightarrow$ versatility in sample environments
- Automatic correction for axial divergence
- High intensity
- 2D medical imaging detector for complementary measurements (e.g. PDF analysis)

Sample spinners/stage

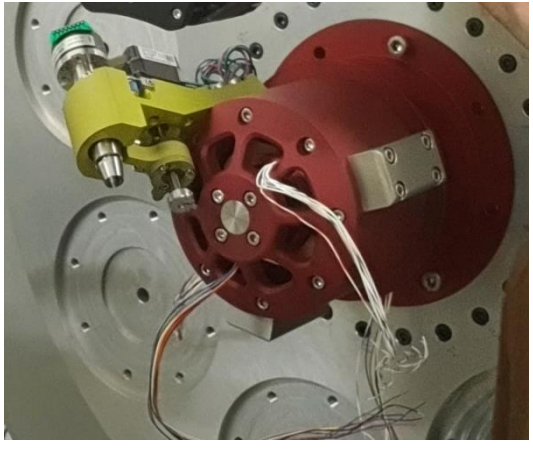
Green spinner

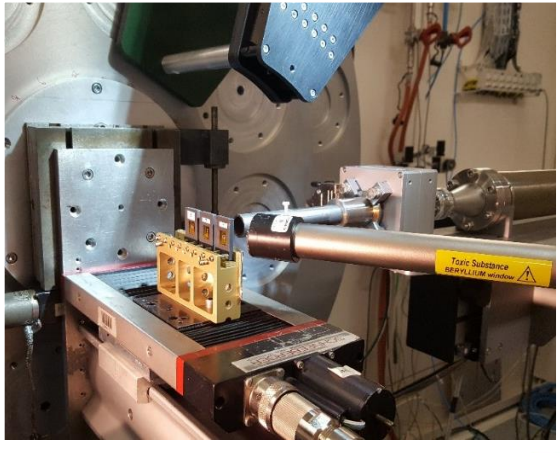




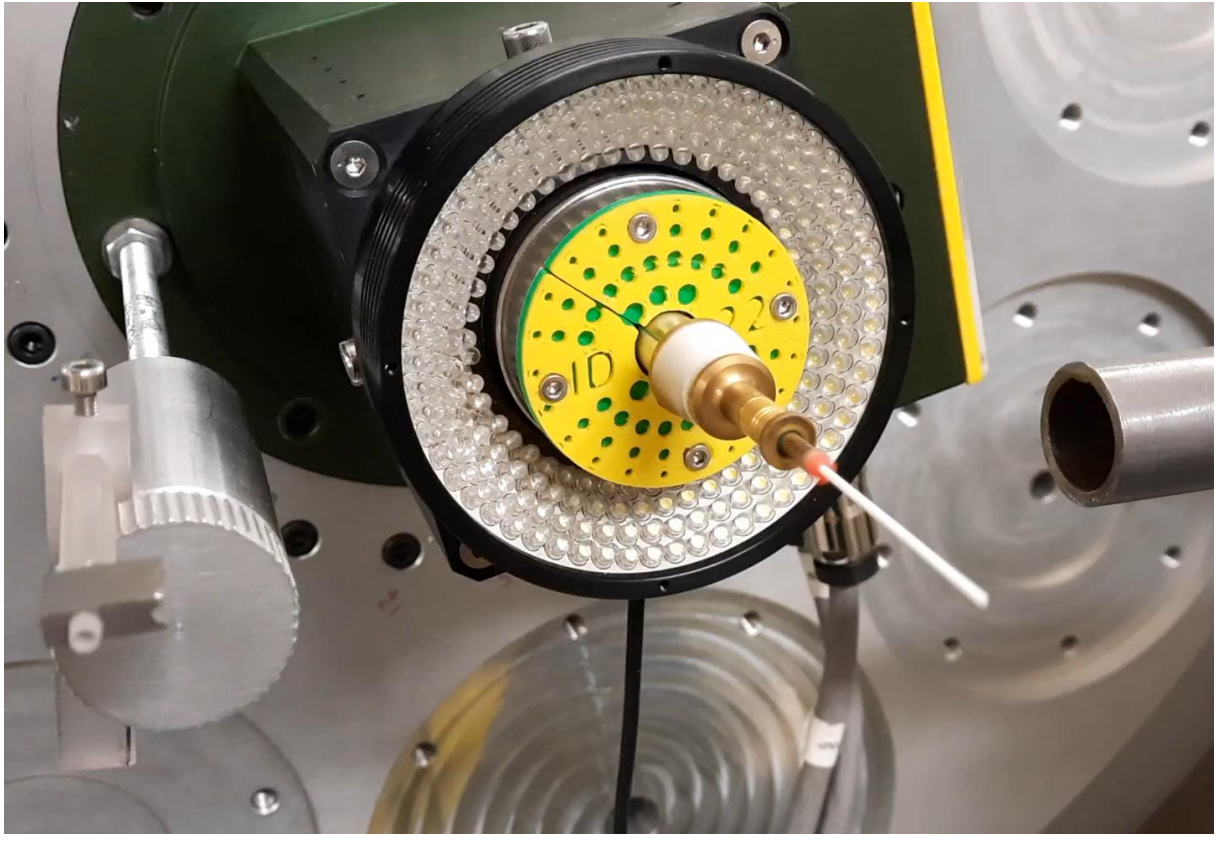
Yellow spinner

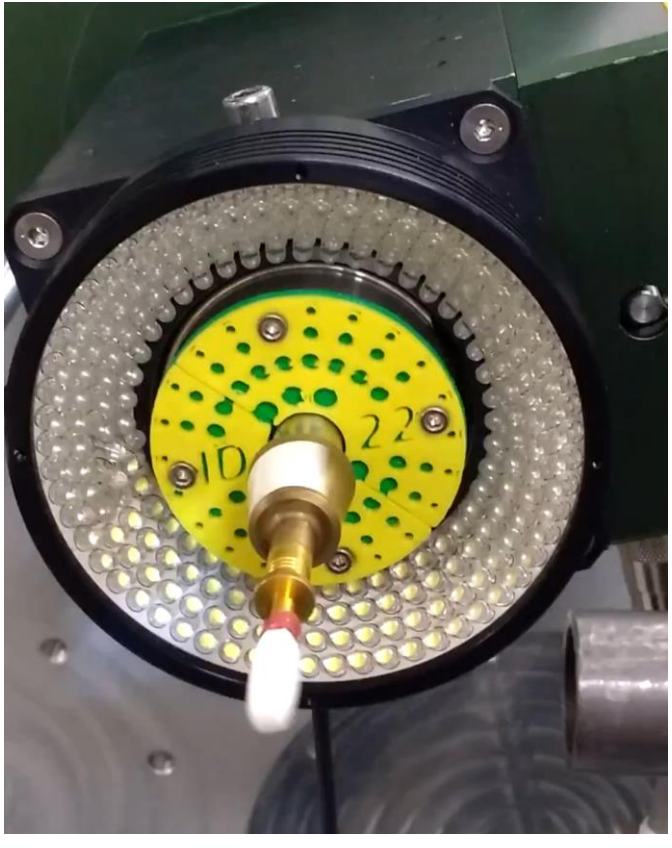
Red spinner

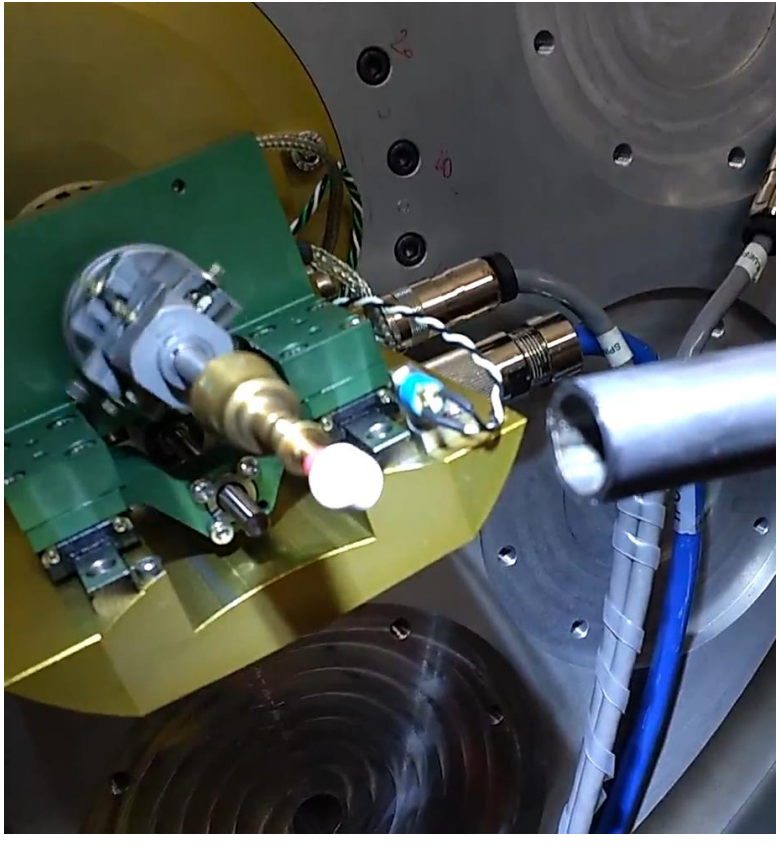




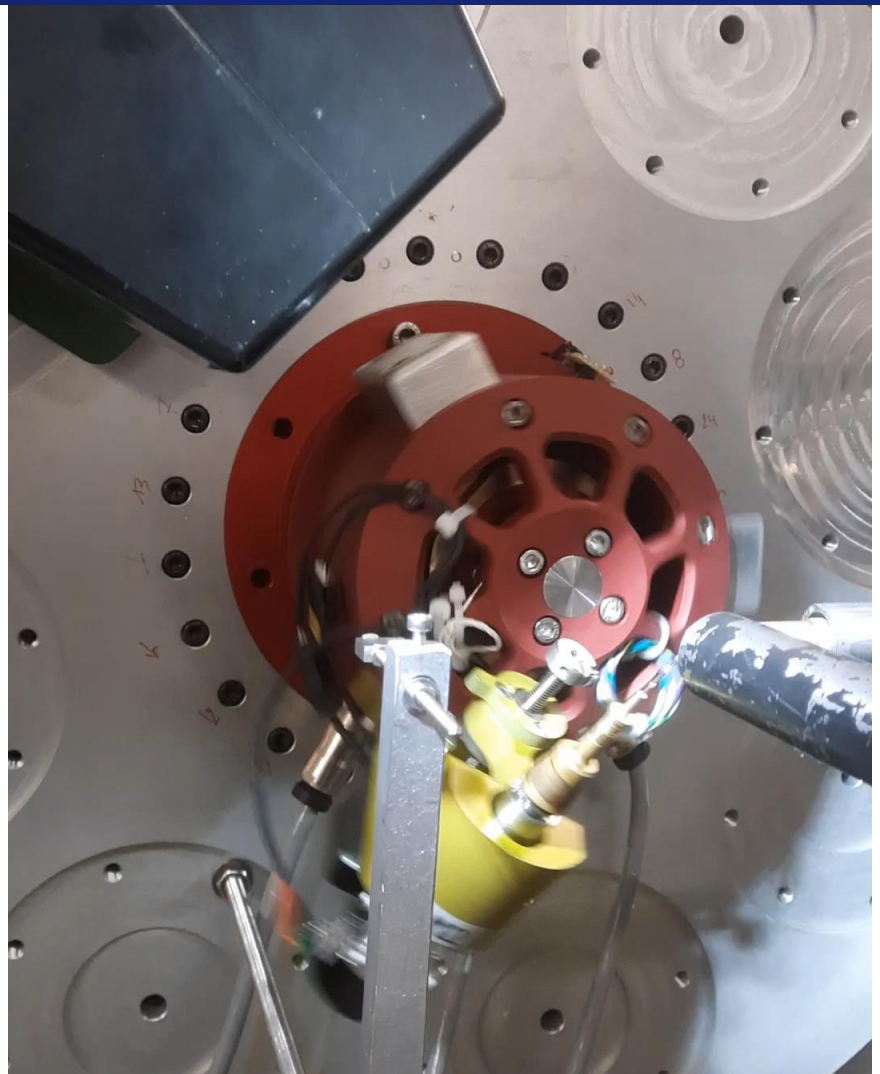
XYZ stage


ID


ID 22



Red spinner



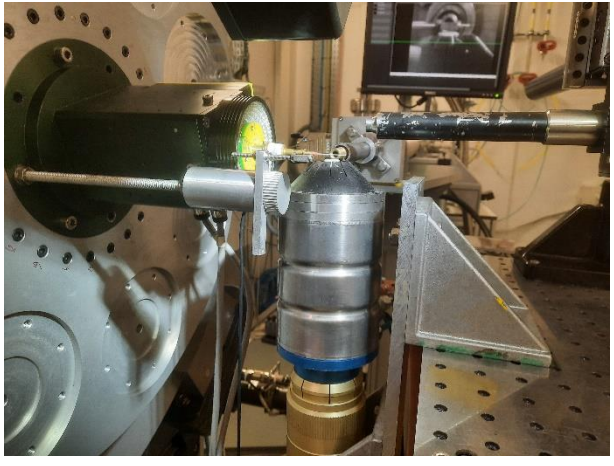
Powder Diffraction = Sample Environments

Routine temperature range ≈4 K – 1600°C

- Cryostream N_2 gas blower, 80–500 K
- Liquid-He flow cryostat, down to 4 K
- Hot air blower, 950°C
- Mirror and induction furnaces, ≈1600°C
- Gas adsorption cell (0–100 Bar)
- Robotic 75 sample changer

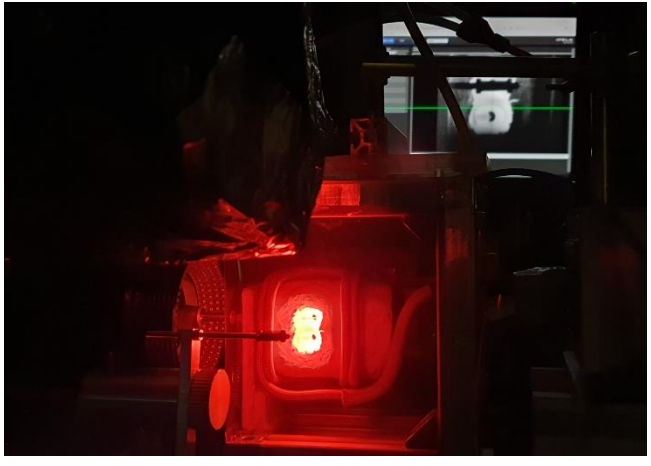
All computer controlled and linked to scans

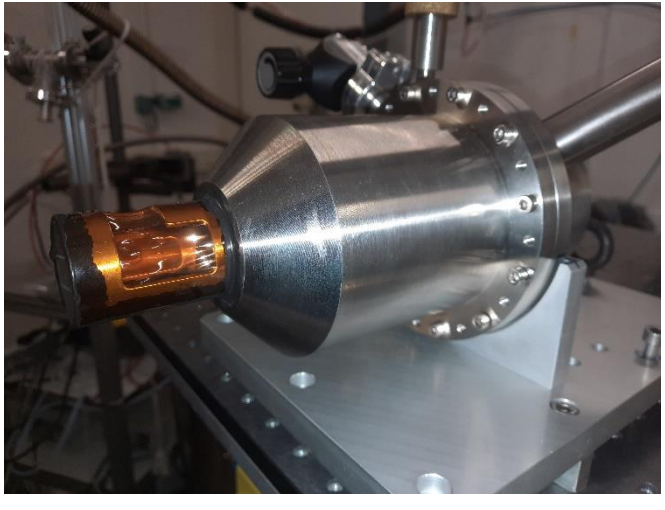
Sample environments



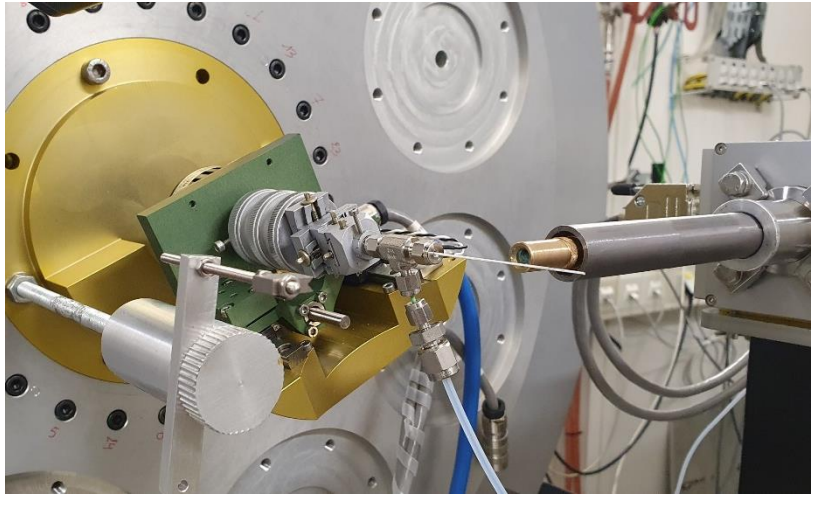
Cryostream & blower ⇐

Induction furnace ⇒



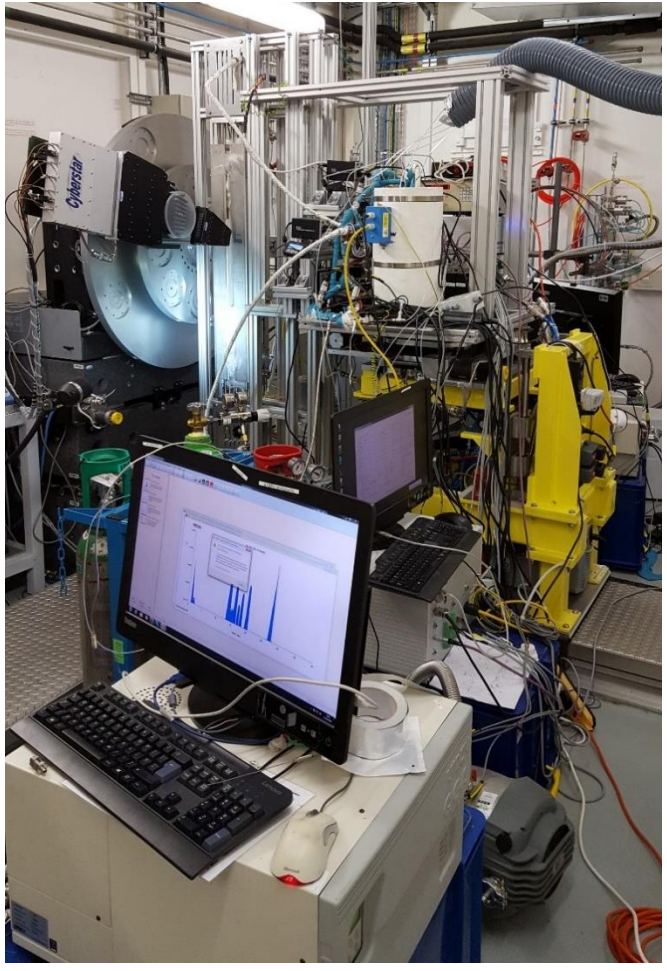


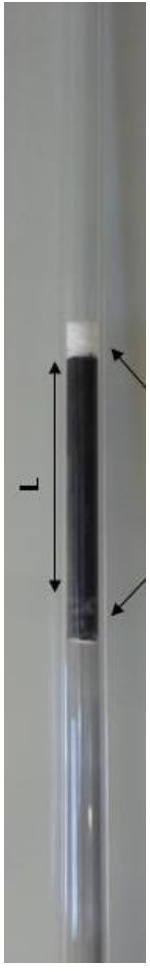
Liquid-He cryostat

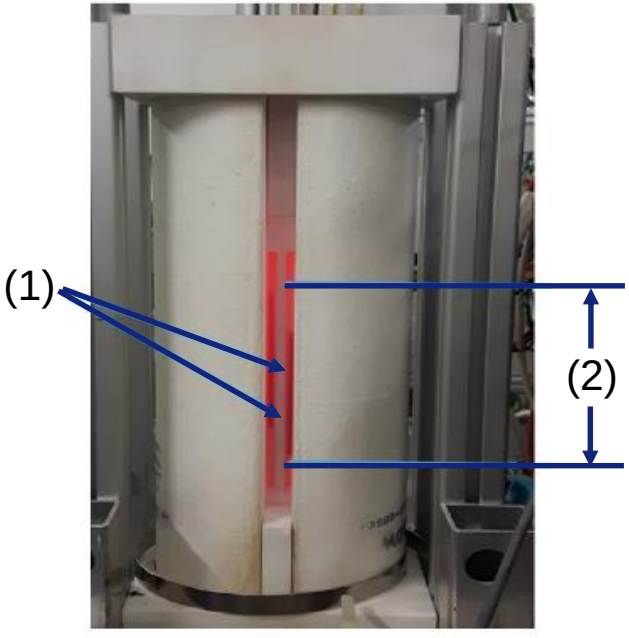


Gas cell

User's *in-situ* catalytic reaction setup





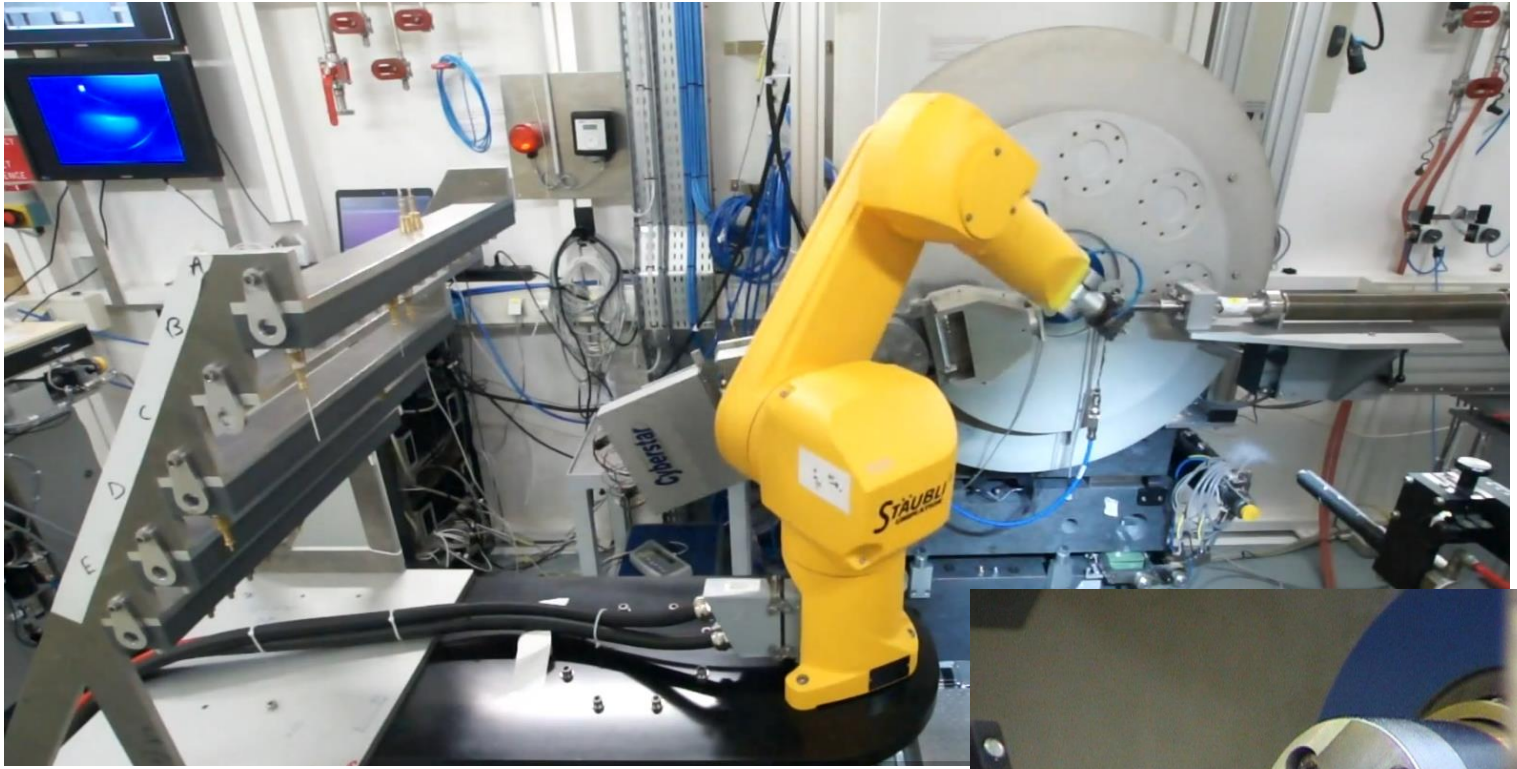


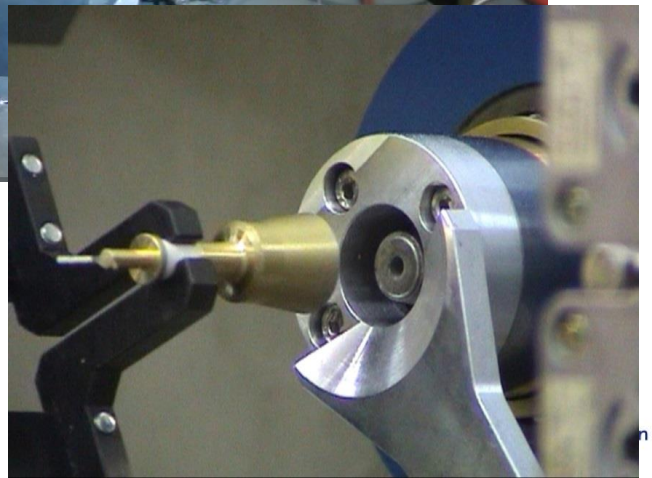
(1) Two reactor beds in parallel

(2) 15 cm isothermal zone (820°C +/- 15°C)

I. Metcalfe *et al.*

Robotic sample changer





See our YouTube video!!

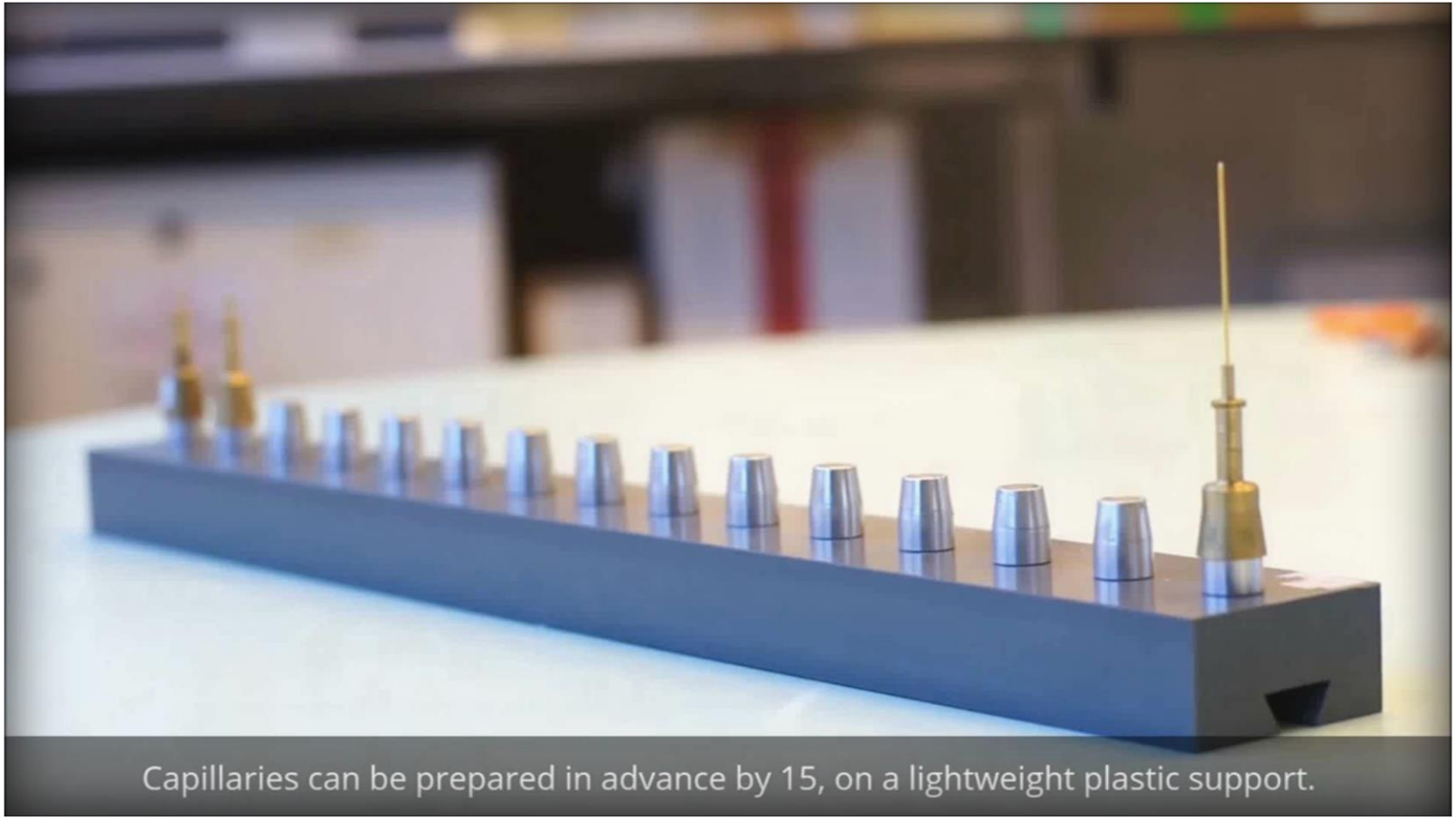
https://www.youtube.com/watch?v=OEhf8Logz44

75 samples

Up to 75 samples, in 5 banks of 15

Not just capillaries

https://www.youtube.com/watch?v=OEhf8Logz44



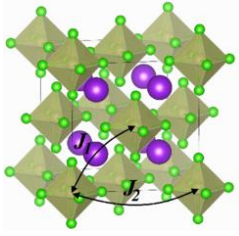
What sorts of experiments are performed at ID22?

- Structural studies – crystal structures, atomic PDF analysis, etc.
- *In-situ* studies – evolution with temperature, time, atmosphere, voltage, etc., phase changes, solid-state chemistry, gas adsorption, electrochemistry.
- Anomalous scattering – distinguish neighbouring elements in the Periodic Table.
- High throughput – many samples, varied compositions or preparation conditions, etc.
- Quantitative analysis – many phases, trace phases.
- Microstructure – detailed analysis of peak shapes.
- Residual strain – mapping peak positions in components.
- Anything you can fit on. Very flexible instrument!

Applications

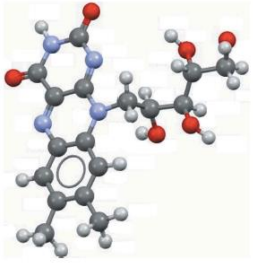
Materials Science

Physics



Khan *et al*. PRB 99, 144425 (2019)

Structural chemistry

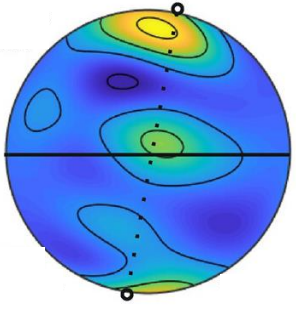


Guerain *et al*. Acta Cryst. C77, 800 (2021)

Cultural Heritage

Autran *et al*. Sci. Rep. 13, 524 (2023)

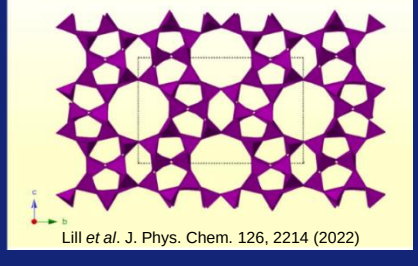
Geosciences



Kuehn *et al*. Geo. Res. Lett. 48 (2021)

Industry

Environment and Energy



Lill *et al*. J. Phys. Chem. 126, 2214 (2022)

(*R*)–rasagiline mesylate

Acta Cryst B STRUCTURAL SCIENCE CRYSTAL ENGINEERING MATERIALS

ISSN 2052-5206

Hydrogen bonding patterns and C—H···π interactions in the structure of the antiparkinsonian drug (*R*)-rasagiline mesylate determined using laboratory and synchrotron X-ray powder diffraction data

Analio J. Dugarte-Dugarte,[a]‡ Robert A. Toro,[b] Jacco van de Streek,[c] José Antonio Henao,[b] Andrew N. Fitch,[d] Catherine Dejoie,[d] José Miguel Delgado[a] and Graciela Díaz de Delgado[a]*

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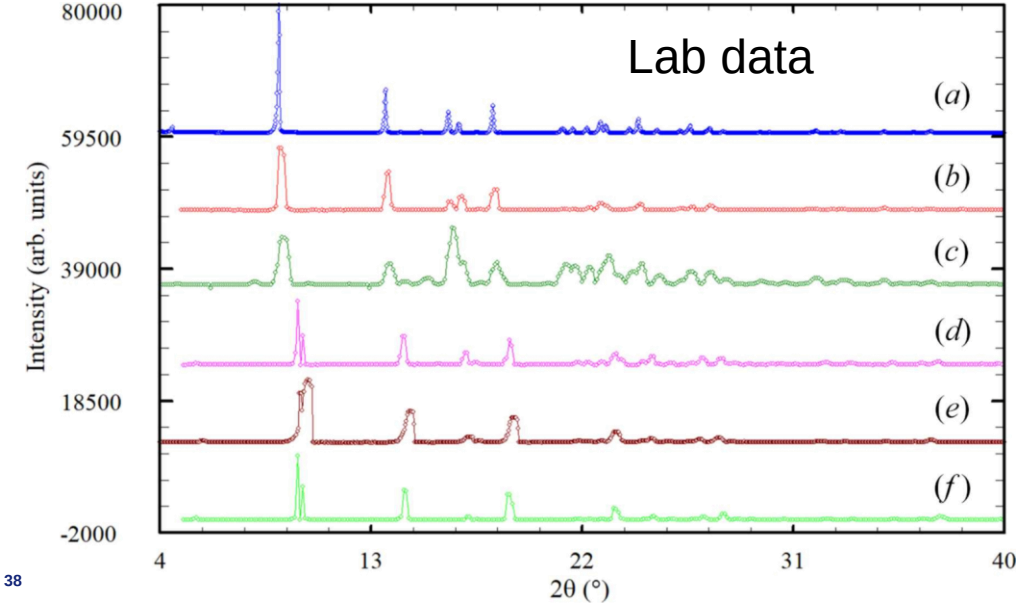

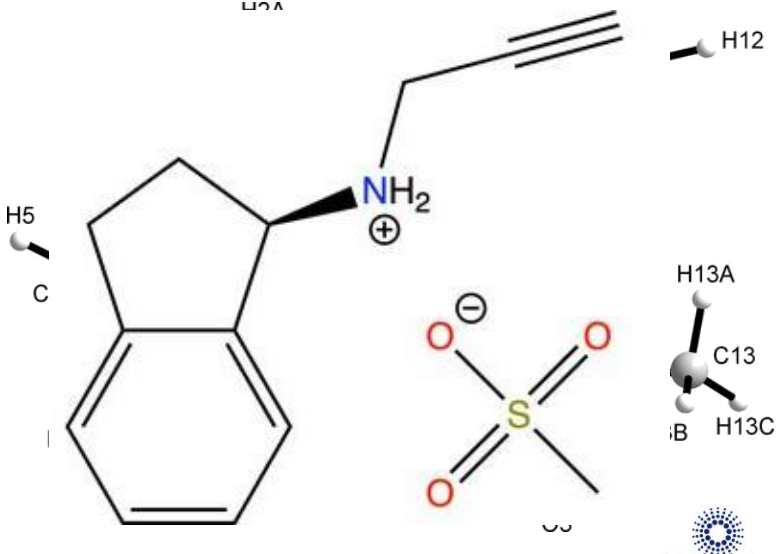

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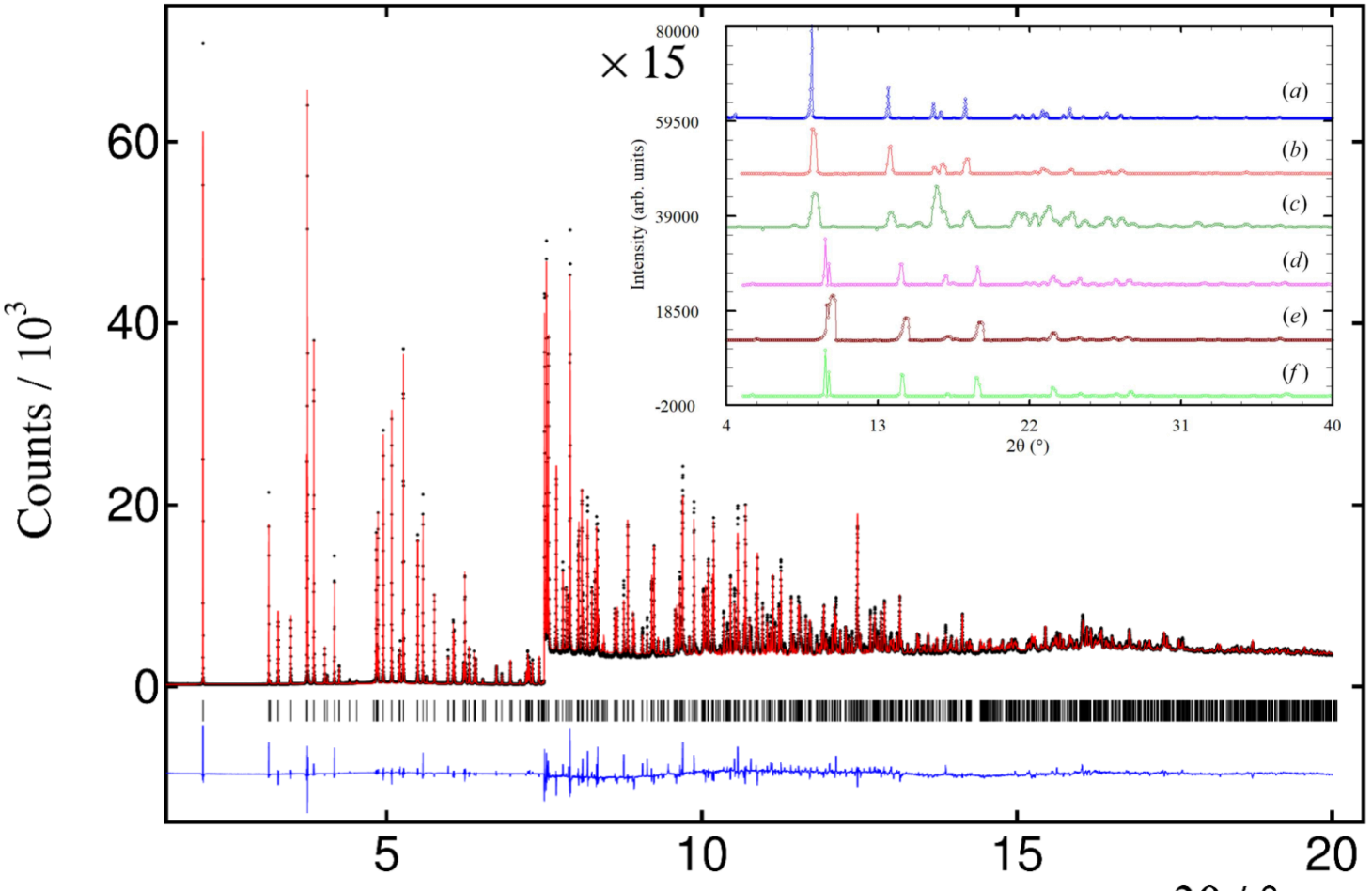
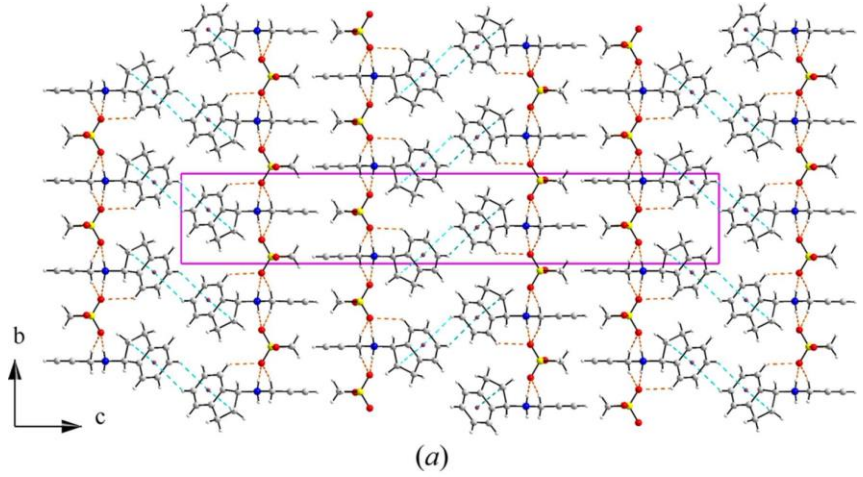
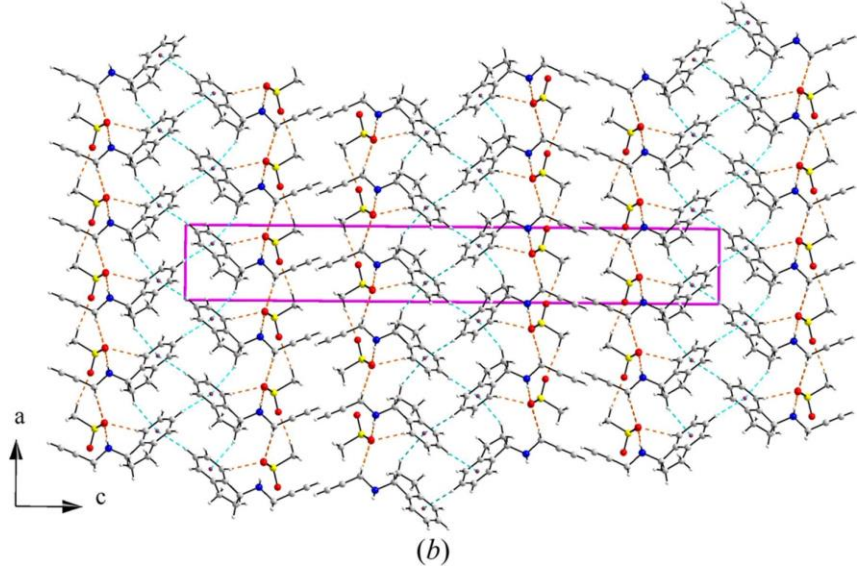
Rietveld fit of solved structure



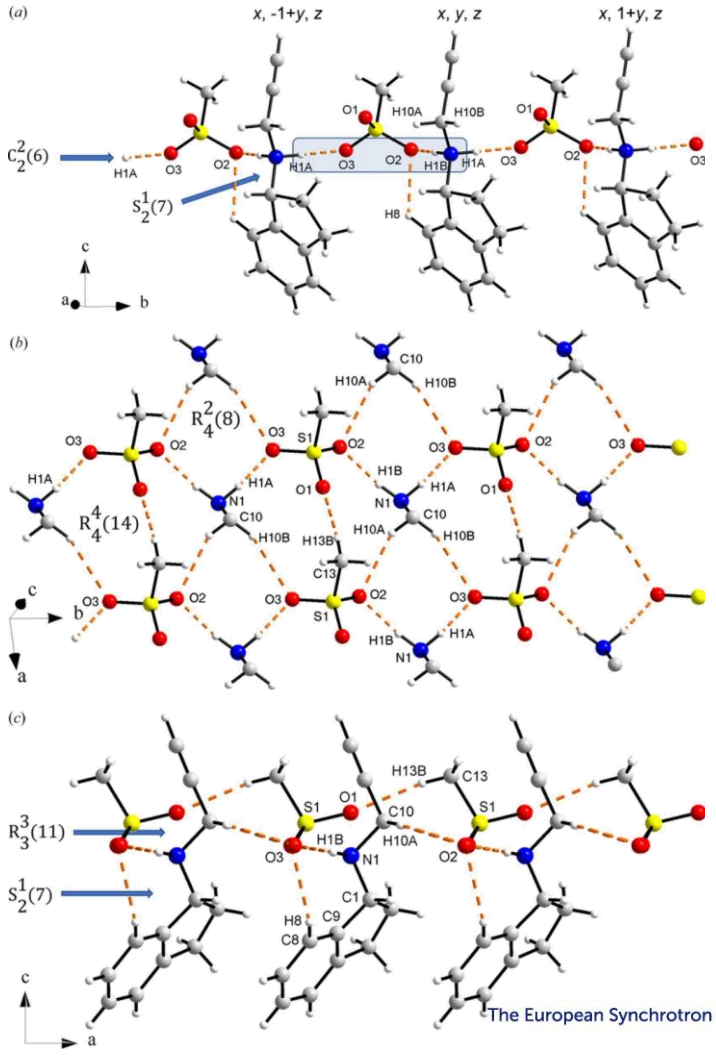
Molecular packing / hydrogen bonding



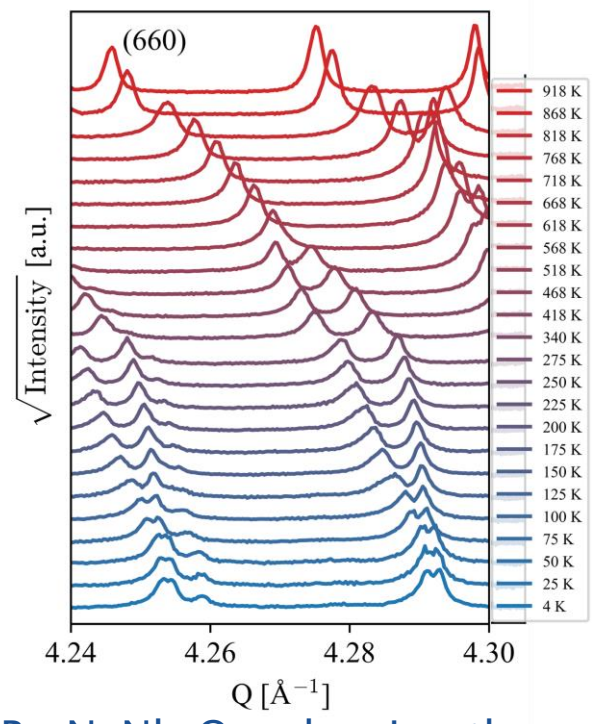
(a)



(b)

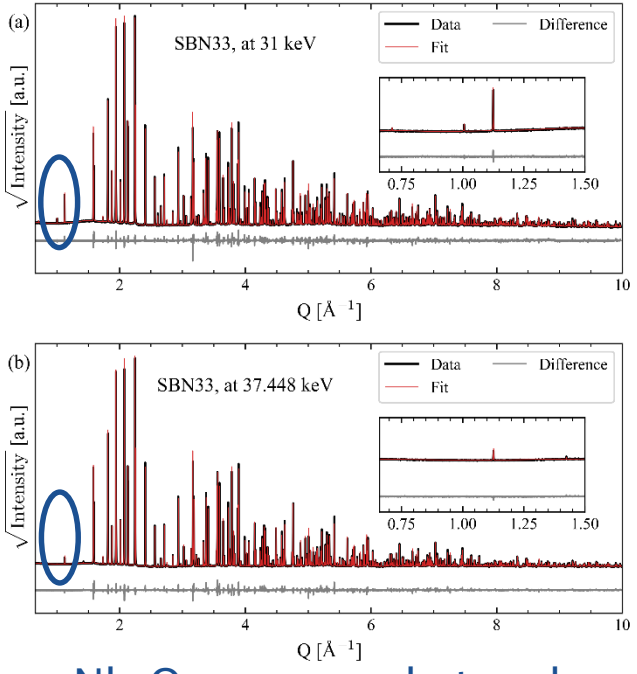


Tetragonal tungsten bronzes



$Ba_2NaNb_5O_{15}$ showing the orthorhombic splitting of the 660 tetragonal peak that persists to 4 K

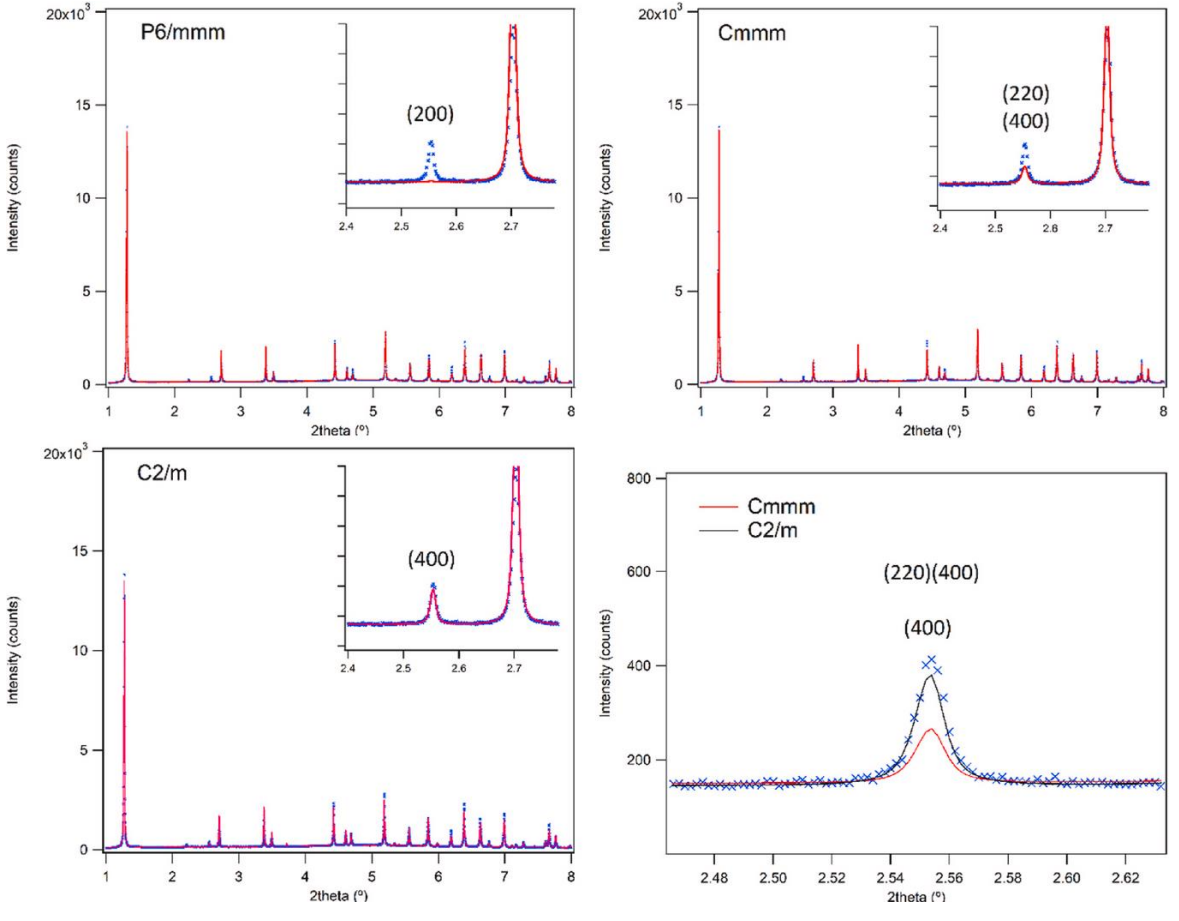
Grendal *et al*. *J. Appl. Cryst.* 56, 1456 (2023)

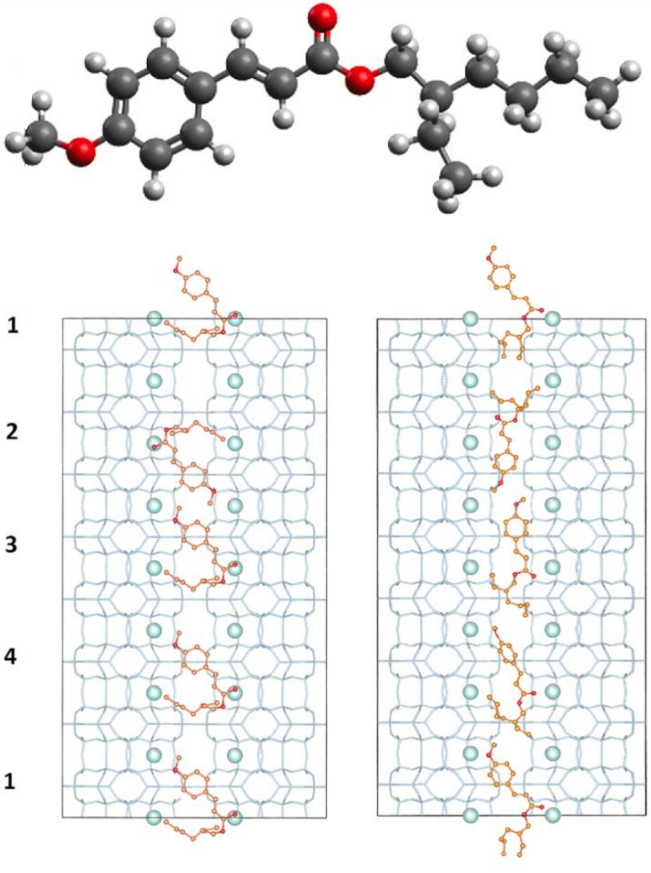


$Sr_xBa_{1-x}Nb_2O_6$ measured at and away from the Ba K edge, showing subtle changes in peak intensities from which the Ba/Sr distribution can be deduced.

Grendal *et al*. *ACS Omega* 8, 37592 (2023)

Sunscreen adsorbed in zeolite-L





Confalonieri *et al. Micropor. Mesopor. Mat.* 344, 112212 (2022)

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ID22 – the high-resolution powder-diffraction beamline at ESRF

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