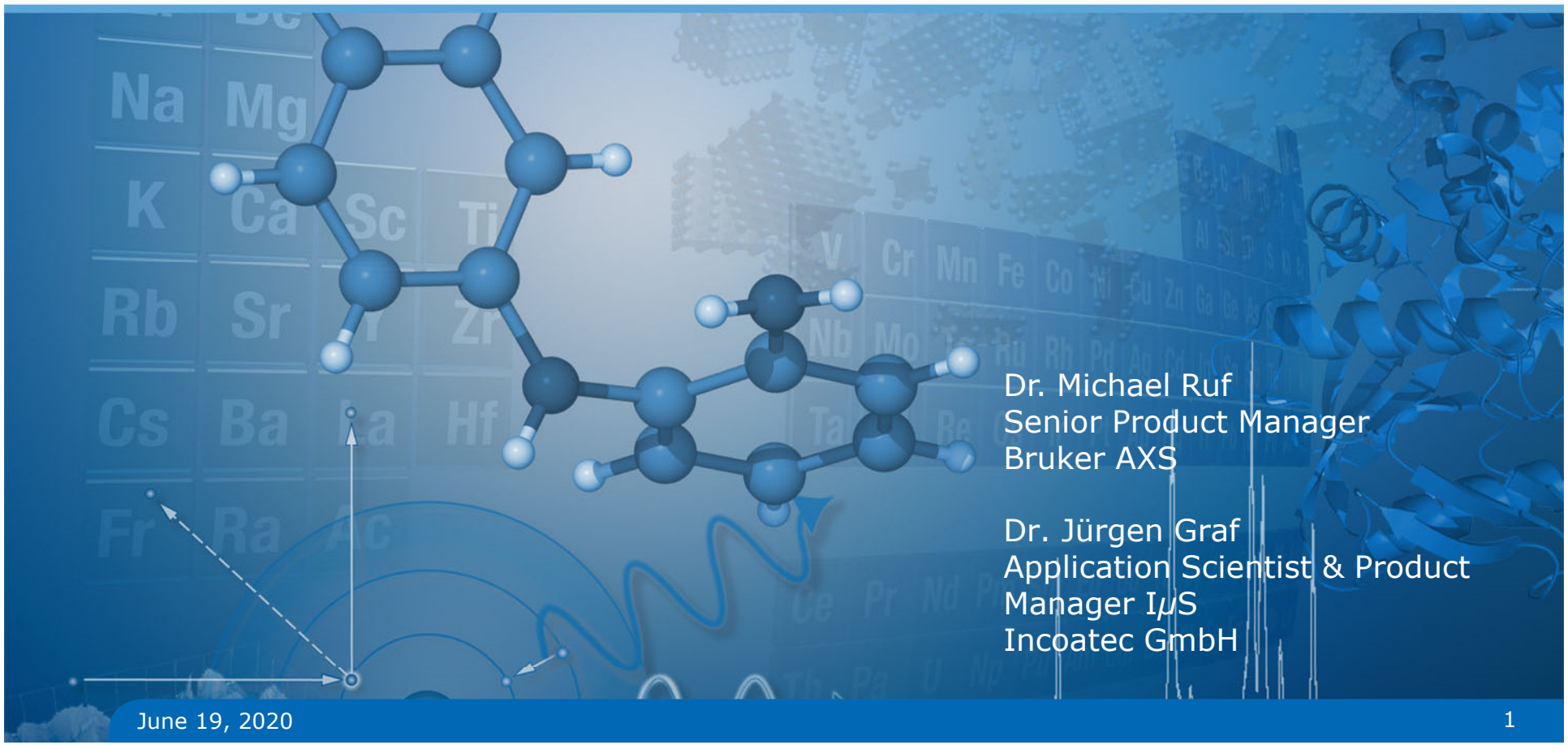
Bruker Educational SC-XRD Webinar

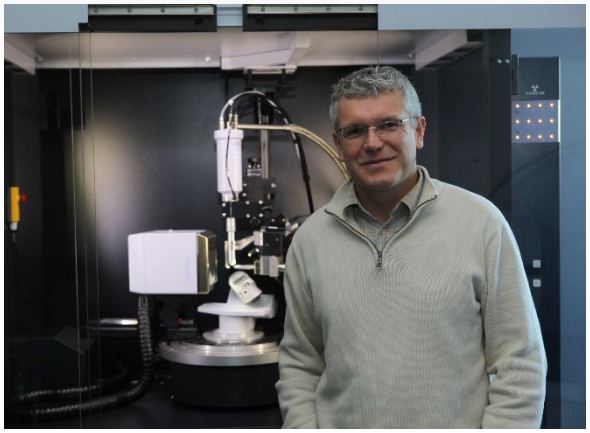
Smallest Crystals: How Modern Microfocus Sources Can Help You Determine the Structure From Weak Diffractors



Dr. Michael Ruf
Senior Product Manager
Bruker AXS

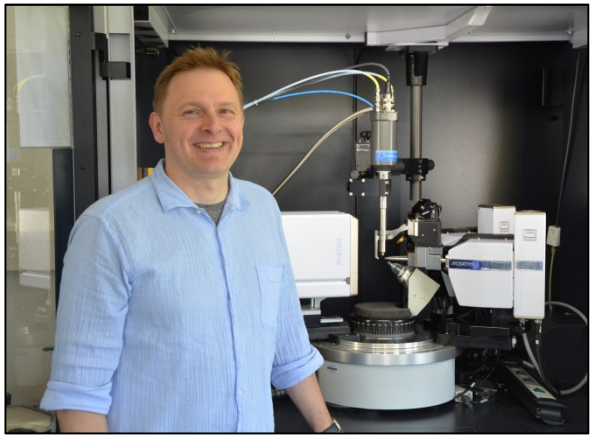
Dr. Jürgen Graf
Application Scientist & Product Manager IμS
Incoatec GmbH

Who is talking?



Dr. Michael Ruf:

- In Madison, USA
- Senior Product Manager
- PhD in Chemistry
- Joined Bruker AXS in 1998
- Michael.Ruf@bruker.com

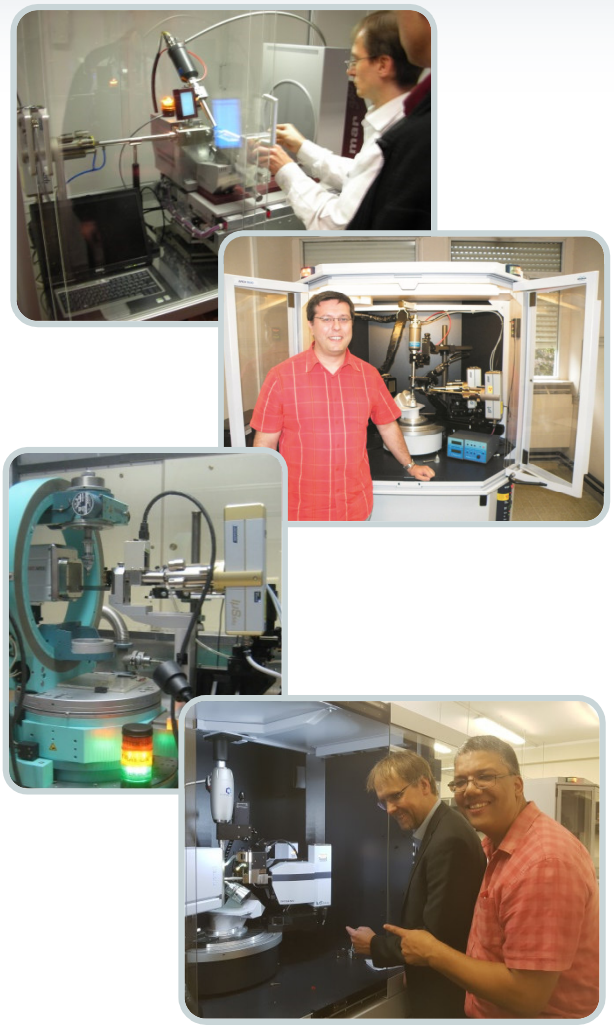


Dr. Juergen Graf:

- In Geesthacht, Germany
- Application Scientist & Product Manager IμS
- PhD in Chemistry
- Joined Incoatec in 2004
- Juergen.Graf@incoatec.de

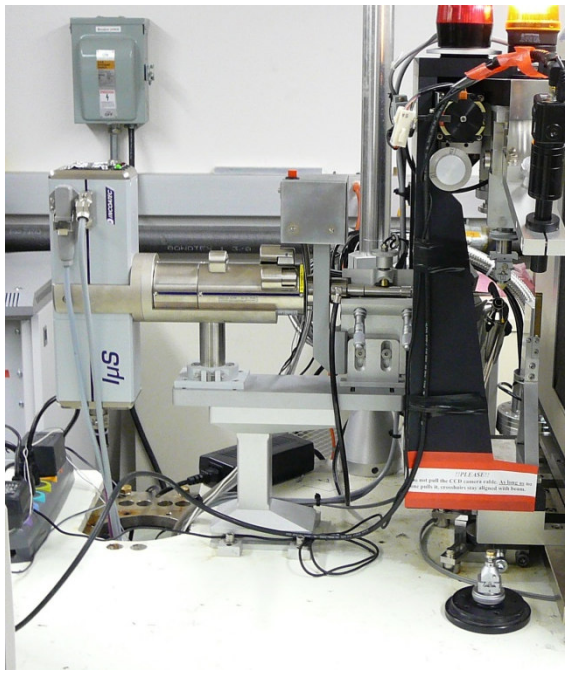
The IμS Success Story

2002	Foundation of Incoatec as joint venture with Bruker
2006	Cu-IμS for PX and SAXS
2007	1st IμS is installed at Univ. Kiel
	Mo-IμS for Chemical Crystallography
	Cu-IμS for XRD²
2009	Ag-IμS for Solid State Chemistry,
	High-Pressure and Charge Density Research
2010	100th IμS is installed at CNRS Lille
2011	IμS$^{\text{High Brilliance}}$ family with D8 VENTURE
2015	500th IμS Customer at Univ. Göttingen
	IμS 3.0 (AsCA Kolkata)
2017	**IμS DIAMOND** (IUCr Hyderabad)
2020	1000th IμS is installed at Univ. Cape Town

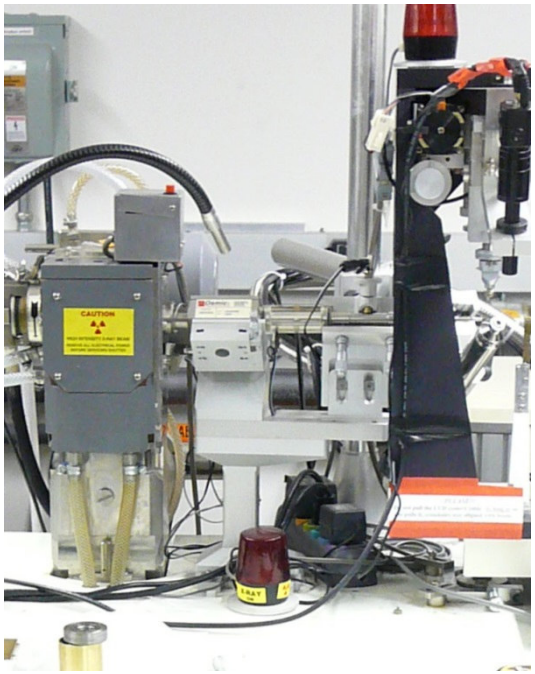


Revolution without Revolutions

- One of the first upgrades: Replacing a 4 kW rotating anode by a 30 W IμS used for protein screening



30 W Cu-IμS plus IP Detector



4 kW Rotating Anode plus IP Detector

IXT Manufacturing at INCOATEC

The IμS 3.0: Microfocus Made Perfect

- The IμS 3.0 is the first and only source **designed** specifically **for crystallography** with no compromises

- IμS 3.0 features
  - more than **twice the intensity** of competing 30 W microfocus sources!
  - about **50% more intensity** compared competing 50 W HB-class microfocus sources!

- Complete **mechanical redesign** for maximum customer convenience
- No more vacuum pump

- Same extraordinary **reliability**
  - 3-year warranty
  - typical tube lifetime > 5 years
  - still no water cooling required!

What is Special About a Tube Optimized for Crystallography?

- All other microfocus tubes on the market were designed primarily for radiography (medical or NDT)

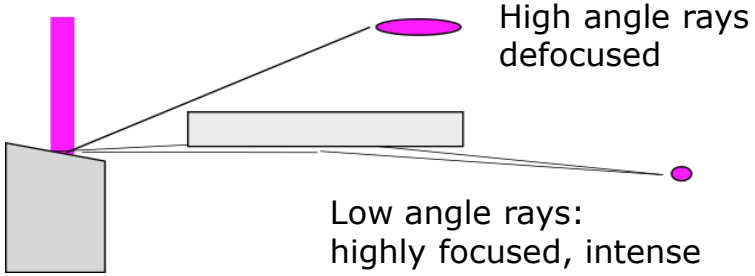
  They therefore feature high take-off angles in order to preserve resolution over a wide field of view

Radiography

- In crystallography we do not need a wide field of view, we only need to produce an intense beam of X-rays

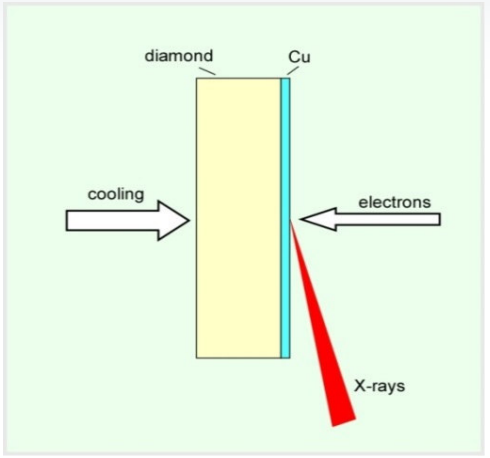
  An elongated electron beam focus on an anode with a lower take-off angle produces higher intensity

Crystallography



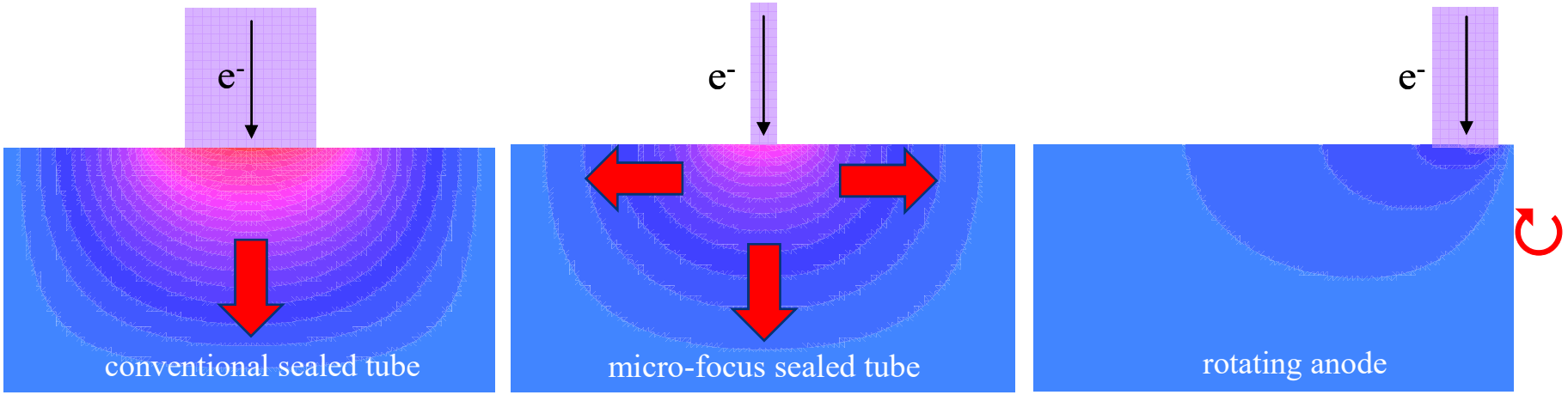
What is the IμS DIAMOND?

- The first microfocus tube that uses an advanced **Diamond Hybrid Anode**
  - Diamond heat sink improves heat dissipation
  - Increased power density on anode
- Coupled to **synchrotron-class** multilayer Montel optics
- Intensity of modern microfocus rotating anodes
  - **Twice the intensity** of competing 50 W HB-class sources
- Same proven mechanical platform and same lifetime as IμS 3.0
- No routine maintenance
- Available for Cu, Mo and Ag radiation



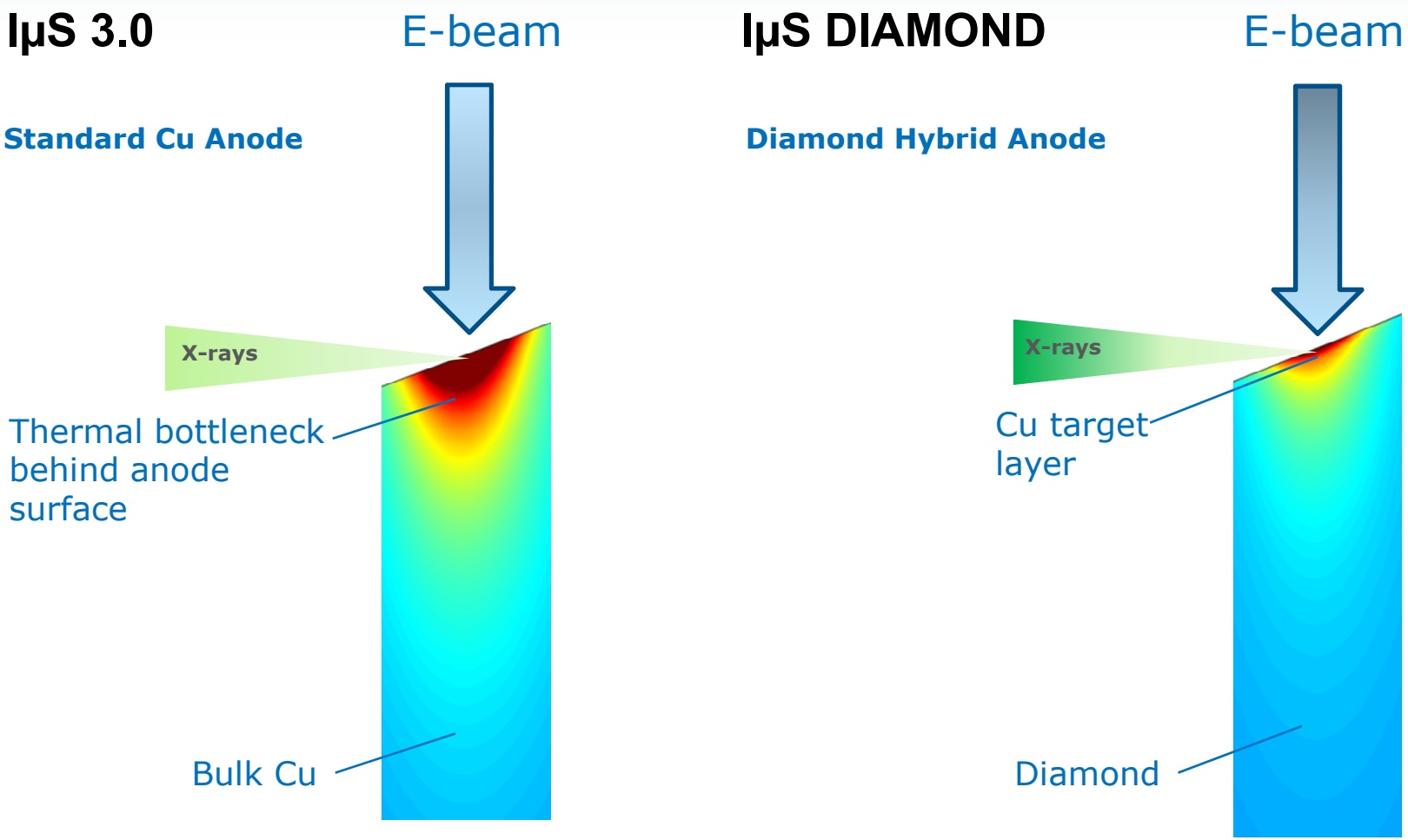
Brightness of Microfocus Sources

- Power Load in All Solid-target X-ray Sources is Limited by Heat Dissipation



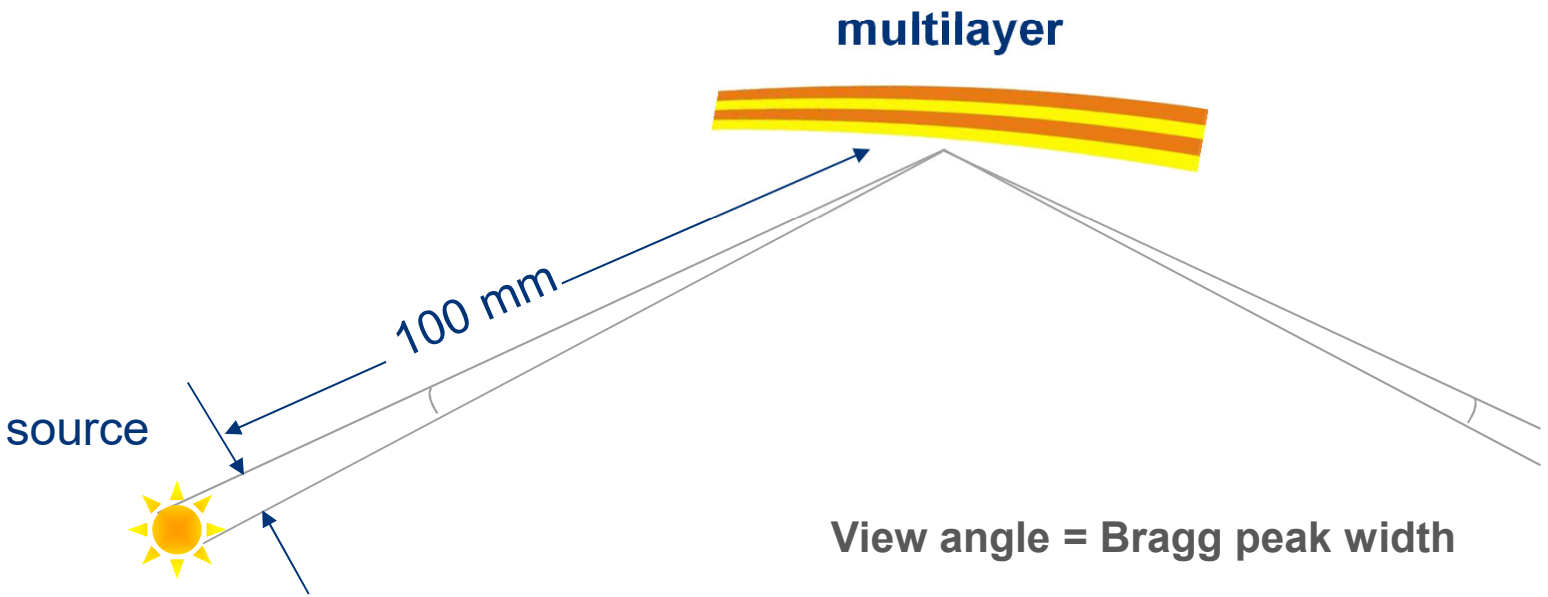
■ Large Spot	■ Small Spot	■ Large or Small Spot
■ Quasi-1D heat flow limits power density	■ 2D heat flow allows more efficient cooling	■ Additional heat spread by rotation
■ ~ 0.5 kW/mm^2	■ ~ 5 kW/mm^2	■ > 15 kW/mm^2
Relative B: 1	**Relative B: ~ 10**	**Relative B: > 10**

What is the IμS DIAMOND?



The Perfect Match

- Multilayer Mirrors and Microfocus Sources are a **Perfect Match** for monochromatic beam with tailored beam cross-section and divergence
- **View angle** of the multilayer coating **fits the source size**



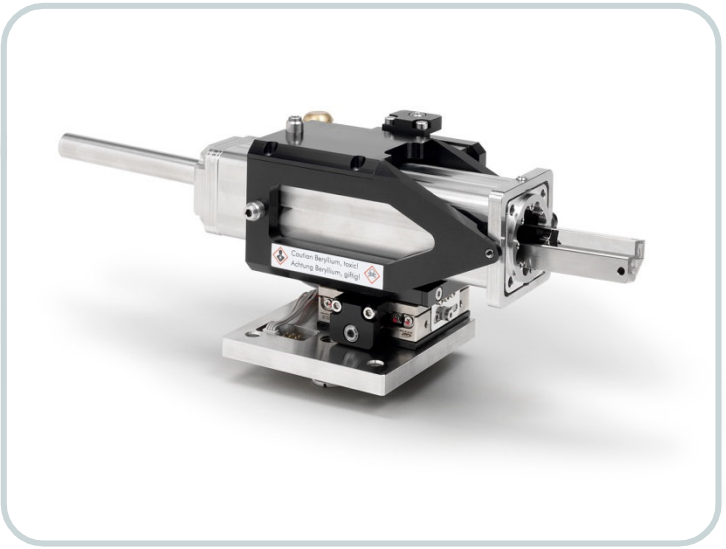
100 mm * 1 mrad = 100 μm

View angle = Bragg peak width

Typical for W/C multilayer:
FWHM = 1 mrad for Cu Kα (= 0.057 °)

HELIOS EF Optics

- HELIOS EF optics is **designed** for the **Cu-IμS 3.0** to deliver a high intensity in a sharply focused beam
- **Large divergence** that matches the typical mosaicity of small and weakly diffracting samples in **chemical crystallography**
- **Twice the intensity** of the HELIOS MX optics

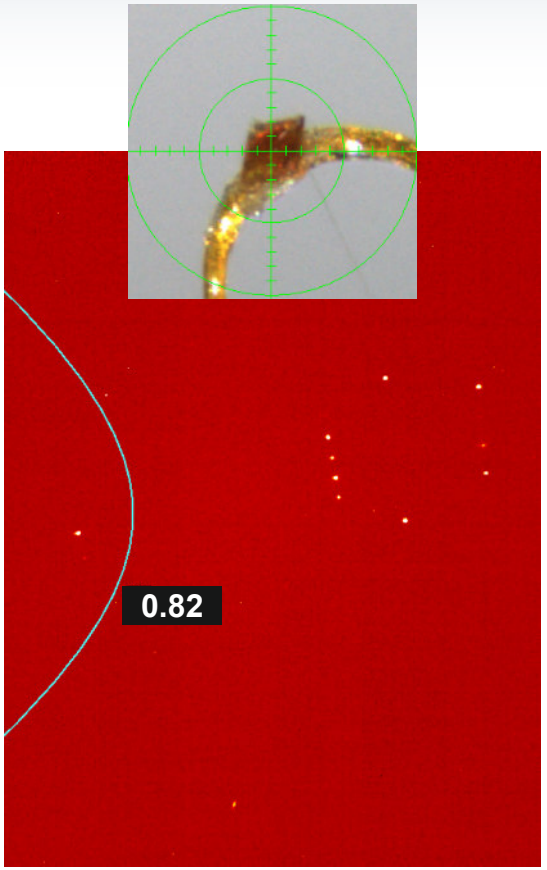


	HELIOS MX	HELIOS EF
Divergence [mrad]	7.5	13.5
FWHM [mm]	0.10	0.10
Intensity [phts/s/mm^2]	> 3.5 10^{10}	> 7.7 10^{10}

HELIOS EF Optics

- Data Comparison for Small Vitamin C Crystal

Size [mm³]	0.04 x 0.10 x 0.10	
Optics	**HELIOS MX**	**HELIOS EF**
Exposure time [s/°]	4 / 8	4 / 8
Ratio of norm. < I >	1	2.14
<I/σ>	27.1 (11.2)	37.0 (25.1)
R(int) [%]	4.91 (14.66)	3.75 (6.82)
*R*1, *wR*2 [%]	3.27; 7.51	2.59; 6.65
Parsons *z*(*v*)	0.16(13)	0.02(8)
d(C-C) [Å]	1.526(5)	1.527(3)



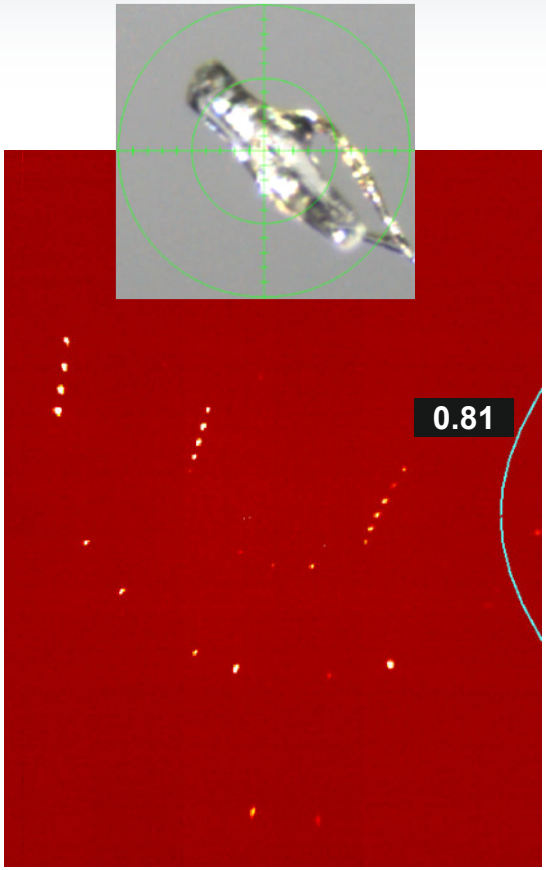
Typical diffraction pattern ($P2_1$, a = 6.3962(2) Å, b = 6.3166(2) Å, c = 17.0992(5) Å, β = 99.347(2)°, Z = 4)

HELIOS EF Optics

- Real Life Example: Larger Crystal of Amide Derivative

Size [mm³]	0.05 x 0.10 x 0.35	
Optics	**HELIOS MX**	**HELIOS EF**
Exposure time [s/°]	6 – 12	6 – 12
Resolution [Å]	0.81 (0.91 – 0.81)	0.81 (0.91 – 0.81)
Multiplicity	9.7 (5.2)	9.6 (7.2)
<I/σ>	54.0 (32.2)	69.9 (55.3)
*R*1, *wR*2 [%]	2.75; 6.84	2.65; 7.05
Parsons *z*(*v*)	0.04(5)	0.01(4)
d(C-C) [Å]	1.525(3) 1.384(3)	1.527(2) 1.385(2)

$C_{14}H_{18}N_2O_2$

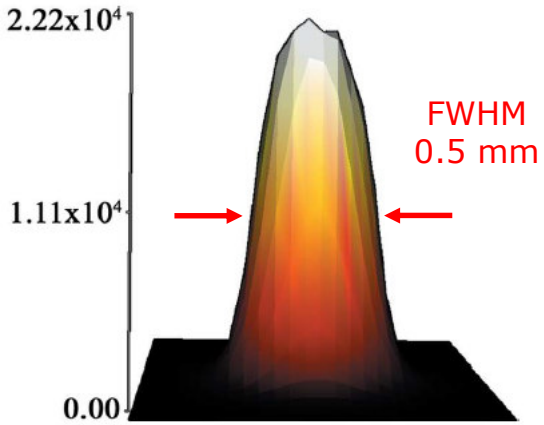


Typical diffraction pattern ($P2_12_12_1$, a = 6.0471(2) Å, b = 9.2264(3) Å, c = 23.0208(8) Å, Z = 4)

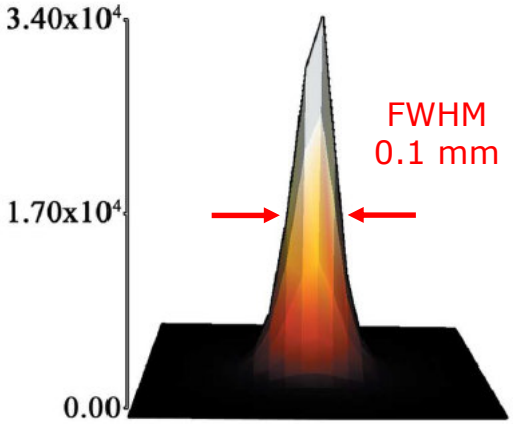
Beam Profile
Top Hat *vs.* Gaussian Beam Profile

Comparison of beam profiles from a focusing multilayer X-ray mirror and from a flat graphite monochromator

Flat Graphite Monochromator



Multilayer Mirror



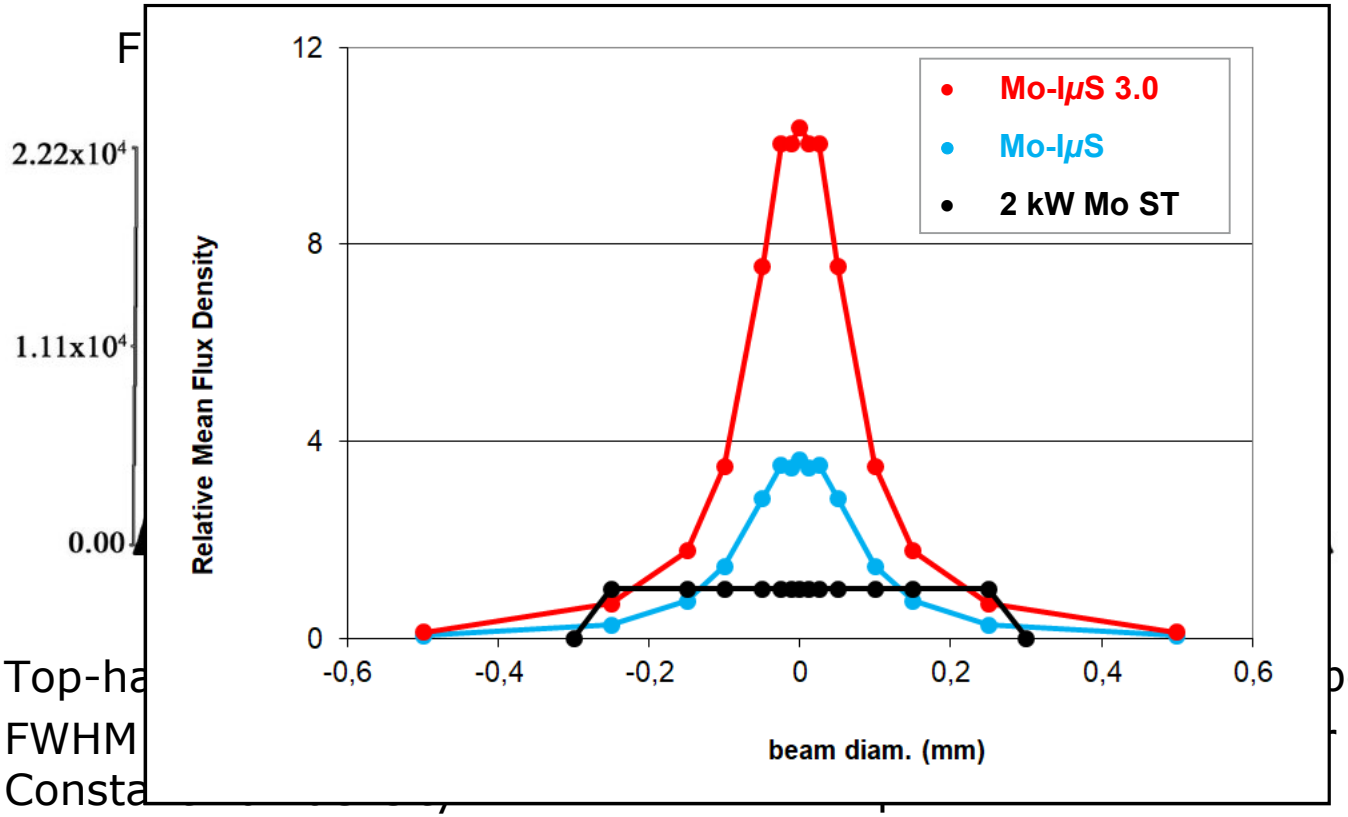
- Top-hat beam profile
- FWHM depends on collimator
- Constant flux density

- Symmetric Gaussian shaped beam
- Flux density increases for smaller sample diameters

Beam Profile

Top Hat *vs.* Gaussian Beam Profile

Comparison of beam profiles from a focusing multilayer X-ray mirror and from a flat graphite monochromator



F

2.22x10⁴

1.11x10⁴

0.00

Top-ha ped beam

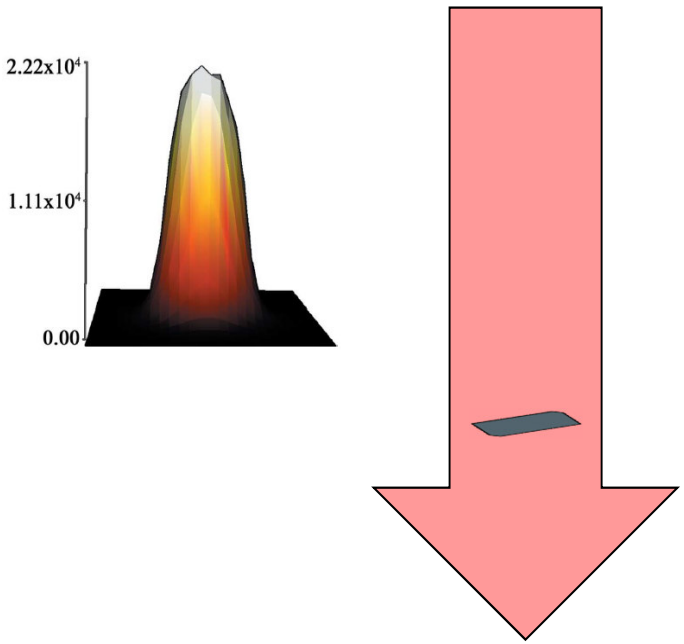
FWHM smaller

Consta

Beam Profile

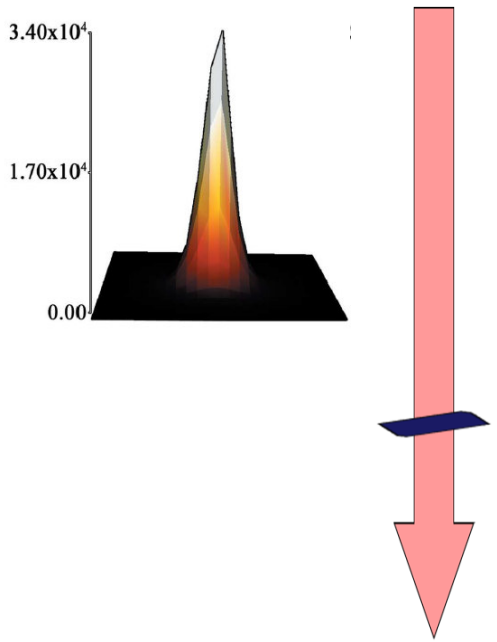
Top Hat *vs.* Gaussian Beam Profile

Top hat beam



Crystal bathed in beam of uniform intensity. Effective diffracting volume does not change on rotating.

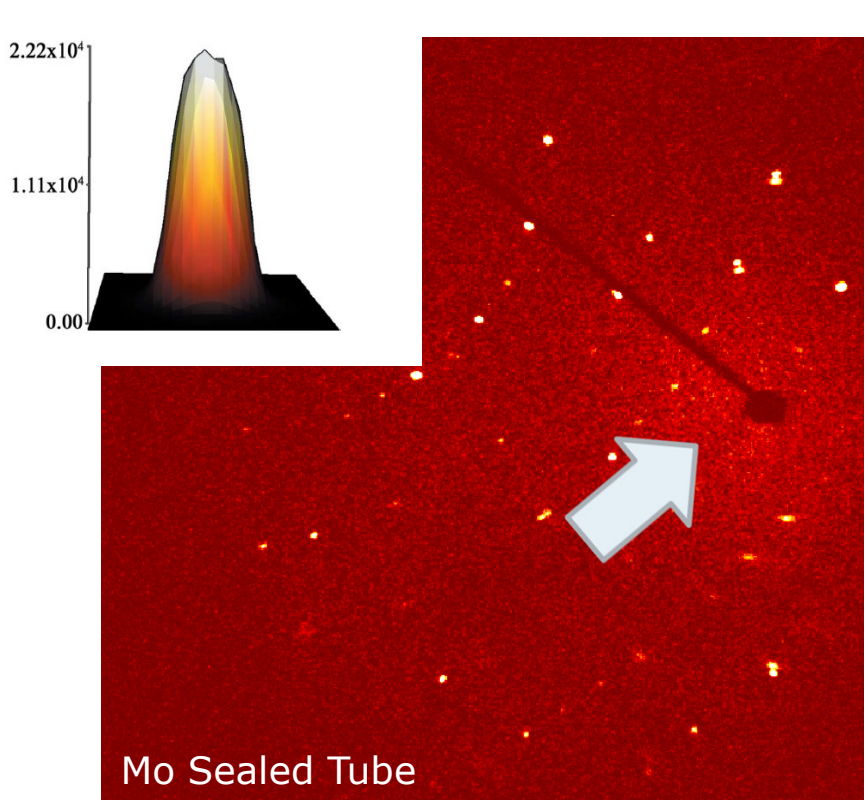
Focusing optics

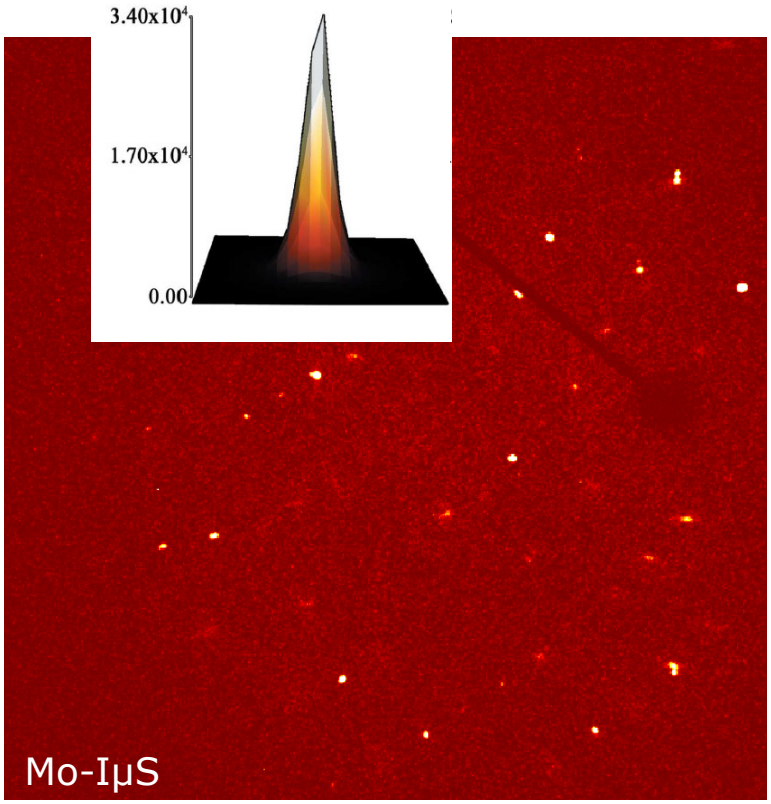


Effective diffracting volume might vary with crystal orientation. Scaling will correct for this. The standard face-indexed correction is inappropriate.

Beam Profile
Top Hat *vs.* Gaussian Beam Profile

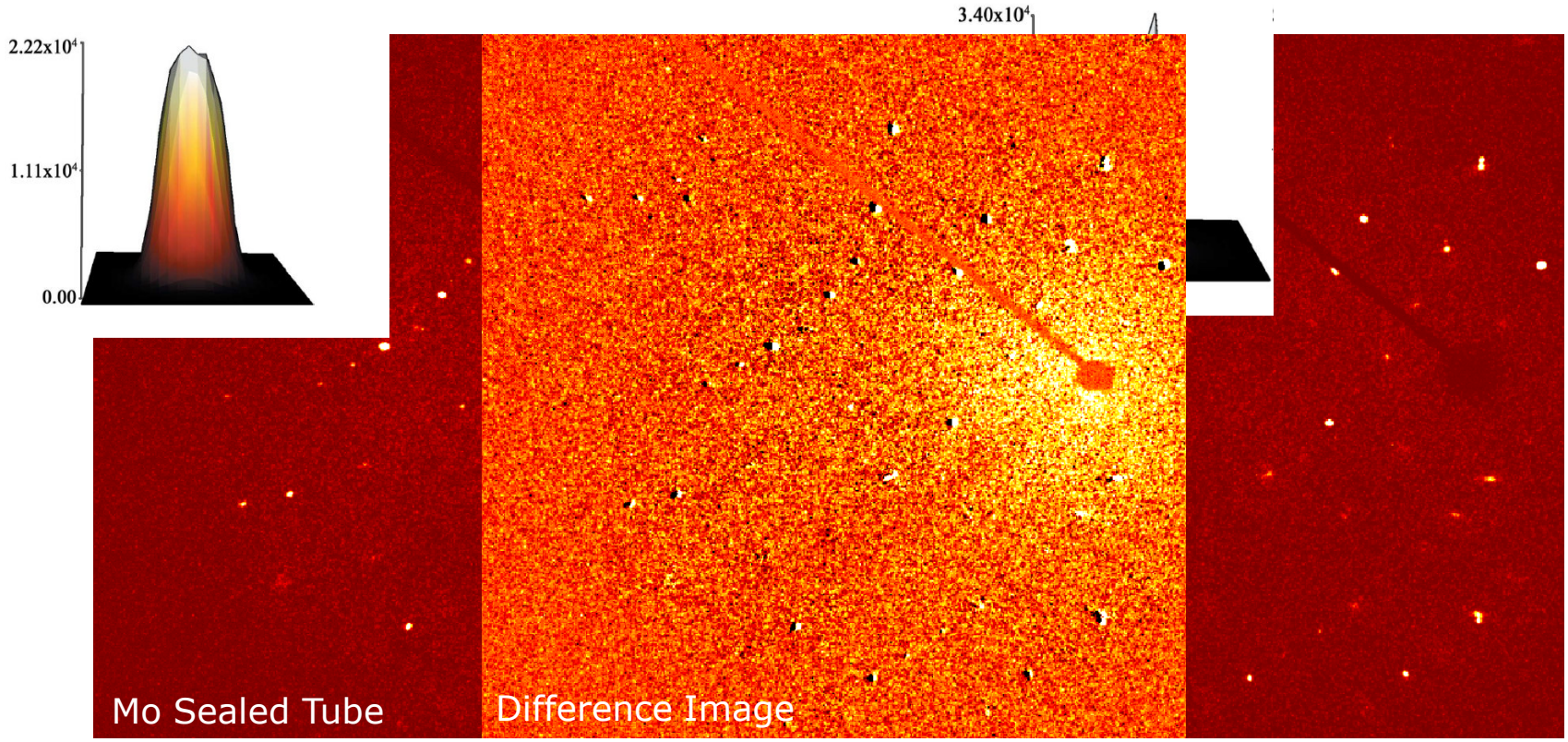
- Top hat beam has more background noise due to more air scattering





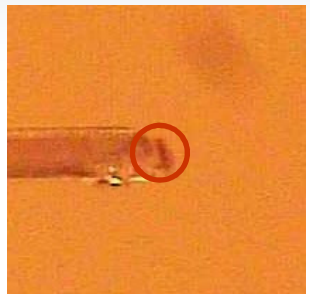
Beam Profile
Top Hat *vs.* Gaussian Beam Profile

- Top hat beam has more background noise due to more air scattering

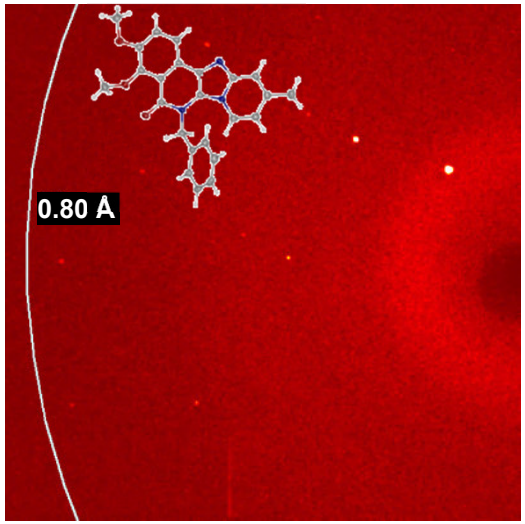


Beam Profile
Top Hat *vs.* Gaussian Beam Profile

- Data comparison from Small Crystal of Organic Compound



Size [mm³]	0.05 x 0.05 x 0.10	
Source	**Mo ST**	**Mo-IμS**
Power [W]	2000	30
Exposure time	90 s/0.3°	30 s/0.3°
Resolution [Å]	0.75 (0.85 – 0.75)	0.75 (0.85 – 0.75)
Ratio norm. < I >	1	7.3
<I/σ>	10.7 (2.1)	15.3 (3.5)
*R*1, *wR*2 [%]	5.55, 10.44	3.88; 9.16
Distance N – C [Å]	1.325(4)	1.325(3)



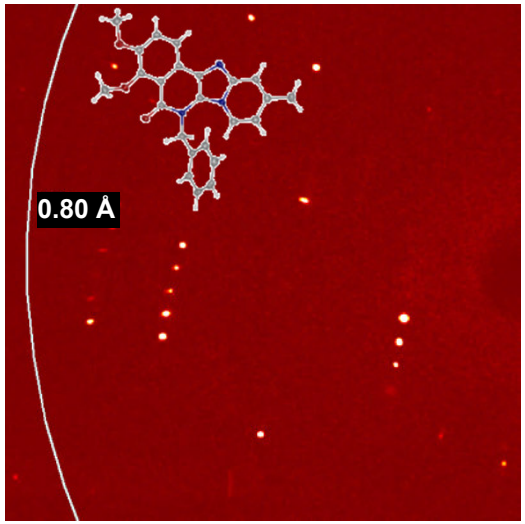
Typical diffraction pattern ($P2_1$, a = 8.3628(6) Å, b = 7.0469(5) Å, c = 15.9737(11) Å, β = 92.210(1)° , Z = 2)

$C_{24}H_{21}N_3O_3$

Beam Profile
Top Hat *vs.* Gaussian Beam Profile

- Data comparison from Large Crystal of Organic Compound

Size [mm³]	0.15 x 0.25 x 0.30	
Source	**Mo ST**	**Mo-IμS**
Power [W]	2000	30
Exposure time	15 s/0.3°	10 s/0.3°
Resolution [Å]	0.75 (0.85 – 0.75)	0.75 (0.85 – 0.75)
Ratio Norm. < I >	1	3.7
<I/σ>	30.4 (13.6)	37.6 (21.1)
*R*1, *wR*2 [%]	3.17, 8.16	3.08; 8.18
Distance N – C [Å]	1.324(2)	1.323(2)



Typical diffraction pattern ($P2_1$,
a = 8.3628(6) Å, b = 7.0469(5) Å,
c = 15.9737(11) Å, β = 92.210(1)° , Z = 2)

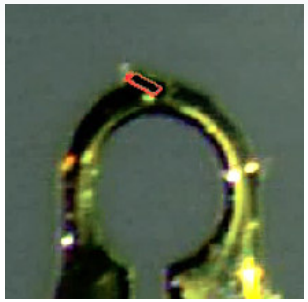
$C_{24}H_{21}N_3O_3$

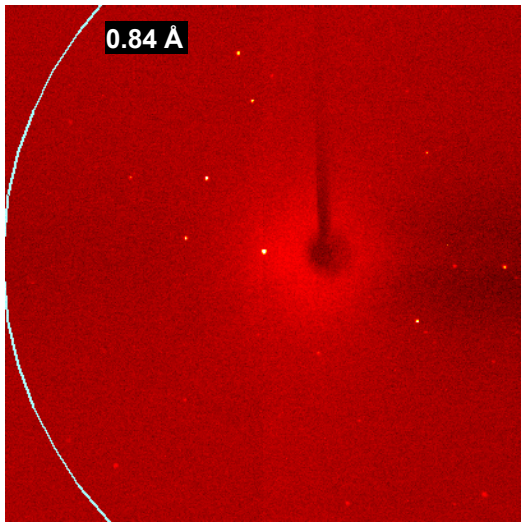
How small?

- Data from a Tiny Crystal of MOF Compound

Size [mm³]	0.002 x 0.010 x 0.018
Source	**Mo-IμS HB**
Power [W]	50
Exposure time	240 s/°, 22 h
Resolution [Å]	0.83 (0.93 – 0.83)
Multiplicity	3.9 (2.2)
<I/σ>	15.1 (4.2)
*R*1, *wR*2 [%]	4.27; 8.83

$C_{18}H_{28}LaO_{23}P_6$





Typical diffraction pattern ($P2_1/m$, a = 8.9215(10) Å, b = 21.209(2) Å, c = 9.1052(10) Å, β = 107.923(3)° , Z = 2)

How small?

- Data from a Tir

Size [mm³]
Source
Power [W]
Exposure time
Resolution [Å]
Multiplicity
<I/σ>
*R*1, *wR*2 [%]

$C_{18}H_{28}LaO_{23}P_6$

Application Note SC-XRD 502

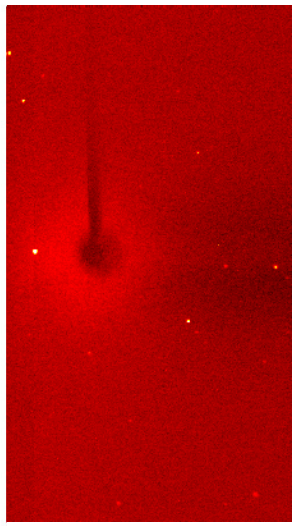
Structural Determination of a Two Micron-sized MOF Crystal

Metal-Organic Frameworks (MOFs) are structures of great industrial and academic interest as they can be at the genesis of a series of new materials with highly attractive properties. Nowadays MOFs can be prepared very fast, under mild conditions in high yields while still combining several functions, taking advantage of the metallic centers, the organic ligands and the framework itself. Some MOFs can even outperform known materials in common industrial applications. $[La(H_4bmt)(H_5bmt)(H_2O)_2]\cdot 3H_2O$ (**1**) has outstanding catalytic activity for the methanolysis of styrene oxide in comparison to that of related MOF-type heterogeneous catalysts. In addition, the inclusion of small amounts of other lanthanides can produce highly photoluminescent materials. [1] **1** can be quickly prepared in 5 minutes at 330 K using microwave-assisted synthesis from the tridentate (benzene-1,3,5-triyltris(methylene))triphosphonic acid (H_6bmt) and $LaCl_3\cdot 7H_2O$. Nevertheless, to fully understand

microscope. The D8 VENTURE consists of a KAPPA goniometer and PHOTON 100 CMOS APS detector, equipped with a IμS microfocus X-ray source providing Mo-Kα radiation (λ = 0.71073 Å). During the entire experiment the sample was kept at 100 K using a KRYOFLEX II low temperature device, controlled with the APEX2 software package.

The experiment

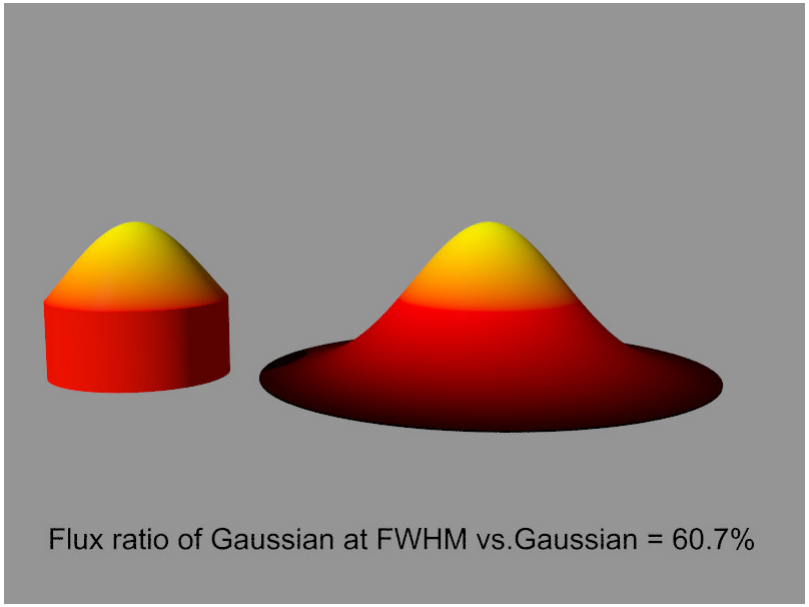
Based on a number of initial scans the unit cell determination routine of the software suite indicated a monoclinic unit cell. From the same scans 0.5 degree frames were suggested by the data collection strategy optimizer and the refined data collection strategy consisted of only three scans. Due to the weak diffraction of this very tiny crystal, an appropriate frame exposure time of 120 s was selected. A total of 645 frames were collected within just 21.5 hours.

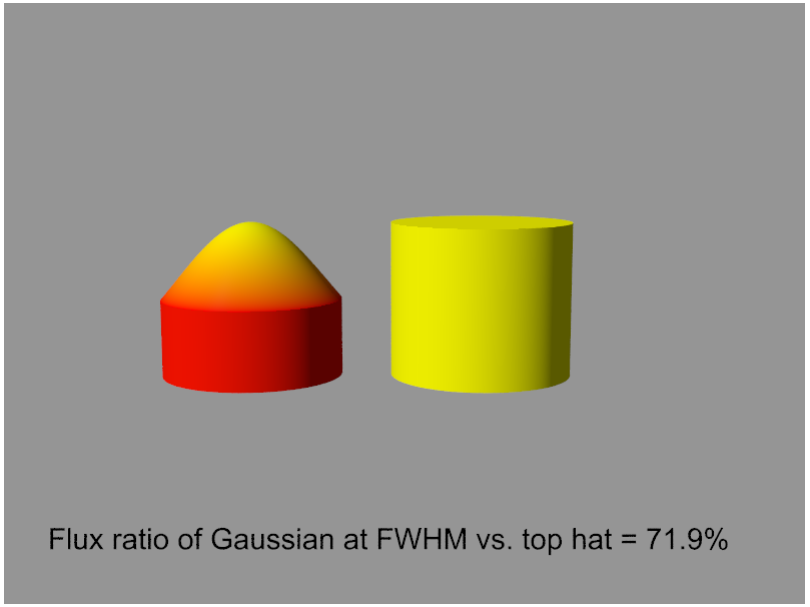


n pattern ($P2_1/m$,
λ, b = 21.209(2) Å,
λ, β = 107.923(3)° , Z = 2)

Microfocus Source Gaussian Beam Profile

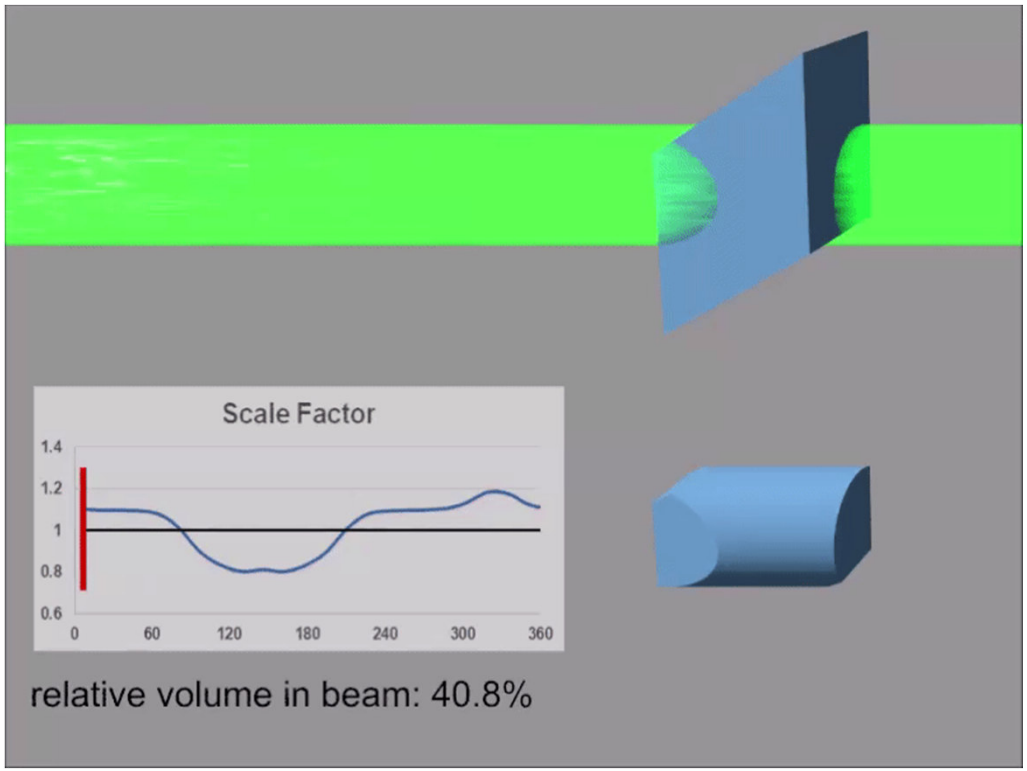
- Classic normal fine focus sources with graphite monochromators exhibit a top hat shaped beam profile
  - Flat graphite crystal
  - TRIUMPH curved graphite crystal





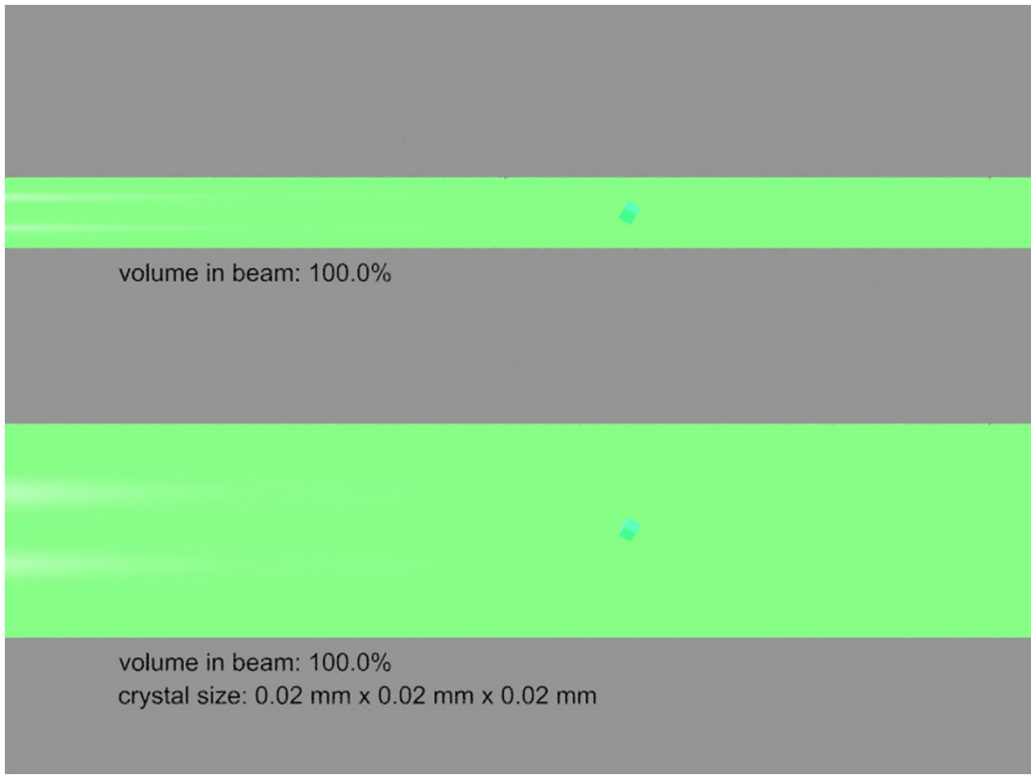
Large Crystal in Large Beam

- Rhombohedral crystal 0.2 ×0.2 ×0.2 mm^3
- Average crystal volume in beam is about 45 %
- Incident beam scale factors from 0.8 to 1.2



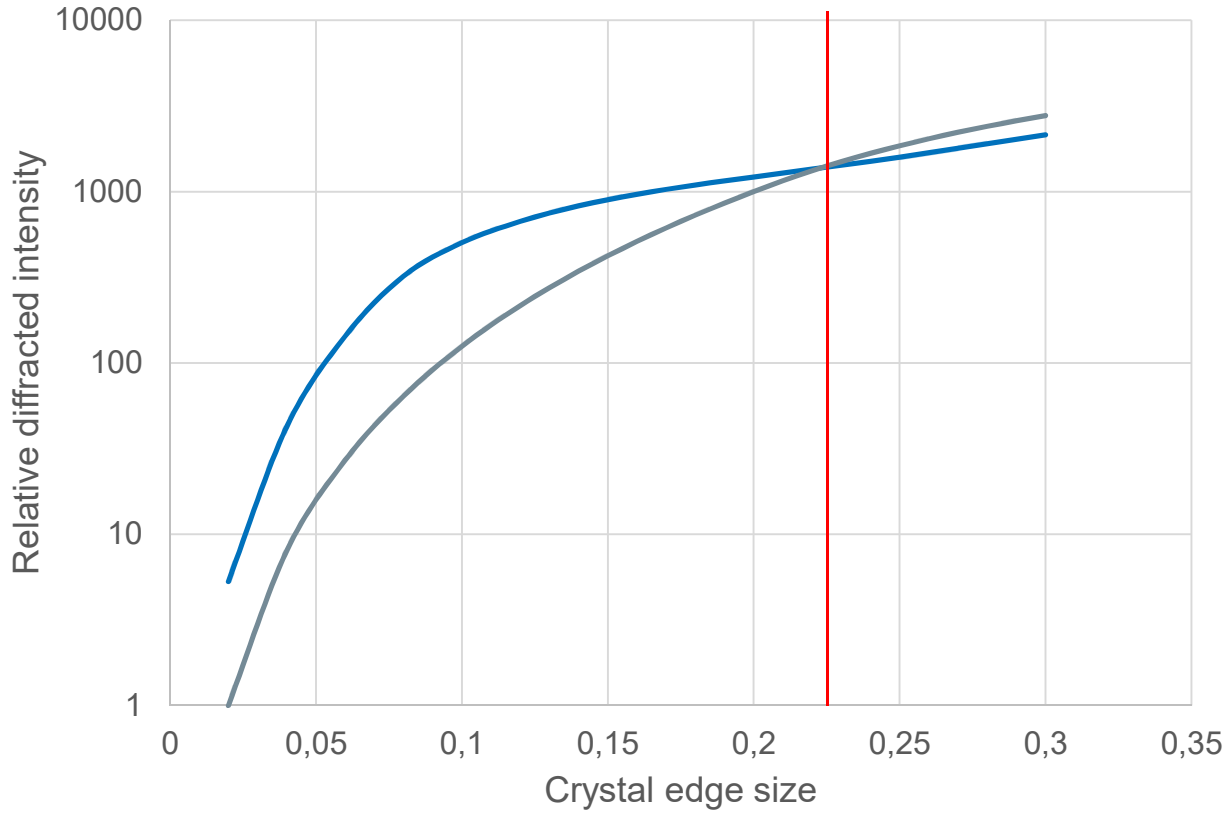
Small Beam *vs.* Large Beam

- Relative crystal volume in a small beam (0.1 mm) and a large beam (0.3 mm)
- The crystal is a cube in this simulation



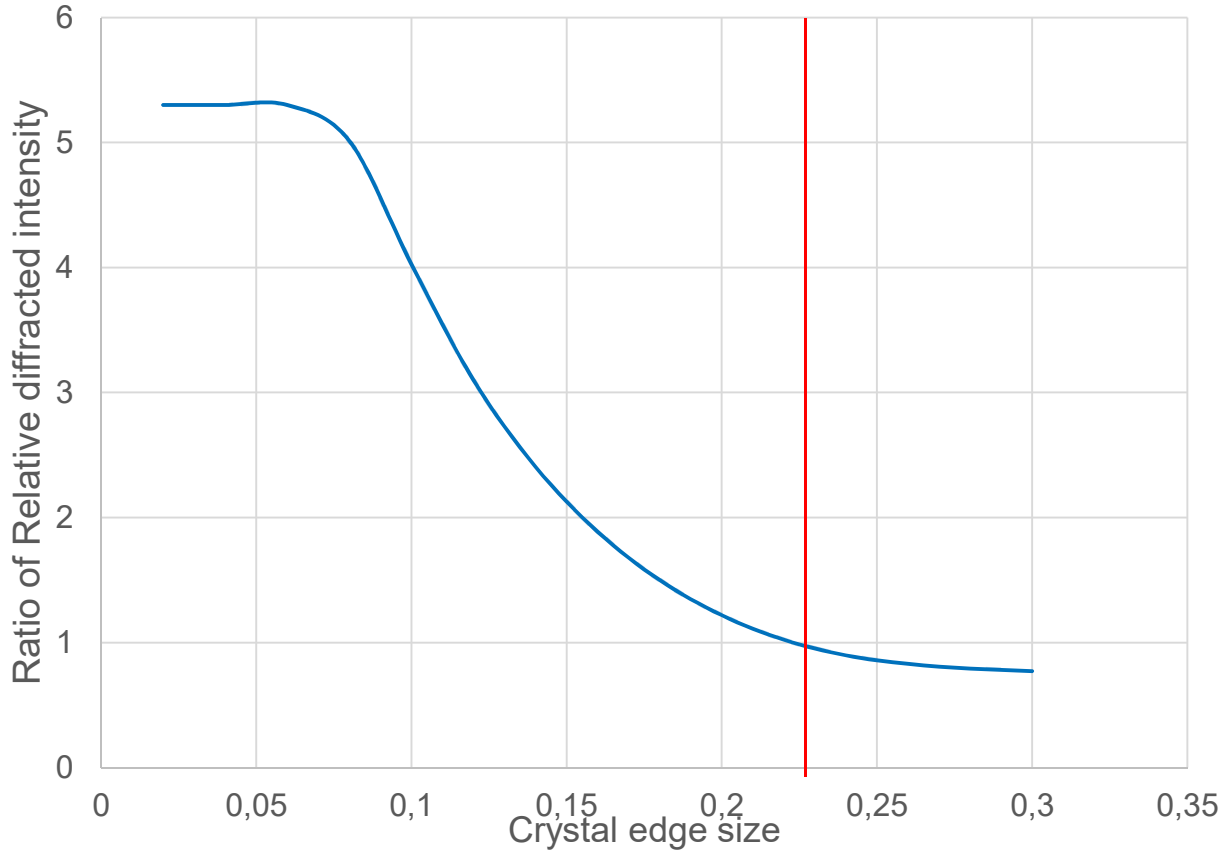
Small Beam *vs.* Large Beam Mo

- The small beam has a 5.3 times higher intensity (Mo)
- Relative diffracted intensity of a small beam (blue) vs large beam (gray)



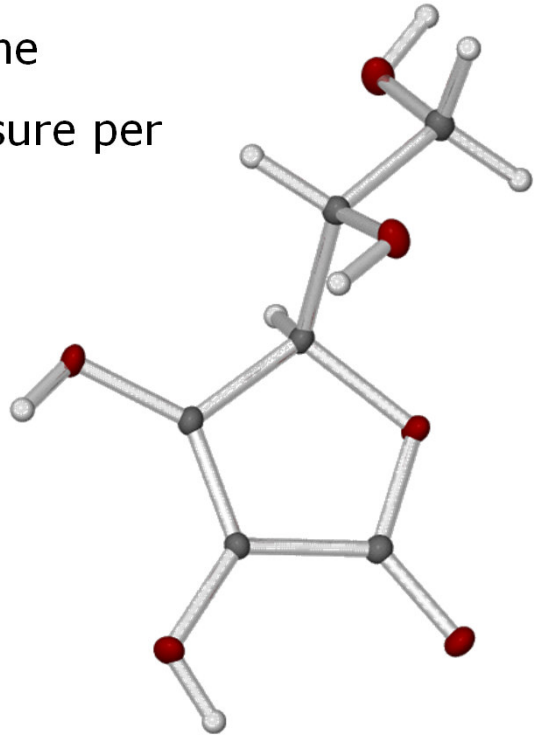
Small Beam *vs.* Large Beam Mo

- Ratio of relative diffracted intensity from the small and the large beam



Small Crystal *vs.* Large Crystal

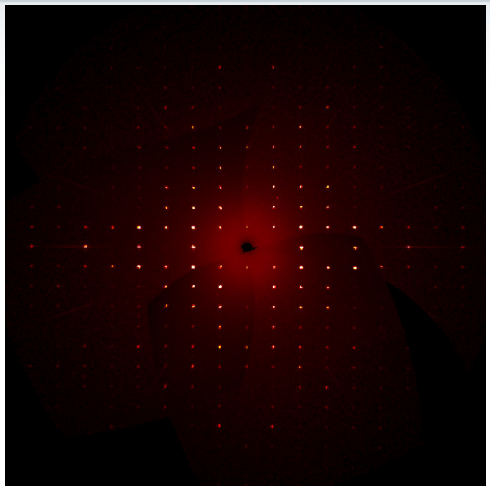
- 4 data sets were collected on Ascorbic Acid crystals
- Data were collected on a small crystal and a large crystal each with a 180 μm and a 360 μm beam
- The large crystal had a 55 times larger volume
- Data were collected to 0.65 Å, with 10s exposure per 0.36° frame

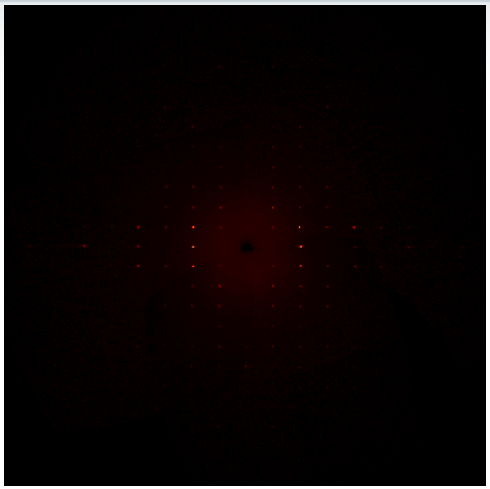


- 0.025 x 0.100 x 0.110 mm^3
- 0.12 x 0.32 x 0.40 mm^3

Small Crystal *vs.* Large Crystal

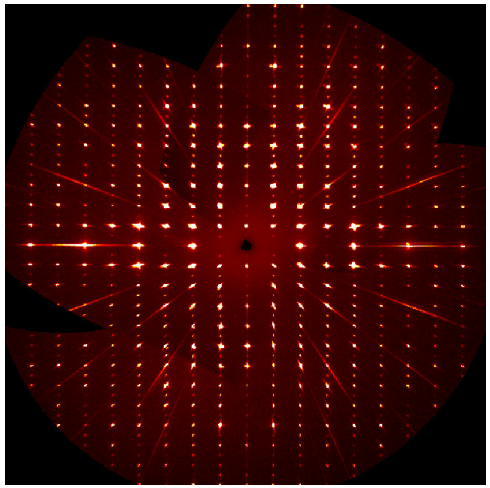
- Small crystal small beam
- R1 = 4.19% for 3915 reflections

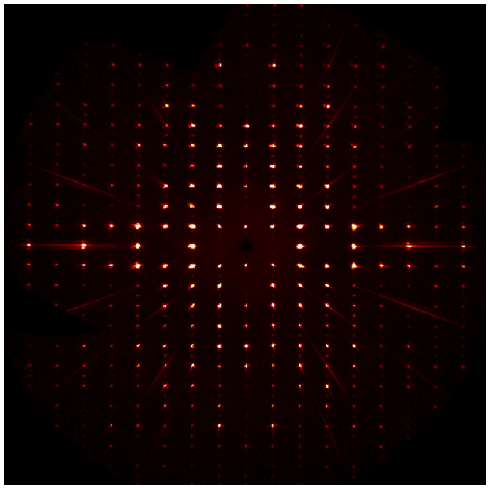




- Small crystal large beam
- R1 = 7.87% for 2061 reflections

- Large crystal small beam
- R1 = 3.08% for 4588 reflections





- Large crystal large beam
- R1 = 3.47% for 4462 reflections

Microfocus Source Examples

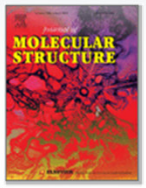
App Lab Madison – Christian Jelsch, Nancy

Aceclofenac Charge Density Study

ELSEVIER

Journal of Molecular Structure

Volume 1205, 5 April 2020, 127600



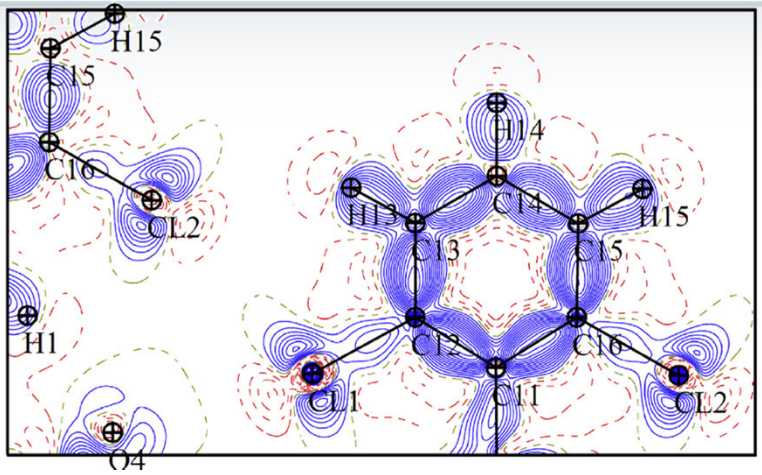
Aceclofenac and interactions analysis in the crystal and COX protein active site

Christian Jelsch [a], Rajendran Niranjana Devi [b, c], Bruce C. Noll [d], Benoît Guillot [a], Israel Samuel [b], Emmanuel Aubert [a]

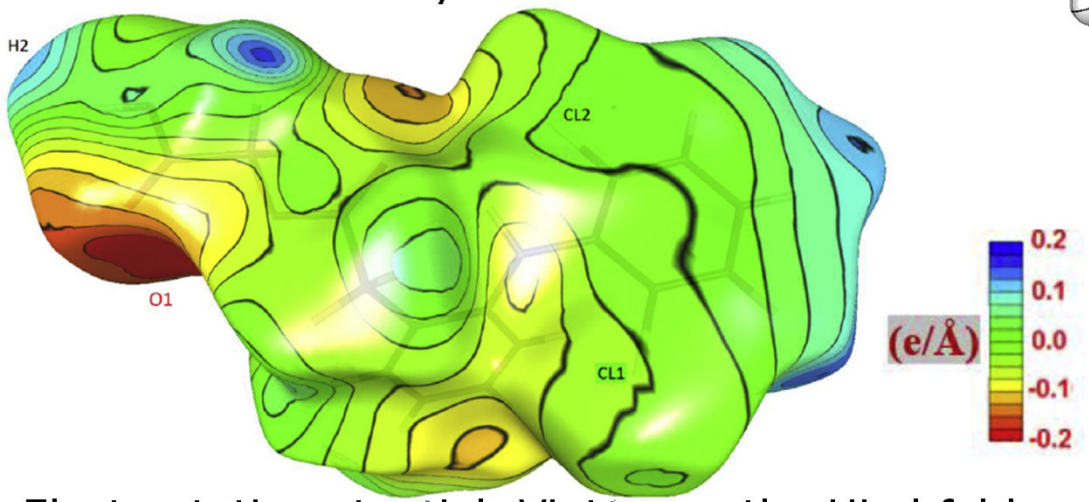
- Data from the D8 QUEST with TRIUMPH were merged with data from a D8 VENTURE for a multi-site charge density experiment
- Both crystals were larger than the beam
  - D8 VENTURE 0.108 × 0.198 × 0.208 mm^3
  - D8 QUEST 0.148 × 0.360 × 0.379 mm^3

App Lab Madison – Christian Jelsch, Nancy

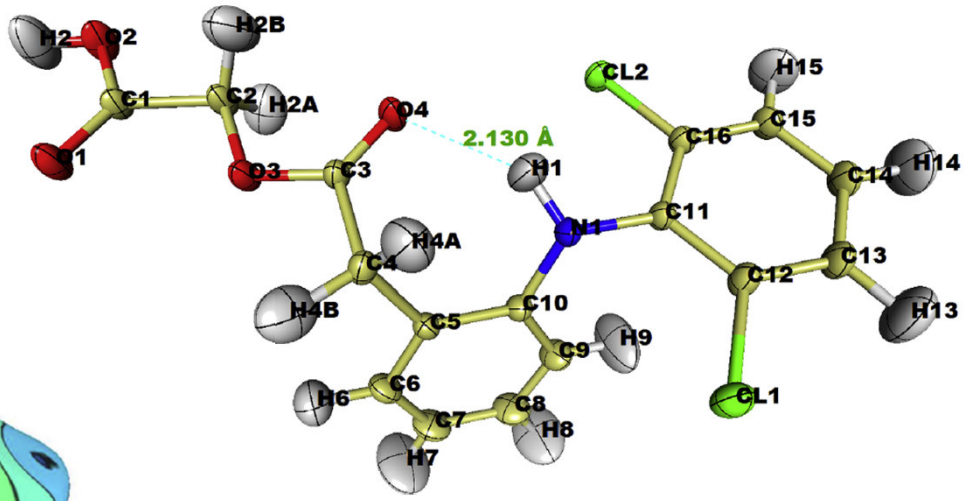
Aceclofenac Charge Density Study



Deformation density



Electrostatic potential Vint(r) on the Hirshfeld surface



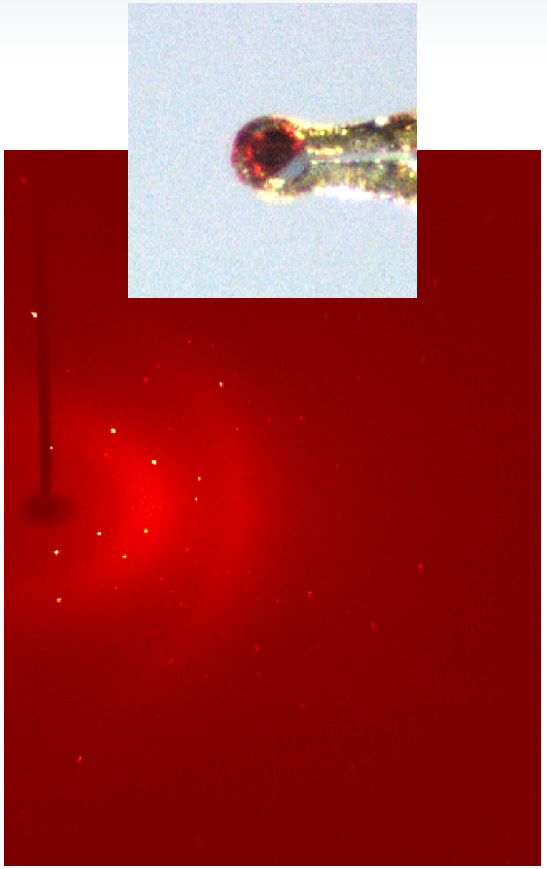
Structure of Aceclofenac

Saarland University – Scheschkewitz Lab

All-Silicon Cyclobutan-1,3-diyl as Reaction Intermediate

Data from a Small Crystal of a Stabilized Tetrasilacyclobutan-1,3-diyl

Size [mm³]	0.02 x 0.08 x 0.08
Source	**Cu-IμS 3.0 MX**
Exposure time	20 s/0.5°
Resolution [Å]	0.80 (0.90 – 0.80)
Multiplicity	6.2 (4.5)
<I/σ>	9.5 (4.7)
*R*1, *wR*2 [%]	5.74, 13.39



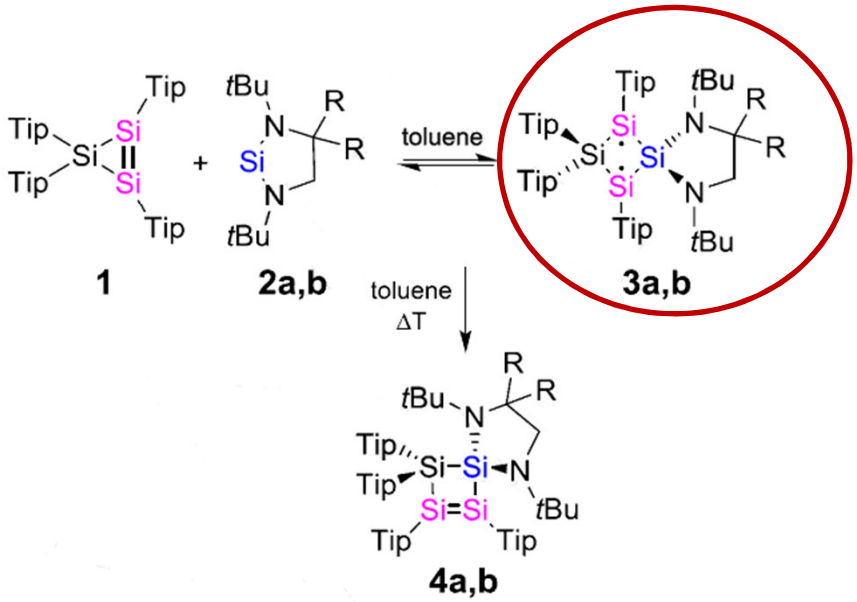
Typical diffraction pattern ($P2_1/n$, a = 14.7011(4) Å, b = 22.3445(7) Å, c = 21.1554(7) Å, β = 96.007(2)° , Z = 4)

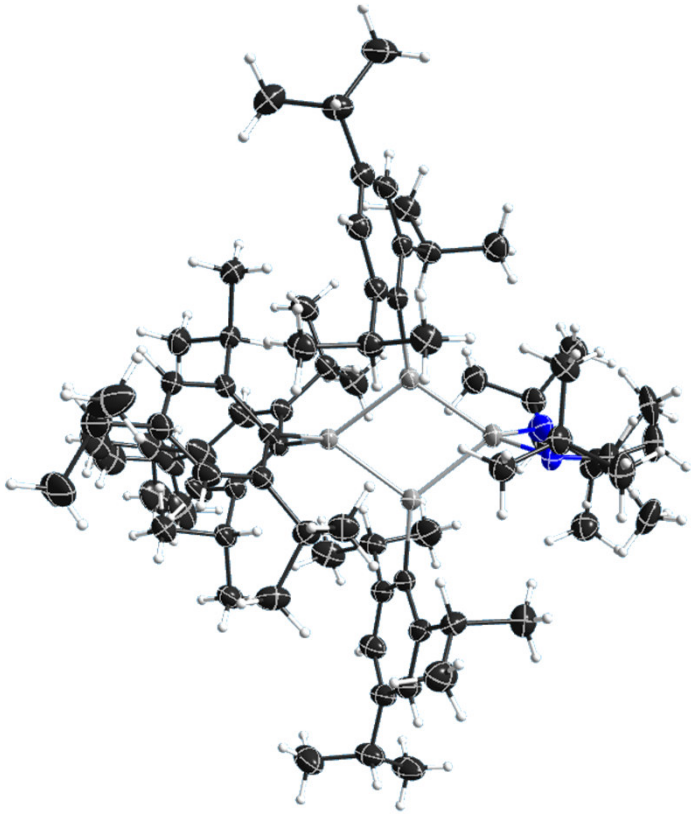
$C_{72}H_{118}N_2Si_4$

Saarland University – Scheschkewitz Lab

All-Silicon 1,3-Cyclobutandiyl as Reaction Intermediate

Reaction Scheme for the Formation of the Stabilized Tetrasilacyclobutan-1,3-diyl





Newcastle University – Michael Probert's Lab
Crystallising the "uncrystallisable"

- Encapsulated Nanodroplet Crystallization as a new robot-assisted high-throughput nanoscale crystallization method:
  - Crystals from challenging "uncrystallisable" small molecule compounds
  - Powerful tool for efficient polymorph screening

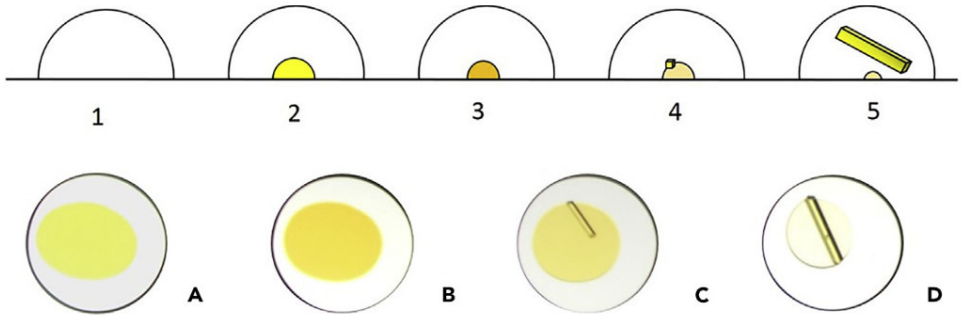
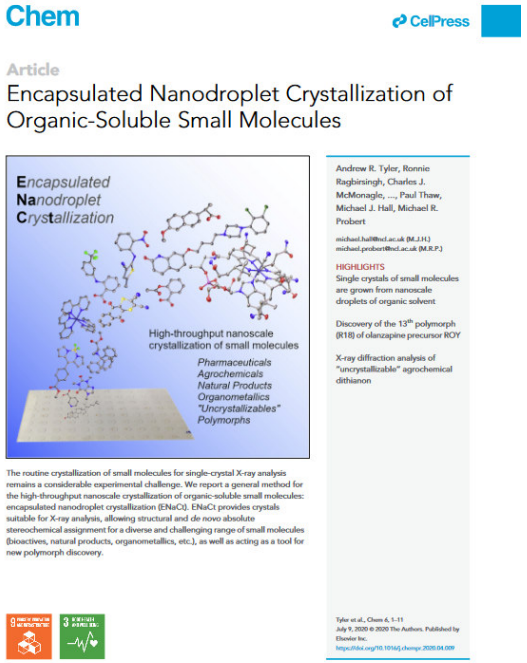


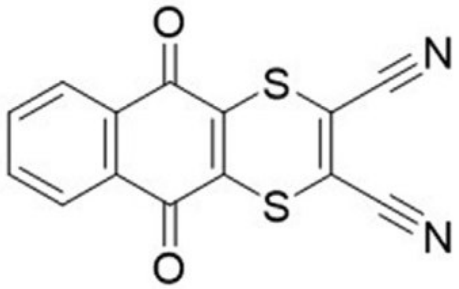
Figure 1. Cross-Section Schematic of an ENaCt Experiment (Top) and ENaCt Experiment with 200 nL Mineral Oil and 50 mg/mL ROY in DMSO (Bottom).
(1) Viscous inert oil dispensed onto a well of a 96-well glass plate, (2) solution of analyte in organic solvent injected into an oil droplet, (3) evaporative solvent loss to supersaturation, (4) nucleation, and (5) crystal growth. (A) solution of solvated analyte under oil, (B) evaporative solvent loss to supersaturation, (C) onset of crystal growth, and (D) complete crystallization.

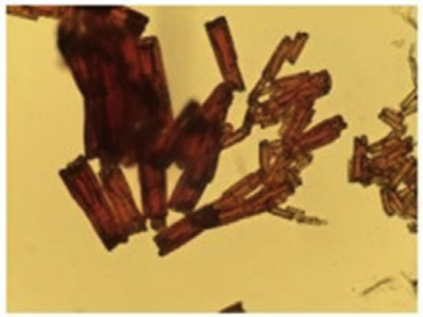

Chem
CellPress
Article
Encapsulated Nanodroplet Crystallization of Organic-Soluble Small Molecules
Encapsulated Nanodroplet Crystallization
High-throughput nanoscale crystallization of small molecules
Pharmaceuticals
Agrochemicals
Natural Products
Organometallics
"Uncrystallizables"
Polymorphs
Andrew R. Tyler, Ronnie Ragbirsingh, Charles J. McMonagle, ..., Paul Thaw, Michael J. Hall, Michael R. Probert
HIGHLIGHTS
Single crystals of small molecules are grown from nanoscale droplets of organic solvent
Discovery of the 13th polymorph (R18) of olanzapine precursor ROY
X-ray diffraction analysis of "uncrystallizable" agrochemical dithianon

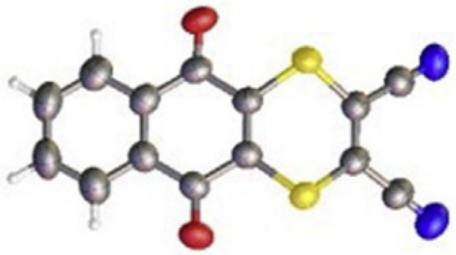
Newcastle University – Michael Probert's Lab

Crystallising the "uncrystallisable"

- First single crystal structure of an "uncrystallisable" molecule: Case study on the fungicide Dithianon
  - Several batches showed crystals formed by Encapsulated Nanodroplet Crystallization
  - Data collected with D8 VENTURE with Cu-IμS 3.0 (0.05 x 0.11 x 0.21 mm^3)







Dithianon (**7**): R_1 = 0.048, $_wR_2$ = 0.1262, Residual e^- density ($e^-Å^3$) min = -0.2 and max = 0.4

200 nL PDMSO, 50 nL of 12.5 mg/mL of dithianon in DMF, 25 nL 2-methyl-2,4-pentanediol.

MIT – Peter Mueller's Lab (IμS 3.0 MX)

Nucleoside Polyphosphates as RNase inhibitors

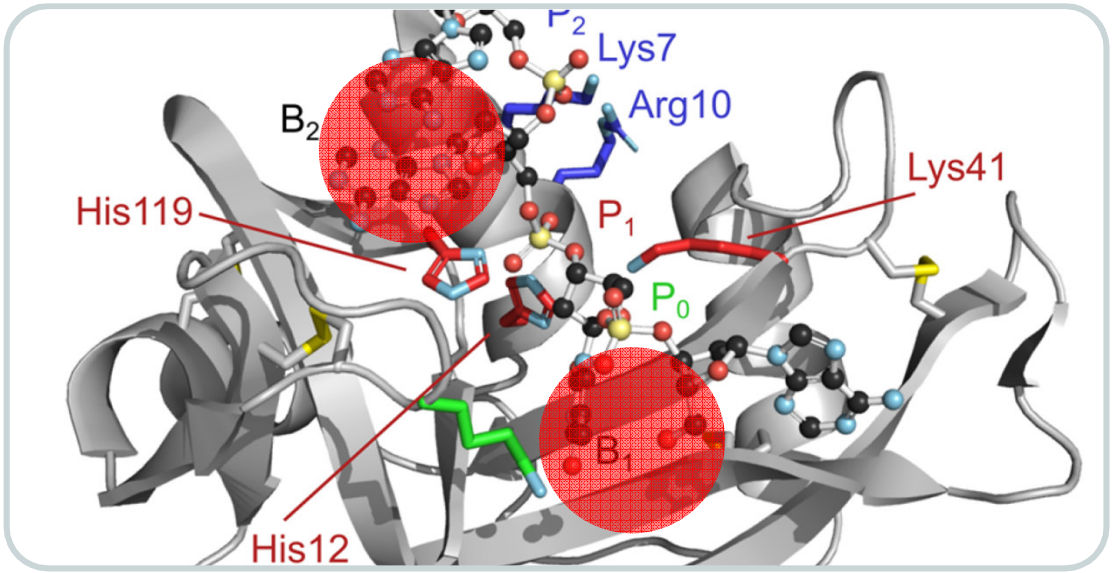
JACS
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
Communication
Cite This: J. Am. Chem. Soc. 2019, 141, 18400−18404
pubs.acs.org/JACS

Nucleoside Tetra- and Pentaphosphates Prepared Using a Tetraphosphorylation Reagent Are Potent Inhibitors of Ribonuclease A

Scott M. Shepard,[†] Ian W. Windsor,[†] Ronald T. Raines,[*] and Christopher C. Cummins[*]

Department of Chemistry, Massachusetts Institute of Technology, Cambridge Massachusetts 02139, United States

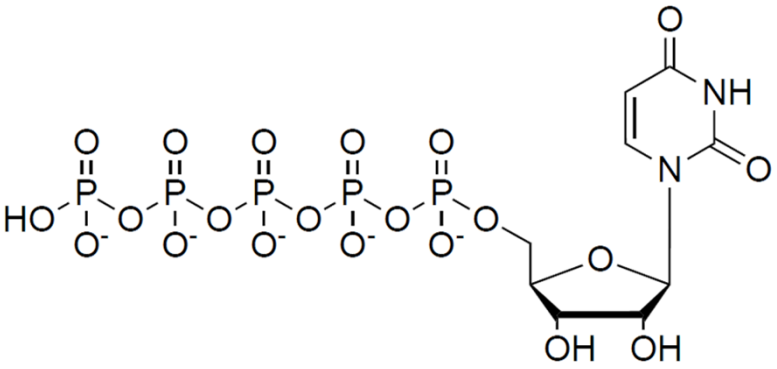
- RNase active sites B1 and B2 bind Uridine and Adenosine Nucleoside Polyphosphates

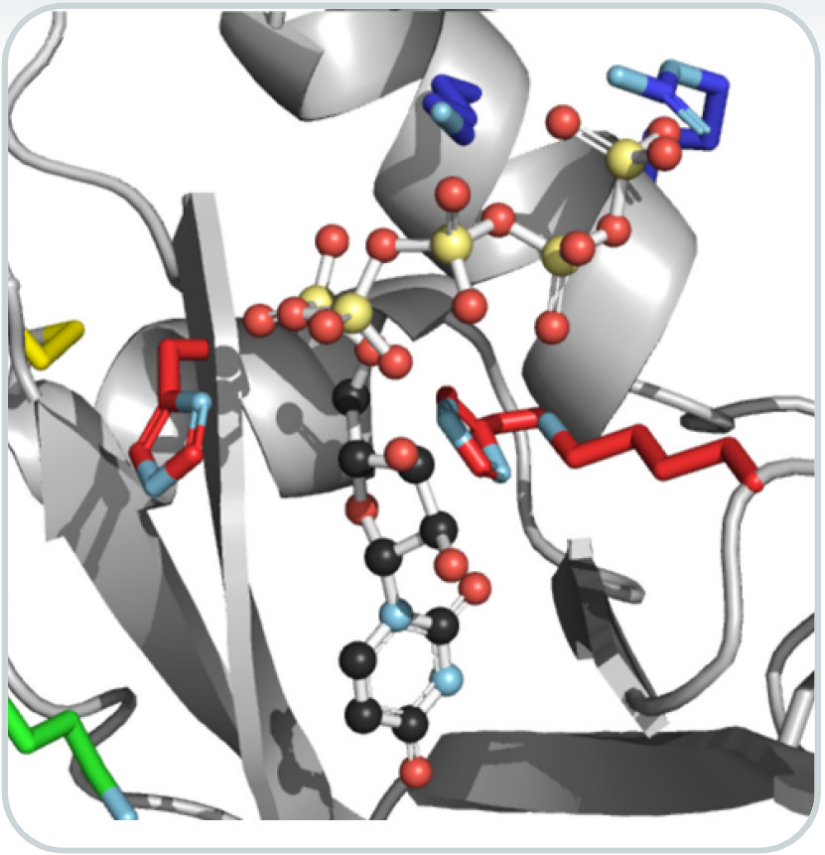


MIT – Peter Mueller's Lab (IμS 3.0 MX)

Nucleoside Polyphosphates as RNase inhibitors

- RNase A ligand structures were solved by molecular replacement using the Phaser program as implemented in PHENIX.
- Refinement was performed with phenix.refine, and model building was conducted with COOT.
- Restraints for inhibitors were prepared with eLBOW in PHENIX and placed with COOT



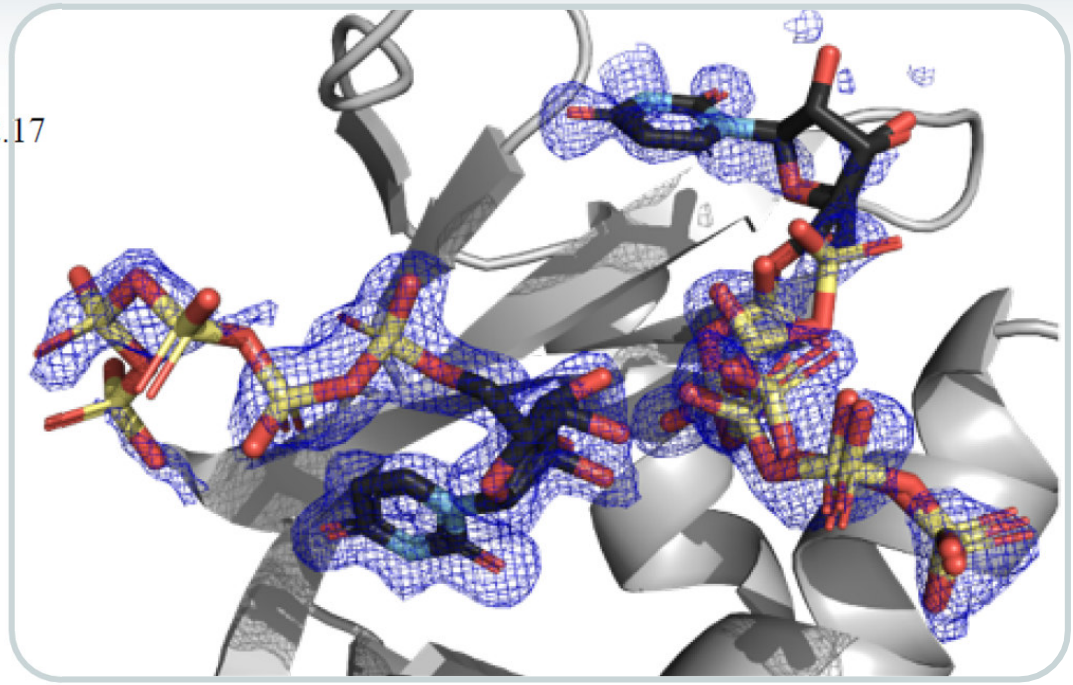


p_5U only binds in the B1 site and alternatively targets the P_1 and P_2 subsites

MIT – Peter Mueller's Lab (IμS 3.0 MX)

Nucleoside Polyphosphates as RNase inhibitors

Ligand (PDB ID)	**p$_5$U** (6pvx)
Data collection	
Space group	*C*121
a, *b*, *c* (Å)	100.54, 32.64, 72.17
α, *β*, *γ* (°)	90, 90.4, 90
Wavelength (Å)	1.54178
Resolution (Å)	29.42–1.55 (1.61–1.55)
Total Reflections	413831 (21558)
Unique Reflections	34001 (2948)
Multiplicity	12.1 (7.3)
Completeness (%)	98.7 (87.1)
Mean $I/\sigma(I)$	25.4 (3.0)
Wilson *B* factor (Å^2)	14.68
R_{merge}	0.060 (0.552)
R_{meas}	0.062 (0.594)
$R_{p.i.m.}$	0.017 (0.215)
$CC_{1/2}$	1.000 (0.823)
Refinement	
Reflections	
Work set	33999 (2948)
Test set	1983 (176)
R_{work}	0.1991 (0.2557)
R_{free}	0.2282 (0.2939)
R.m.s.d., bond lengths (Å)	0.006
R.m.s.d., bond angles (Å)	0.85

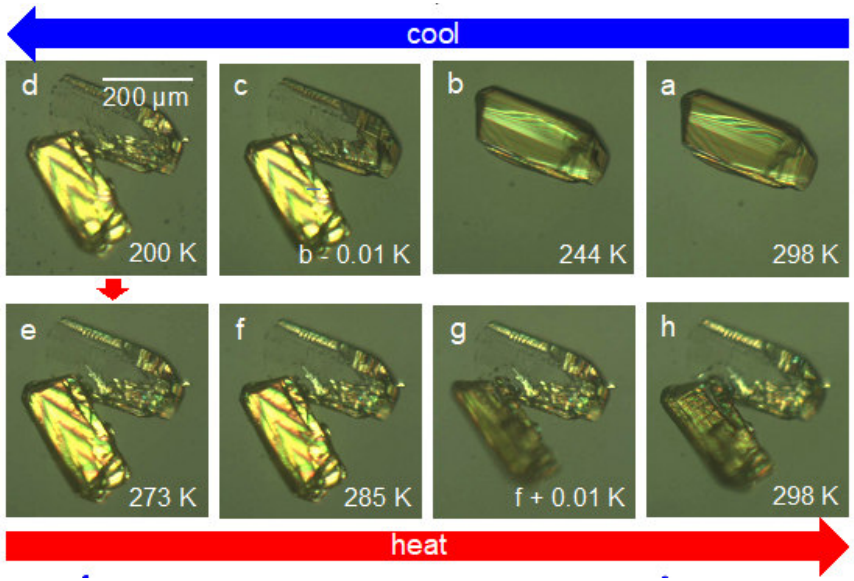


Densities of ligands bound to RNase A.
2Fo - Fc maps at 1σ

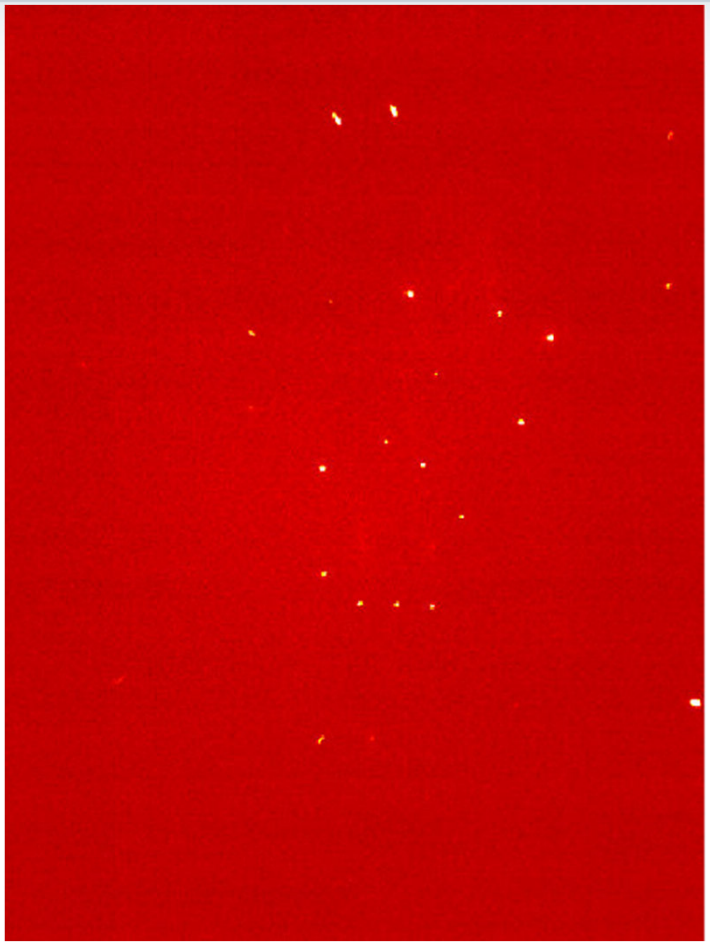
UW Madison – Ilia Guzei's Lab (IμS 3.0 EF)

Light sensitive RT polymorph of nifedipine – RT structure

Submitted for publication:
Reversible solid-state transformations: A new mechanism based on nitro torsion illustrated by nifedipine polymorphs.
Yue Gui, Xin Yao, Ilia A. Guzei, Michael M. Aristov, Junguang Yu, Lian Yu



Solid-state transformations of the γ polymorph

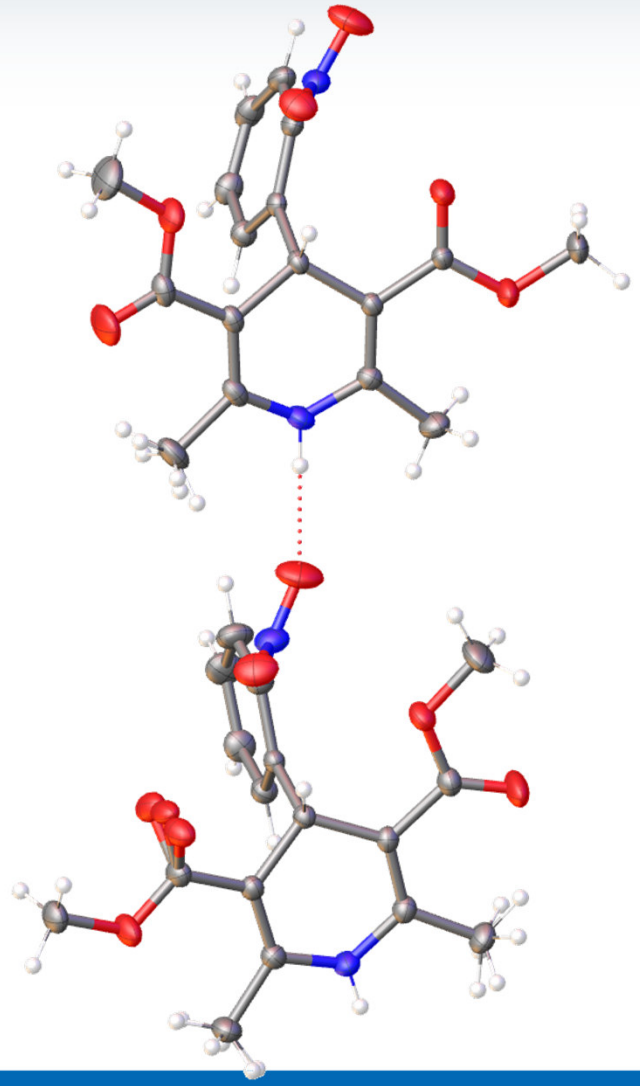


Diffraction pattern of a
0.04 × 0.04 × 0.04 mm^3 crystal

UW Madison – Ilia Guzei's Lab (IμS 3.0 EF)

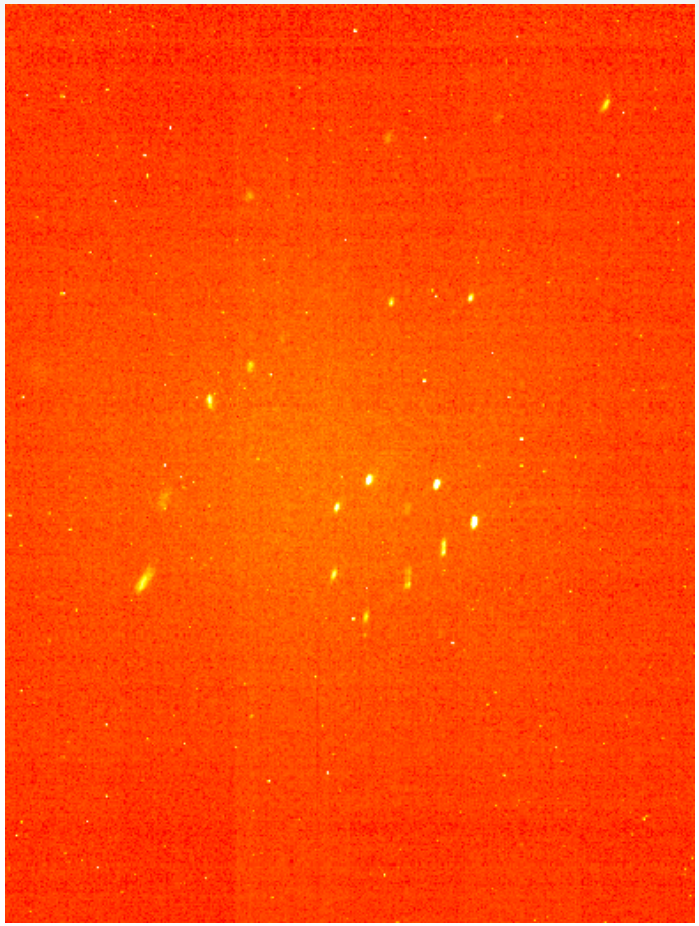
Light sensitive RT polymorph of nifedipine – RT structure

Form	γ'
T, K	296
a, Å	11.450
b, Å	12.301
c, Å	12.356
α, deg	75.63
β, deg	89.09
γ, deg	84.93
V, Å³	1679.19
space group	$P\bar{1}$
R1,%	4.38

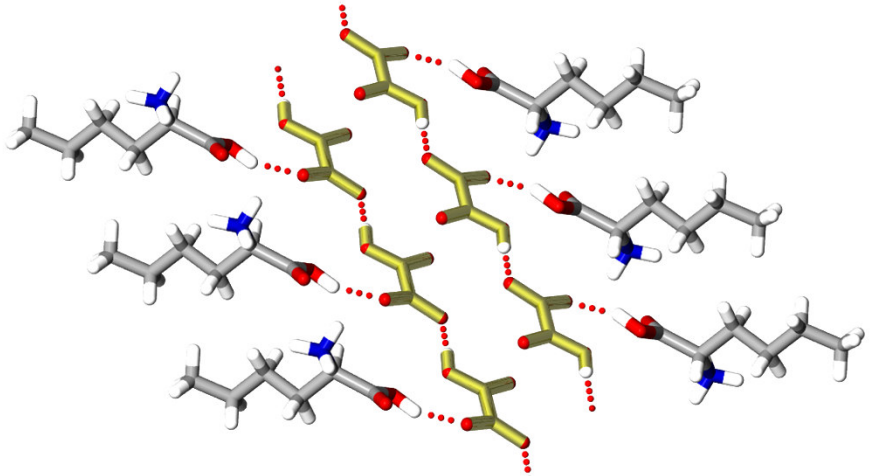


Whitworth University – Kraig Wheeler's Lab (IμS 3.0 EF)

Racemic Norleucinium Oxalate



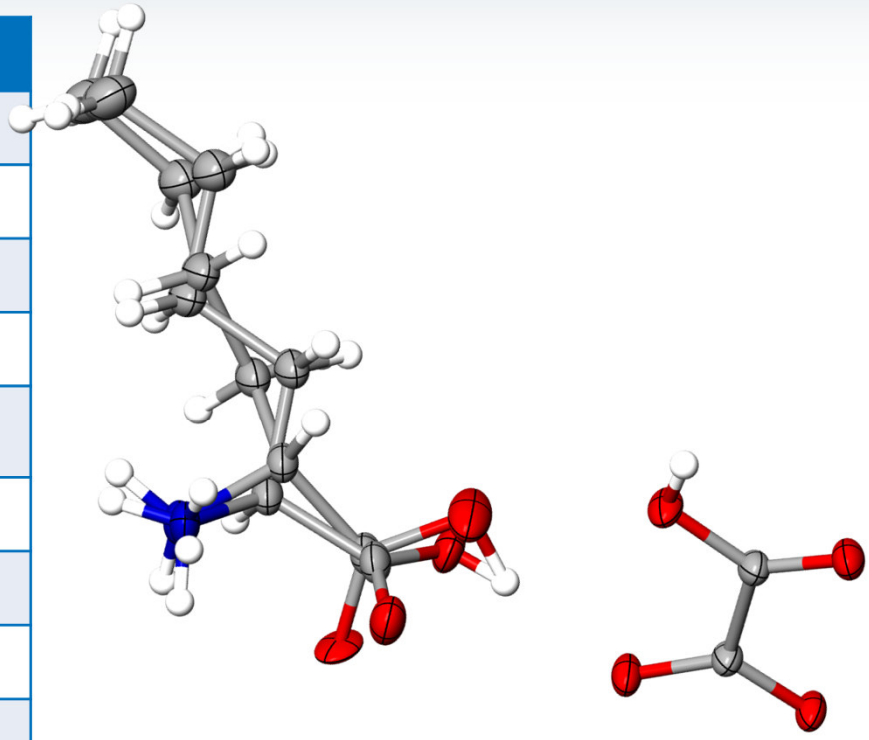
Diffraction pattern of a
0.028 × 0.122 × 0.401 mm^3 crystal



Whitworth University – Kraig Wheeler's Lab (IμS 3.0 EF)

Racemic Norleucinium Oxalate

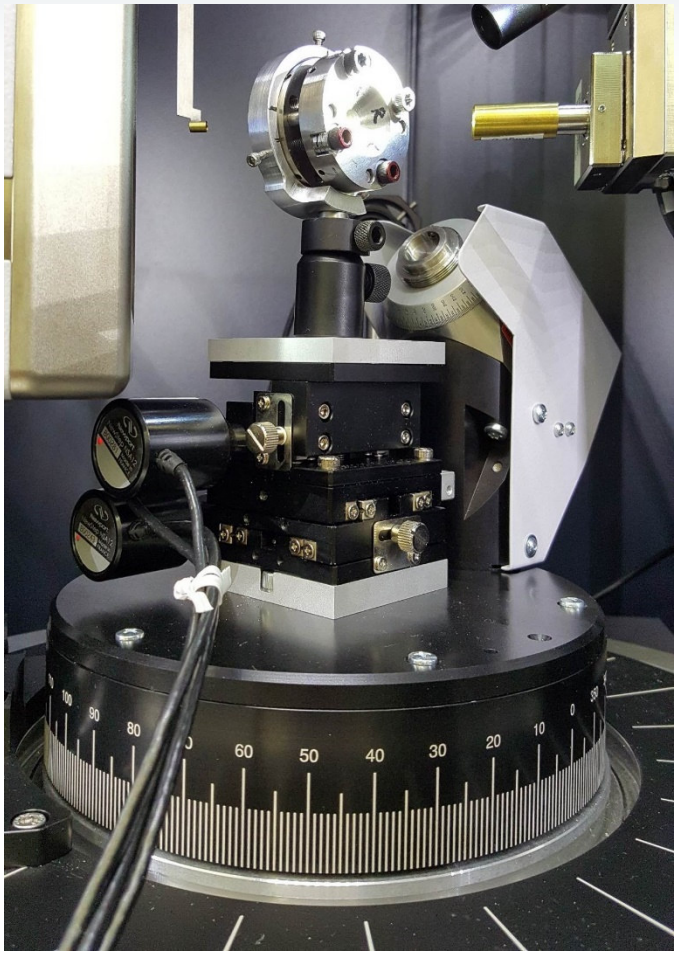
Norleucinium Oxalate	
T, K	100
a, Å	5.6270(5)
b, Å	9.4327(9)
c, Å	10.4431(10)
α, deg	88.922(4)
β, deg	75.976(4)
γ, deg	83.757(4)
V, Å³	534.57(9)
space group	$P\bar{1}$
R1,%	4.76



Norleucinium 90:10 disorder

UH Manoa - Przemyslaw Dera (IμS 3.0 Ag)

High pressure setup with 50 μm beam



Motorized heavy-duty (10lb load capacity) XYZ sample stage is small enough that motors fit underneath the detector. The motorized stage can accommodate cooling sample holder (not shown in the picture) for resistive heating experiments as well as open cradle chi stage.

UH Manoa - Przemyslaw Dera (IμS 3.0 Ag)

High pressure setup with 50 μm beam

	D8 VENTURE UHM	PX^2, MAR165 CCD/APS	D8 VENTURE UHM
Pressure	ambient	2 GPa	2 GPa
Wavelength	0.56086 Å	0.43000 Å	0.56086 Å
Crystal system	Monoclinic		
Space group	C2/c		
Unit cell dimensions	a = 9.835(7) Å	a = 9.8360(15) Å	a = 9.814(2) Å
	b = 18.01(2) Å	b = 18.068(11) Å	b = 18.076(4) Å
	c = 5.303(2) Å	c = 5.2730(7) Å	c = 5.284(2) Å
	b= 104.71(4)°	b= 104.801(13)°	b= 104.78(3)°.
Volume	908.6(13) Å$_3$	906.0(6) Å$_3$	906.4(4) Å$_3$
Theta range for data collection	3.17 to 27.96°.	1.46 to 20.20°.	1.91 to 18.13°.
Exposure time	20 sec/deg	10 sec/deg	40 sec/deg
Index ranges	-16<=h<=16, -29<=k<=30, -8<=l<=7	-11<=h<=12, -12<=k<=8, -5<=l<=7	-8<=h<=8, -17<=k<=17, -5<=l<=5
Reflections collected	5959	1045	3765
Independent reflections	2254 [R(int) = 0.0409]	375 [R(int) = 0.0740]	364 [R(int) = 0.1003]
Completeness to	27.96°/98.5 %	20.20°/18.5 %	18.13°/54.2 %
Data / restraints / parameters	2254 / 0 / 104	375 / 0 / 63	364 / 0 / 51
Goodness-of-fit on F_2	1.043	1.035	1.006
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0718	R1 = 0.0437, wR2 = 0.1187	R1 = 0.0728, wR2 = 0.1731
R indices (all data)	R1 = 0.0500, wR2 = 0.0820	R1 = 0.0438, wR2 = 0.1187	R1 = 0.1203, wR2 = 0.2129
Largest diff. peak and hole	0.799 and -0.692 e.Å^{-3}	0.492 and -0.430 e.Å^{-3}	0.735 and -0.662 e.Å^{-3}

Comparison single crystal data collected on X-ray Atlas at ambient and high pressure, and at APS experimental station 13BM-C on the same sample of ferroactinolite $Ca_2(Mg_{2.5\text{-}5.0}Fe^{2+}{}_{2.5\text{-}5.0})Si_8O_{22}(OH)_2$ amphibole. The sample crystal was 20 × 20 × 5 micrometers in size, loaded in Ne.

IU Bloomington- Maren Pink's Lab (IμS 3.0 Mo, IμS DIAMOND Cu)

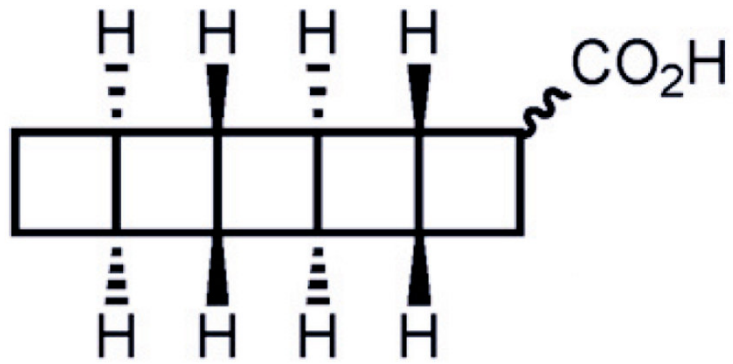
Natural Products - Ladderanes

Angewandte International Edition Chemie
GDCh A Journal of the German Chemical Society

Research Article | Free Access

Lessons in Strain and Stability: Enantioselective Synthesis of (+)-[5]-Ladderanoic Acid

Erin N. Hancock, Erin L. Kuker, Prof. Dean J. Tantillo, Prof. M. Kevin Brown

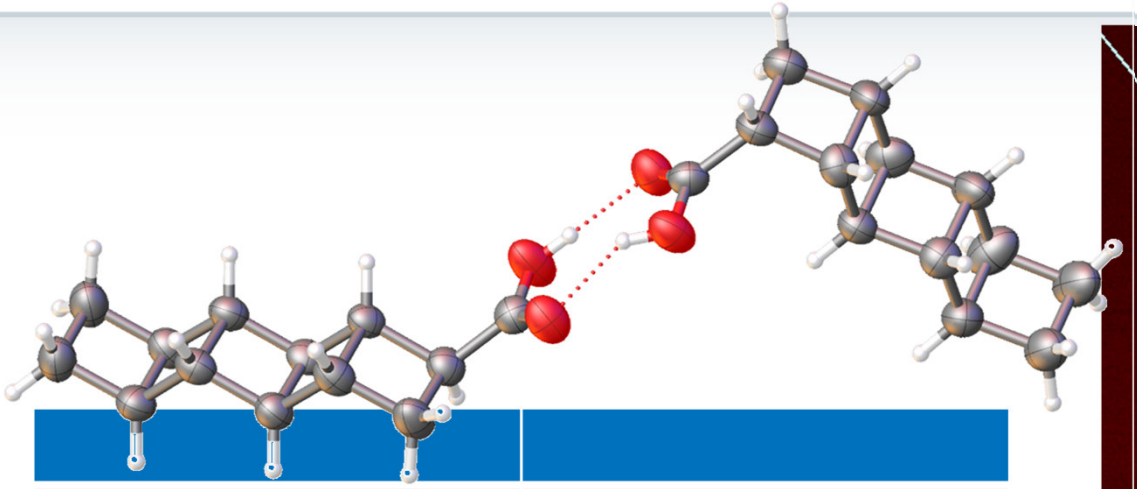


…The ladderane family of natural products is a unique class of molecules that is characterized by a series of fused cyclobutanes.[1, 2] Because of their complex structure and unknown biological function,[3] several groups have developed routes to these molecules (Scheme 1)…

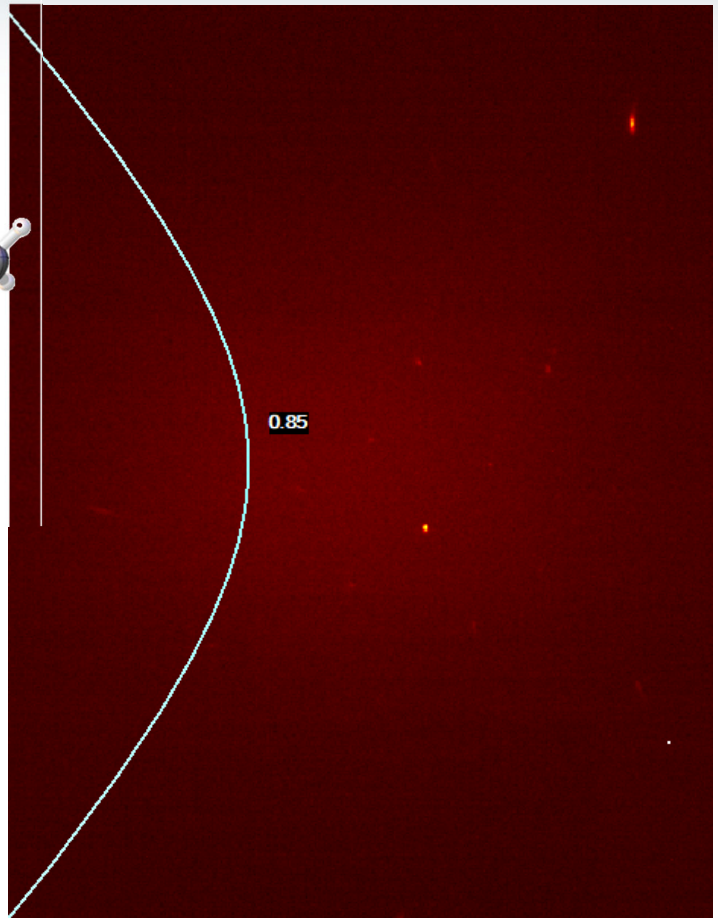
These ladderane crystals are super thin and bendable. So, high intensity sources are a must. Without the IμS, I would have taken them to the synchrotron. The IμS has taken a chunk out of what we would have to bring to ChemMatCARS otherwise.

IU Bloomington- Maren Pink's Lab (IμS DIAMOND Cu)

Natural Products - Ladderanes



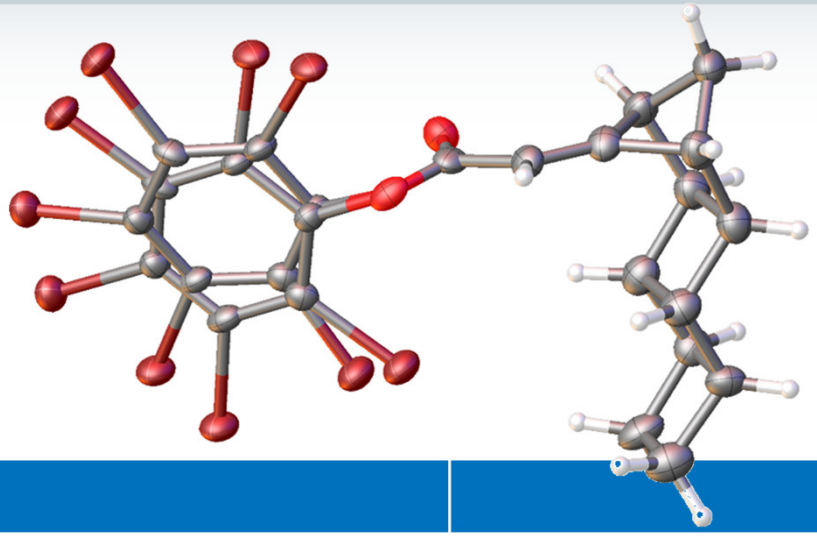
Crystal system	Monoclinic,
space group	C2
Unit cell dimensions	a = 8.9241(6) Å
	b = 6.1187(4) Å
	c = 38.303(2) Å
	b = 90.524(4)°.
R1	7.59%
wR2	20.71%



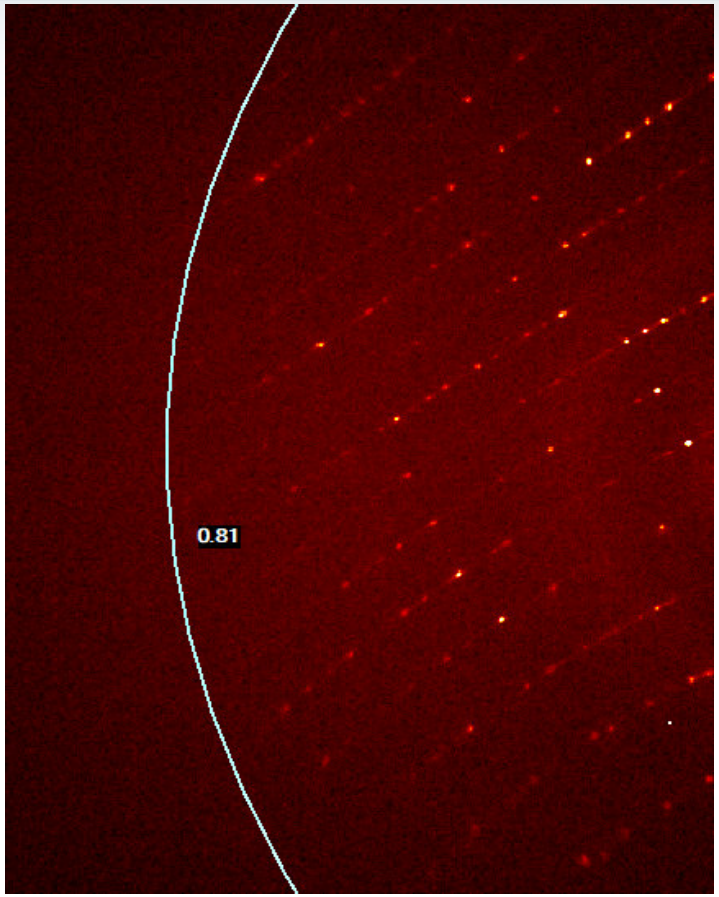
Diffraction pattern of a
0.09 × 0.06 × 0.01 mm^3 crystal 25s/0.5°

IU Bloomington- Maren Pink's Lab (IμS 3.0 Mo)

Natural Products - Ladderanes



Crystal system	Orthorhombic,
space group	$P2_12_12$
Unit cell dimensions	a = 25.325(3) Å
	b = 27.082(3) Å
	c = 9.0645(10) Å
R1	5.63%
wR2	12.95%



Diffraction pattern of a $0.09 \times 0.07 \times 0.02$ mm^3 crystal 20s/0.5°

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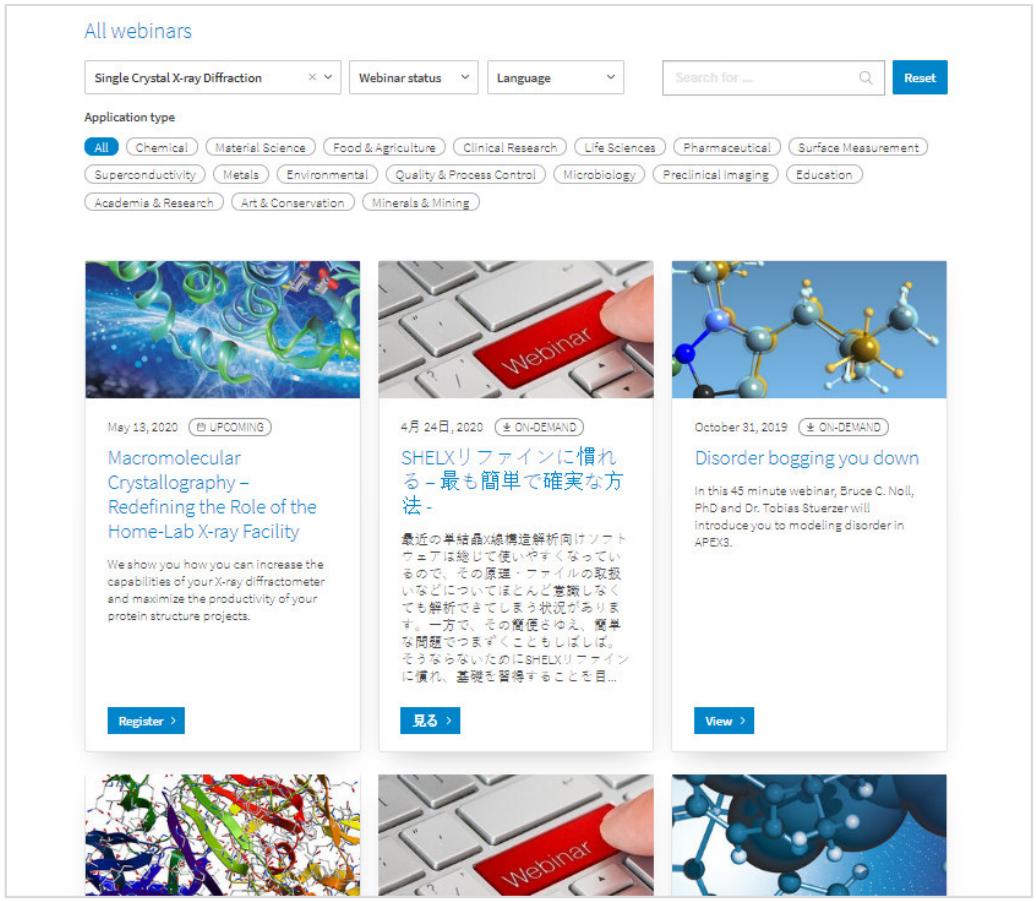

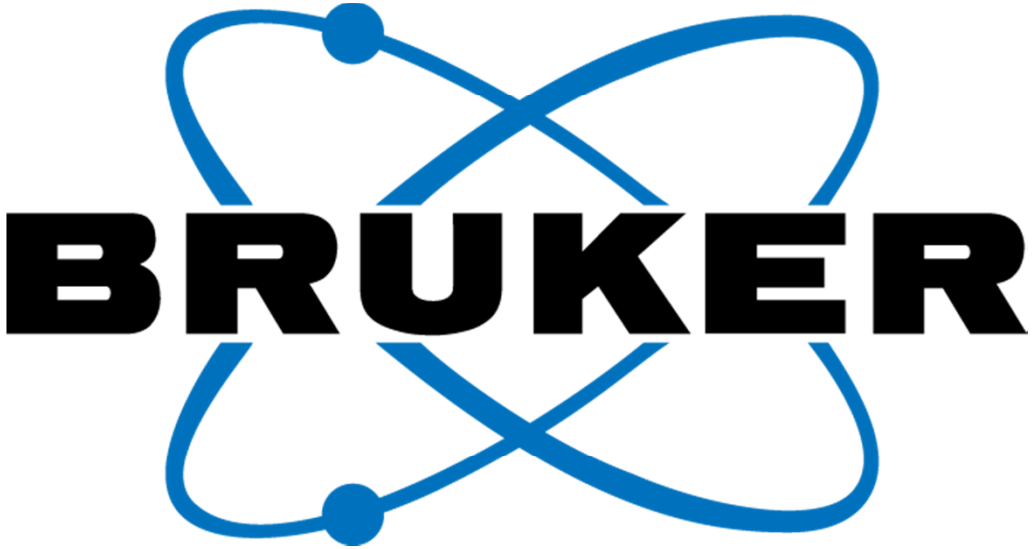

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