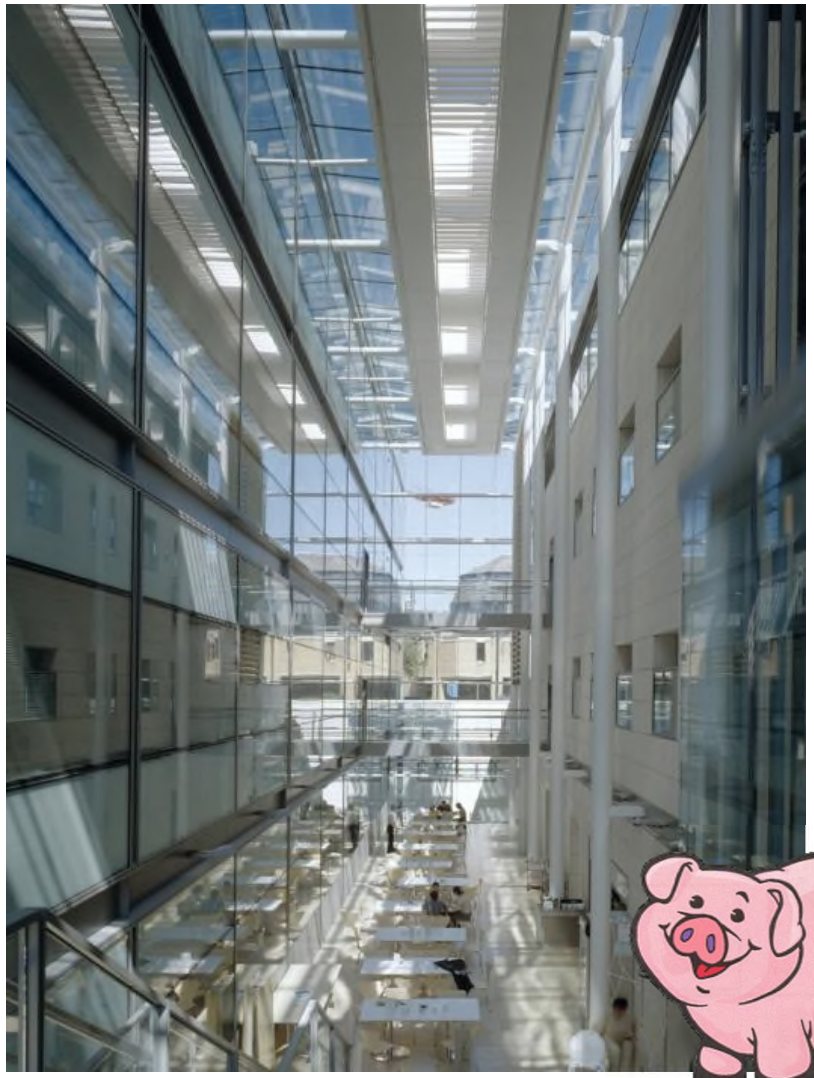
Making a Silk Purse out of a Sow's Ear

Publishing Difficult Data

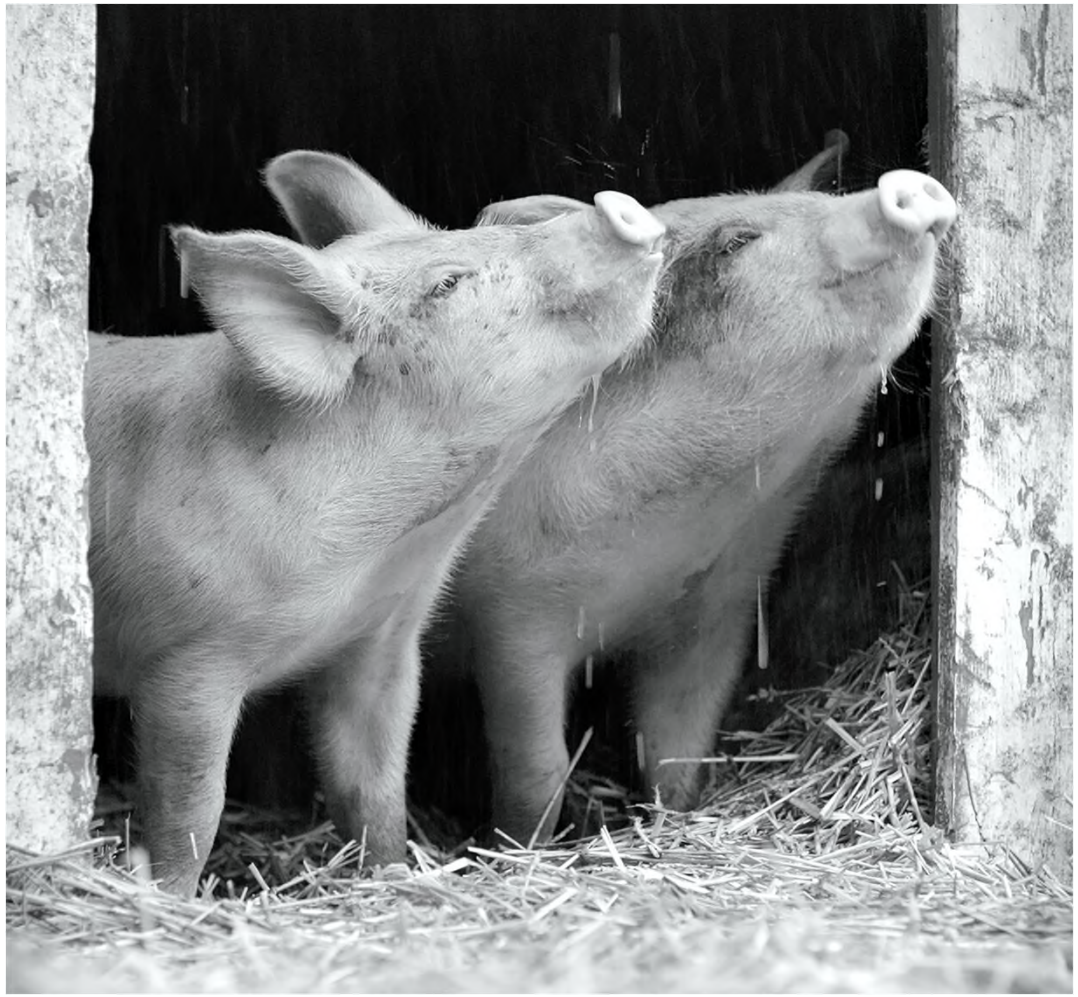
Amber L. Thompson

Oxford

Chemical Crystallography, Oxford



A Chemical Crystallography Service



- “Just crystal Structures”
- Support for research
- Support for manuscripts
- Minimal involvement in “the Science”
- “Routine”

“Stamp Collecting”

Increased Automation

User

- Choose and mount crystal
- Start screening image collection

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- Start the data collection

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- Publish the result

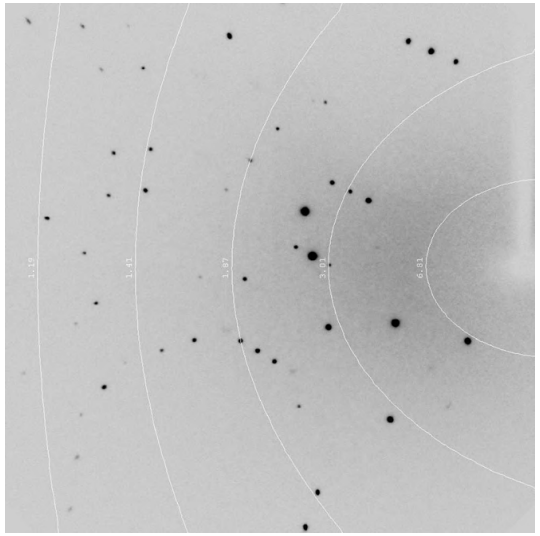
Automation

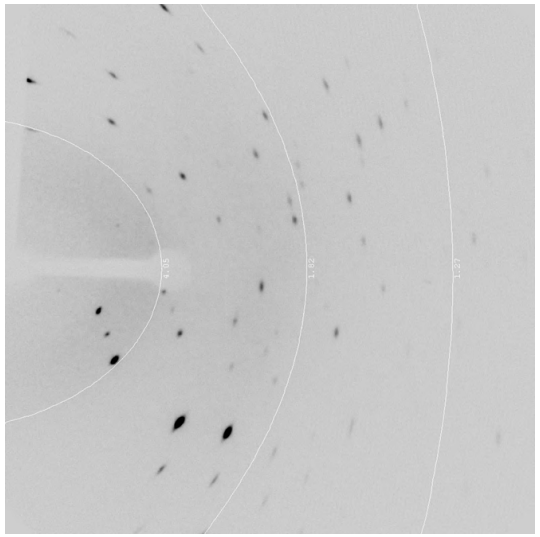
- Index the data
- Determine the Laue class
- Decide the acquisition time
- Decide the strategy

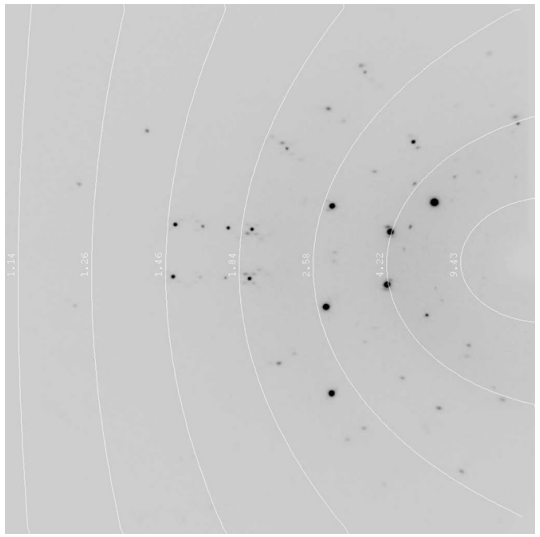
- Integrate the data
- Determine the space group
- Solve and refine the structure

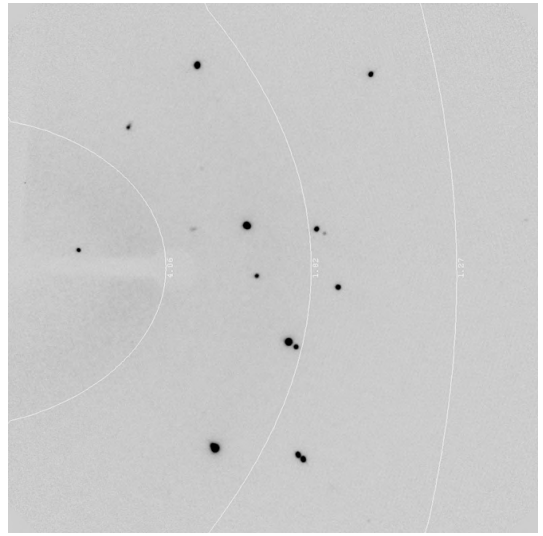

The
Piggin'
Problem...

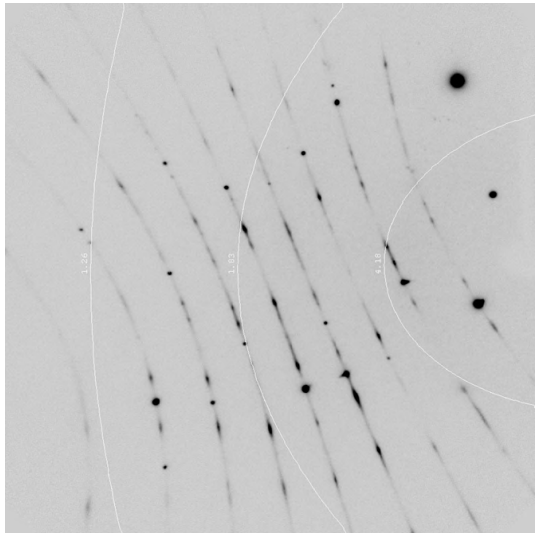
Image Inspection

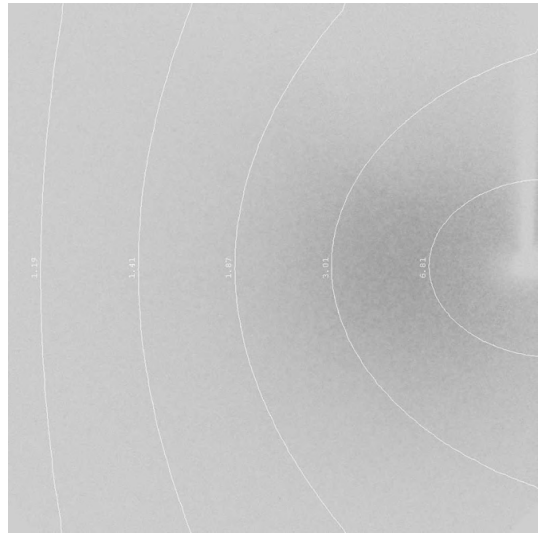




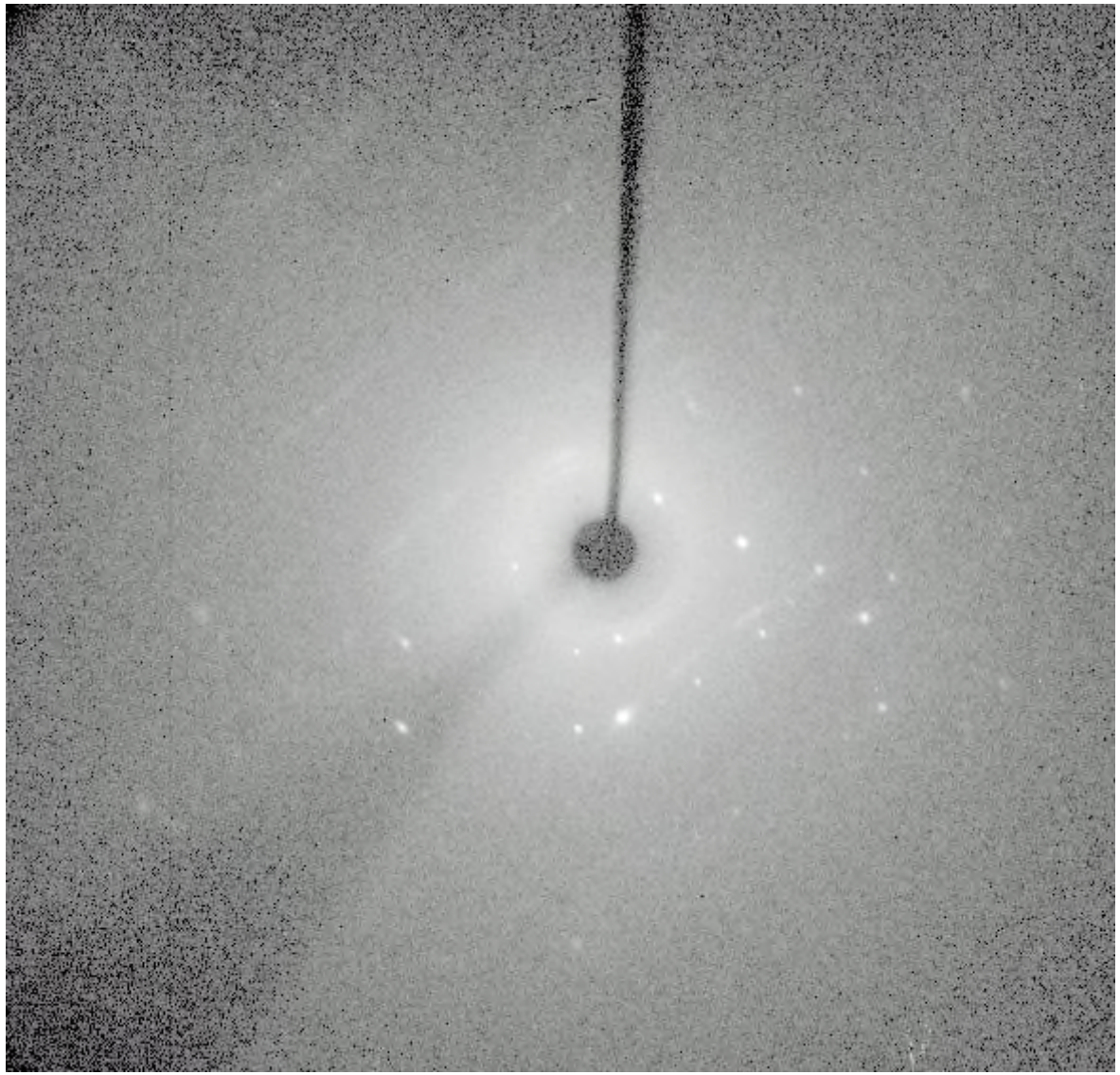




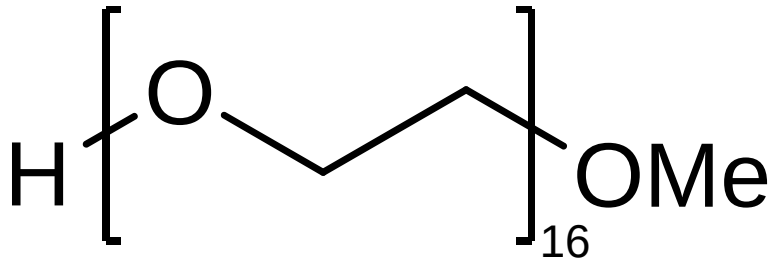


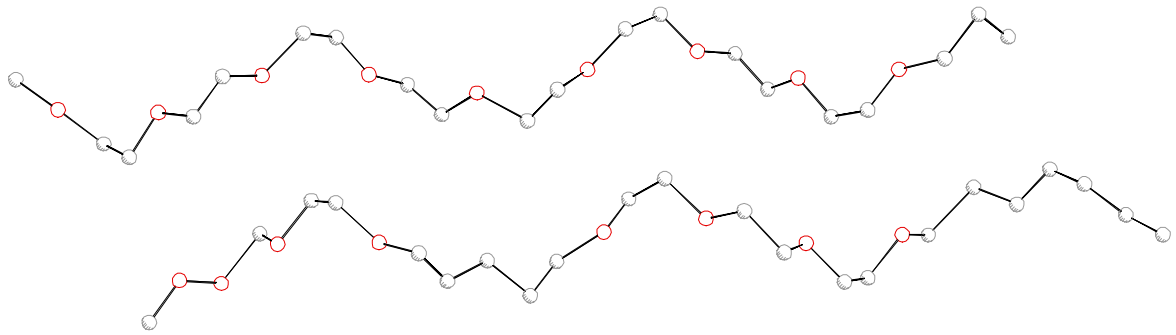


Thompson, *Cryst. Rev.,* 2019, **25**(1), 3-53.

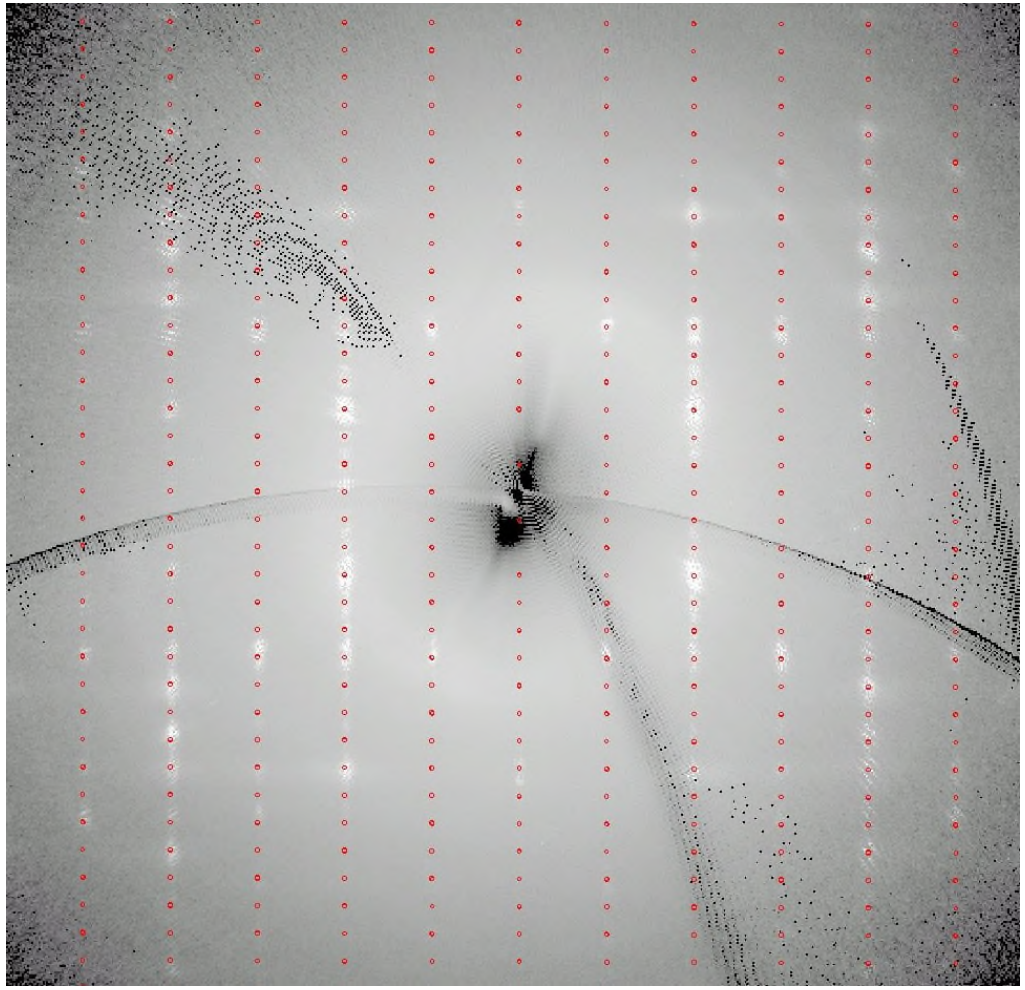


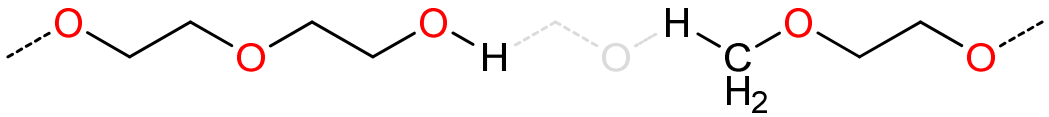
PEG_{16}

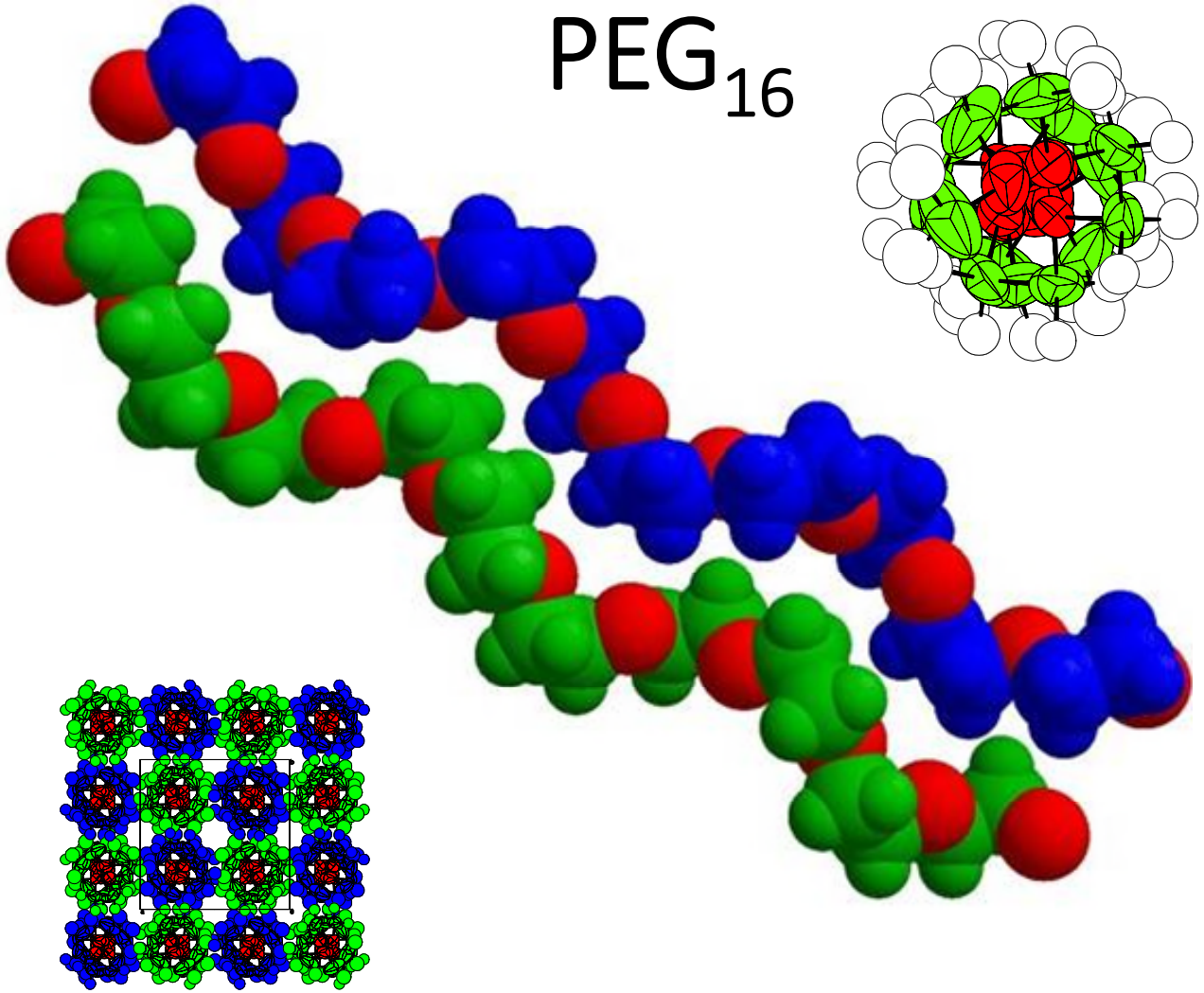




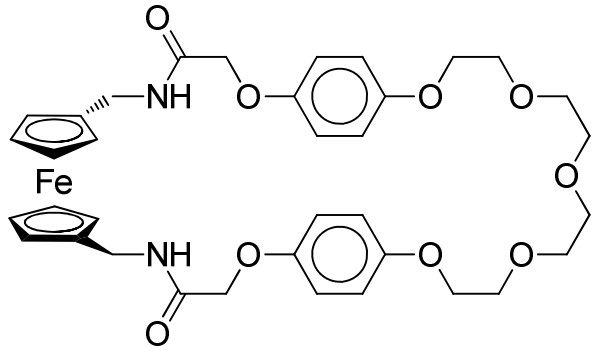
French *et al.*, *Angew. Chem. Int. Ed.,* 2009, **121**, 1274-1278.



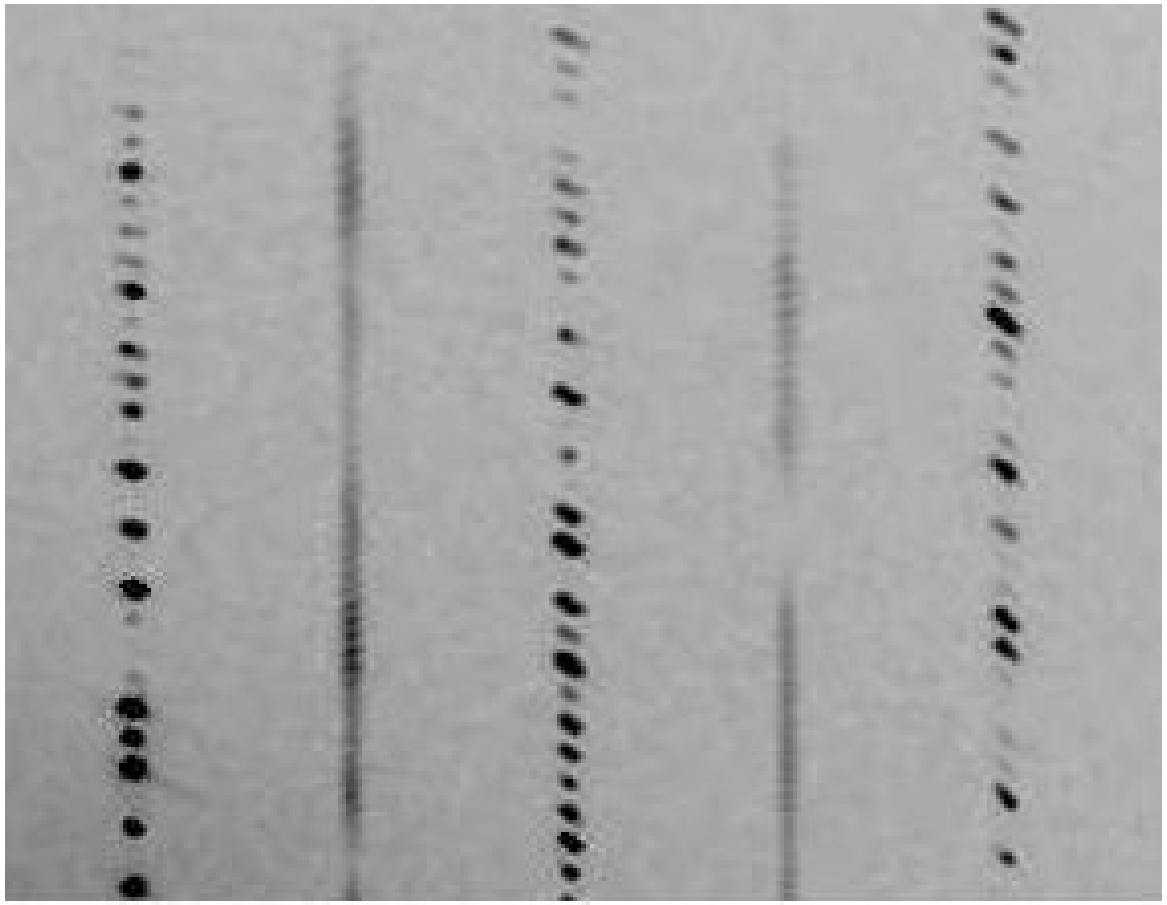


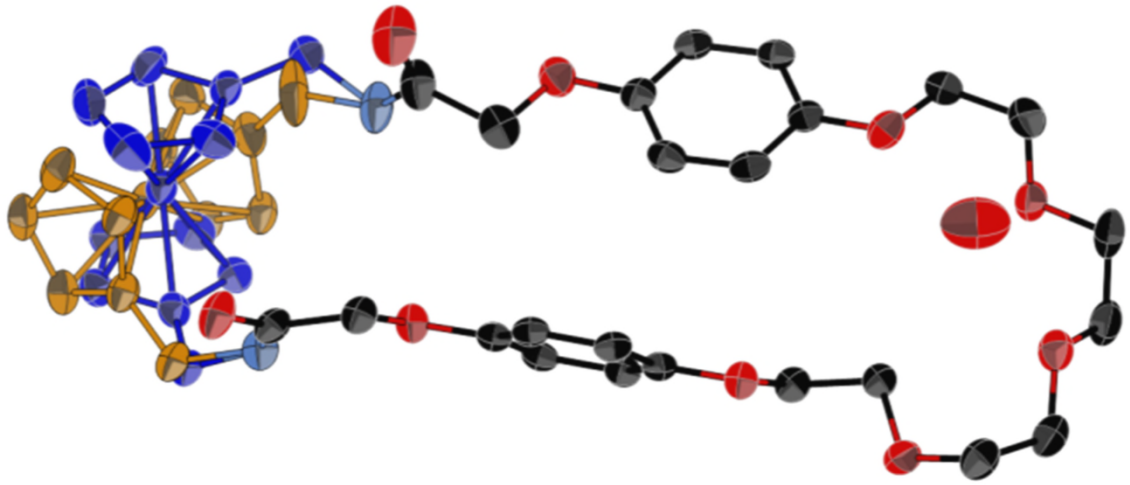


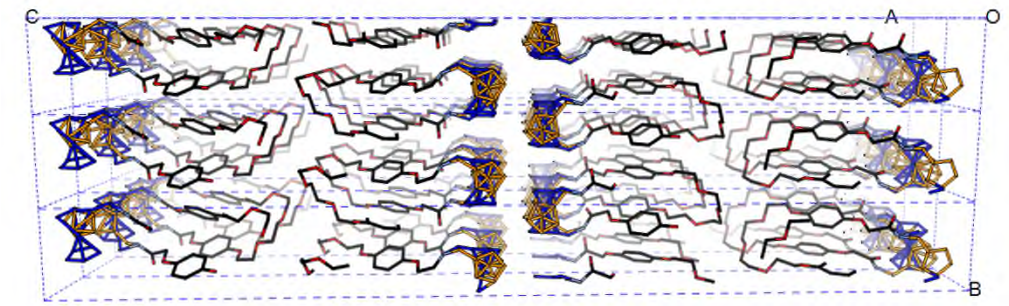
French *et al.*, *Angew. Chem. Int. Ed.*, 2009, **121**, 1274-1278.



Ferrocene Macrocycle

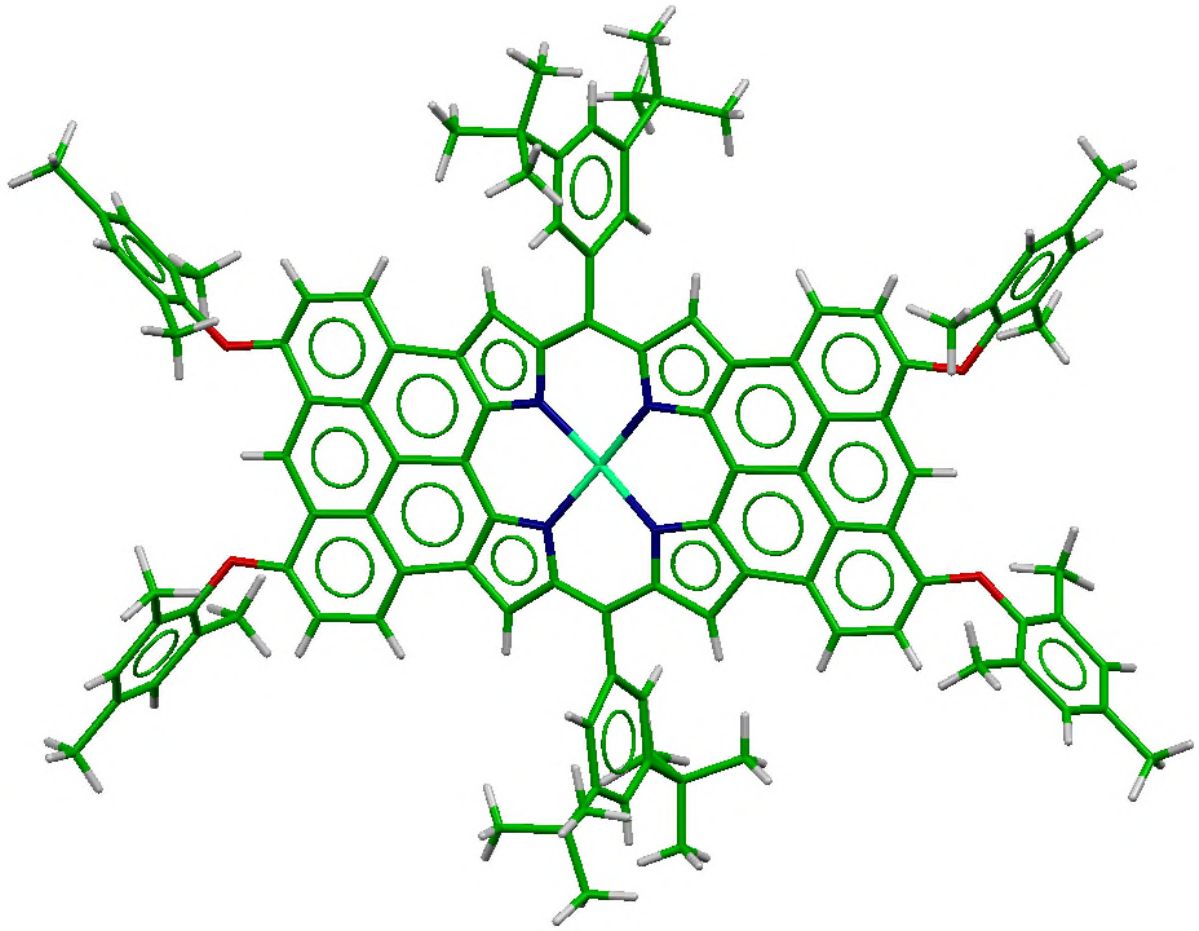


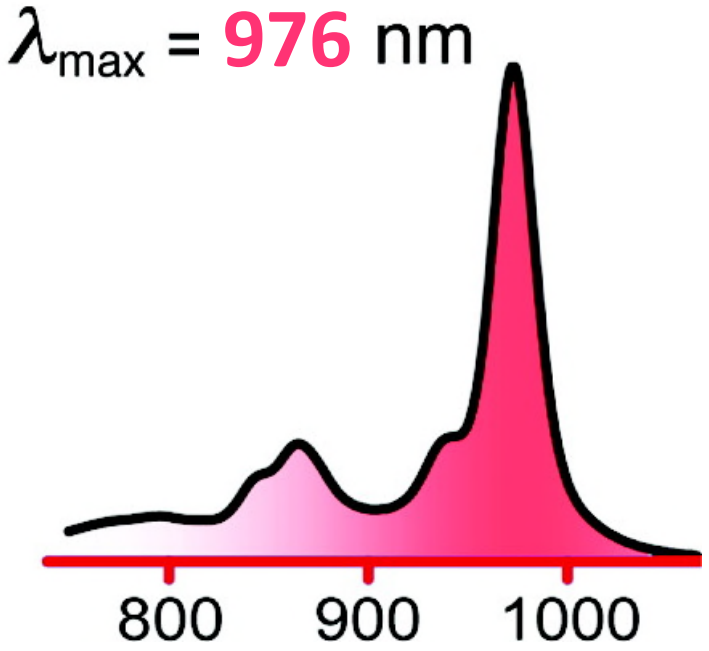




Evans *et al.*, *Eur. J. Inorg. Chem.,* 2012, **6**, 939-944.

Poor Structure or Poor Data?





Davis *et al.*, *Org. Lett.*, **2010**, 12, 2124

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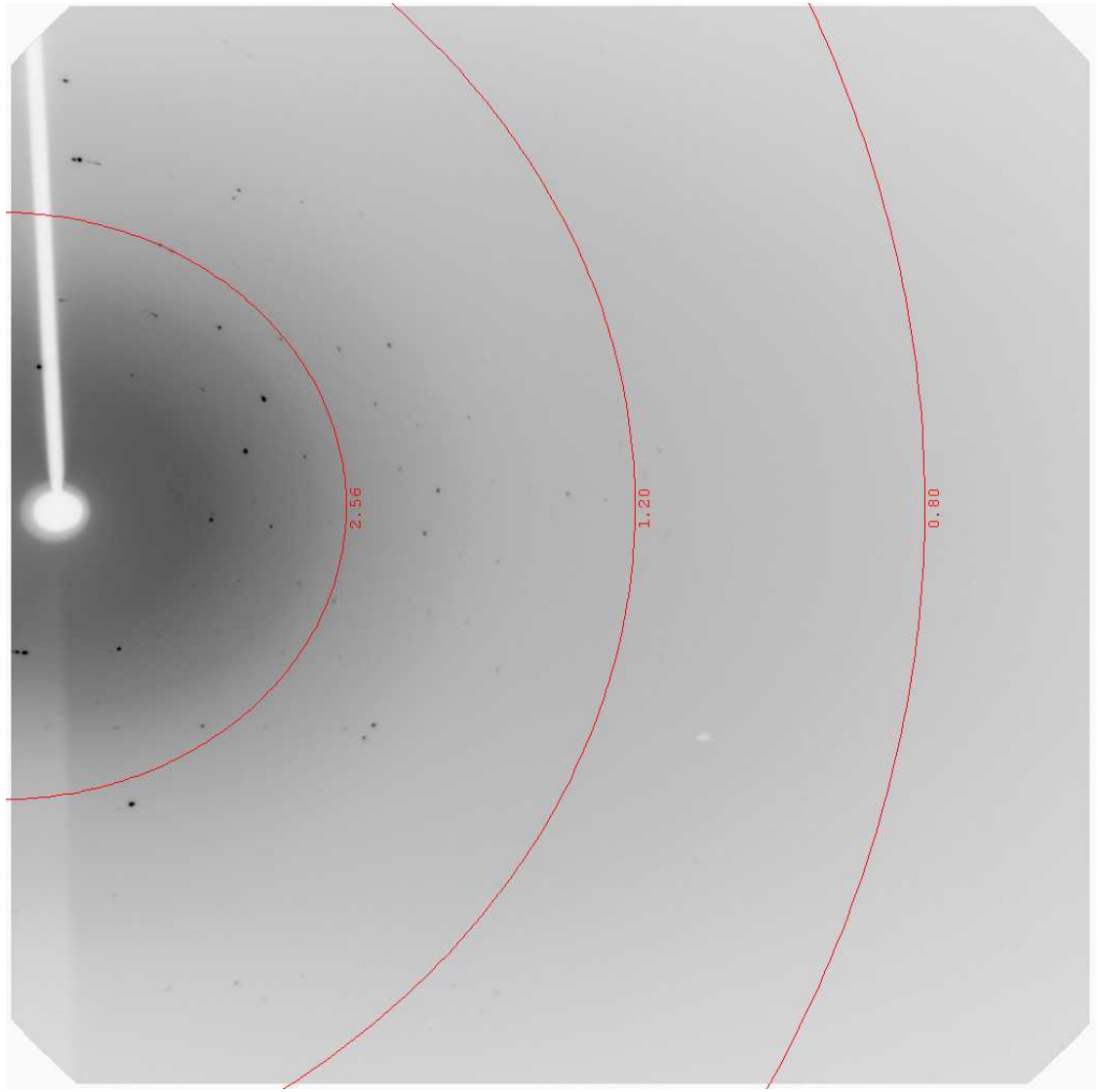
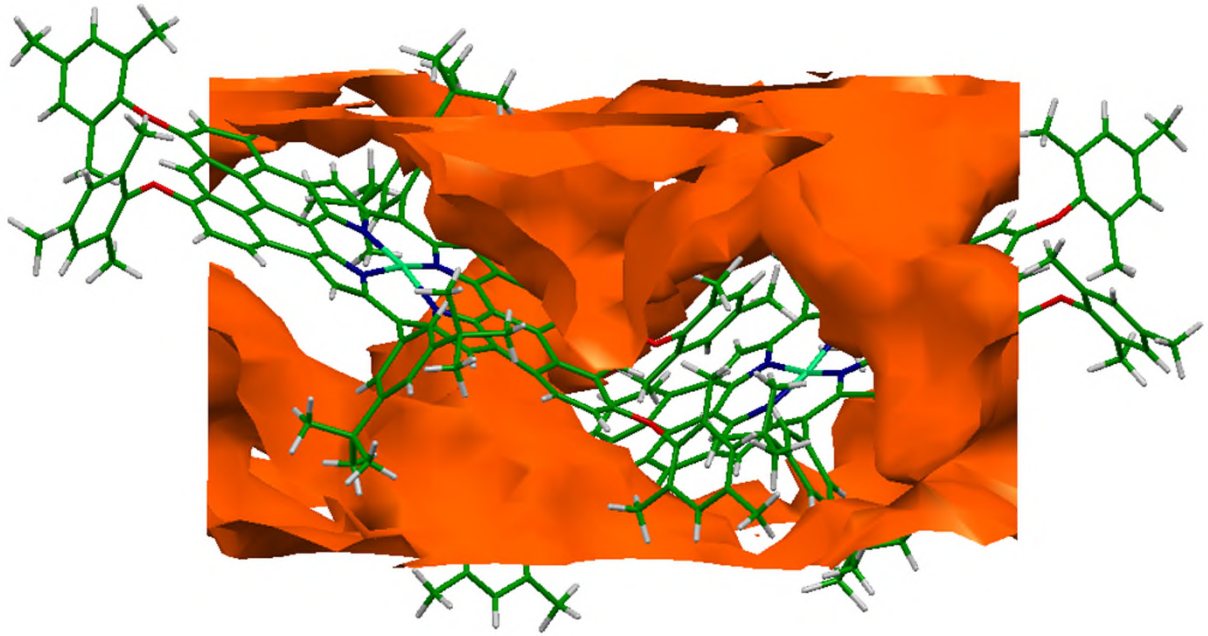
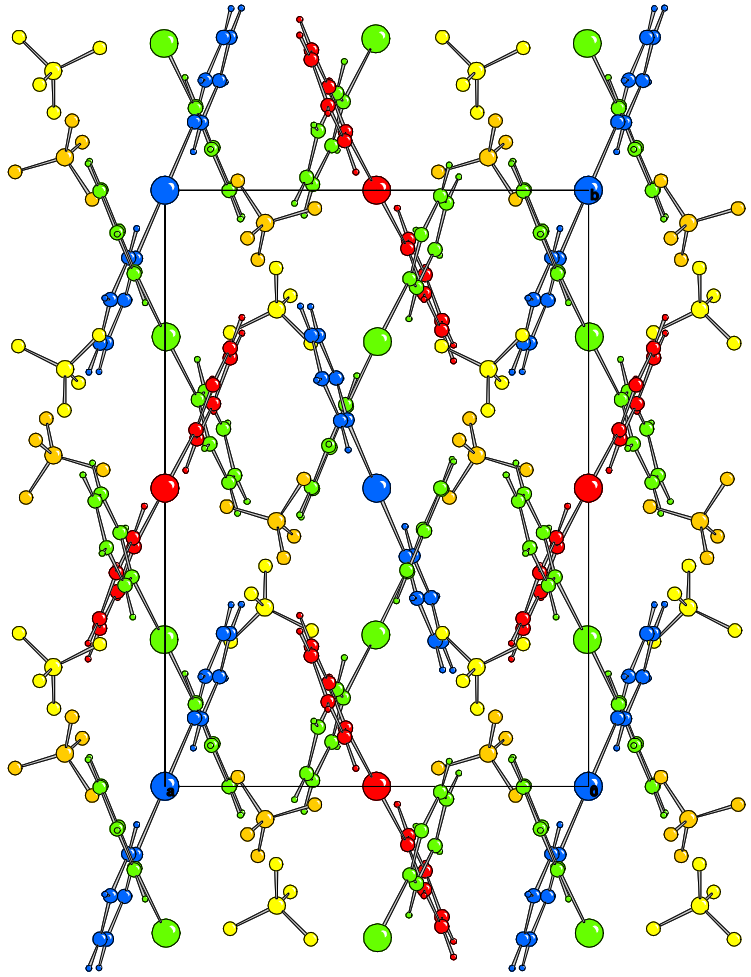




52% Solvent

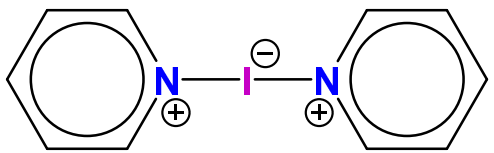
Davis *et al.*, *Org. Lett.*, **2010**, 12, 2124

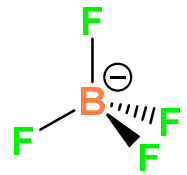
Barluenga's Reagent



150 K $P2_1/n$

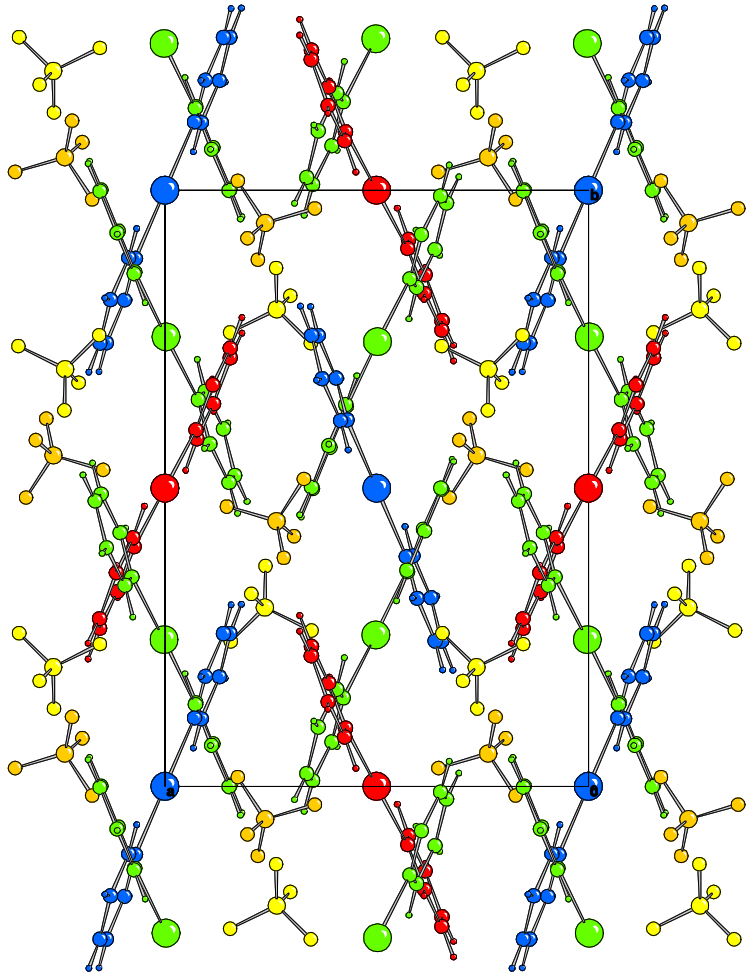
- Justin Chalker & Ben Davis (February 2009)
  - A safer, better synthesis
  - "It's a known structure, we just want to confirm that it's the same…"

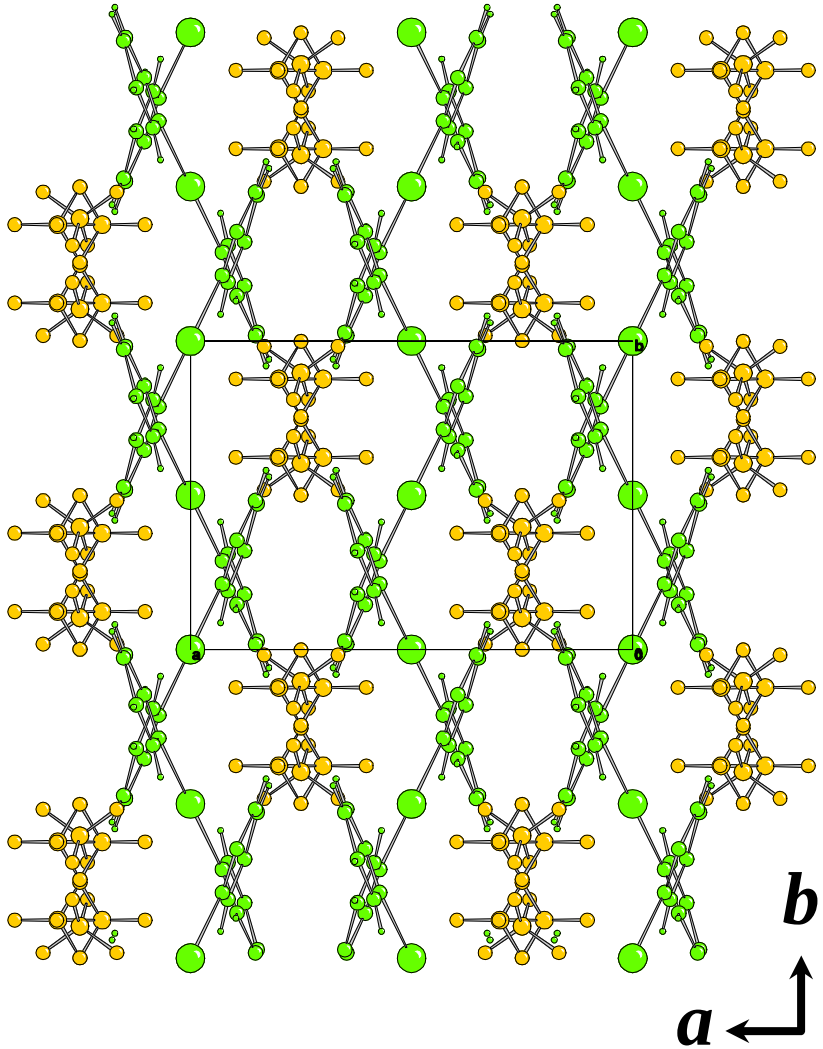




Chalker *et al.*, *Org. Syn.,* 2010, **87**, 288-298.

Barluenga's Reagent





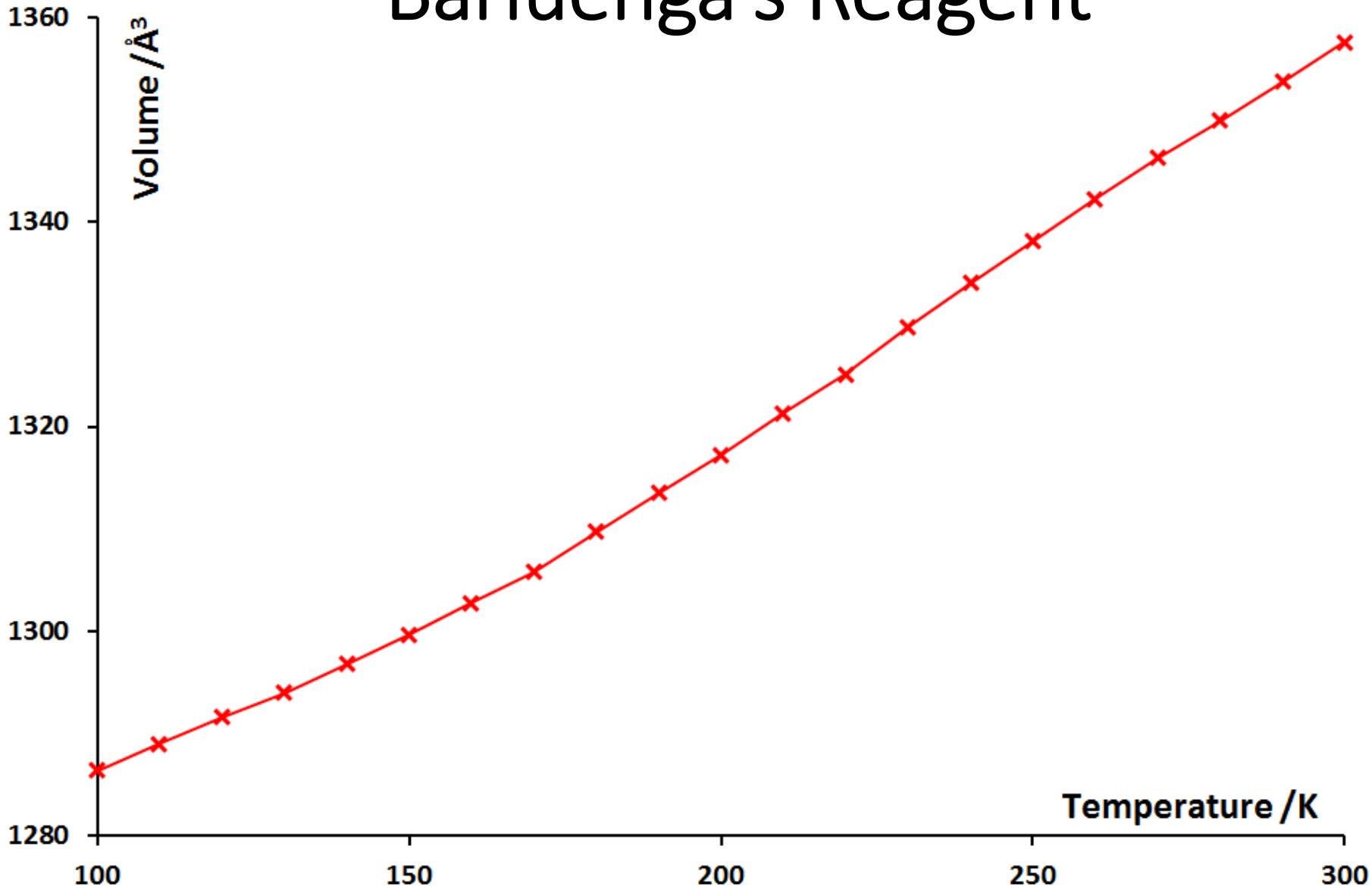
250 K

a= 12.3206 Å
b= 7.9297 Å
c= 14.7047 Å
β= 113.290˚

150 K

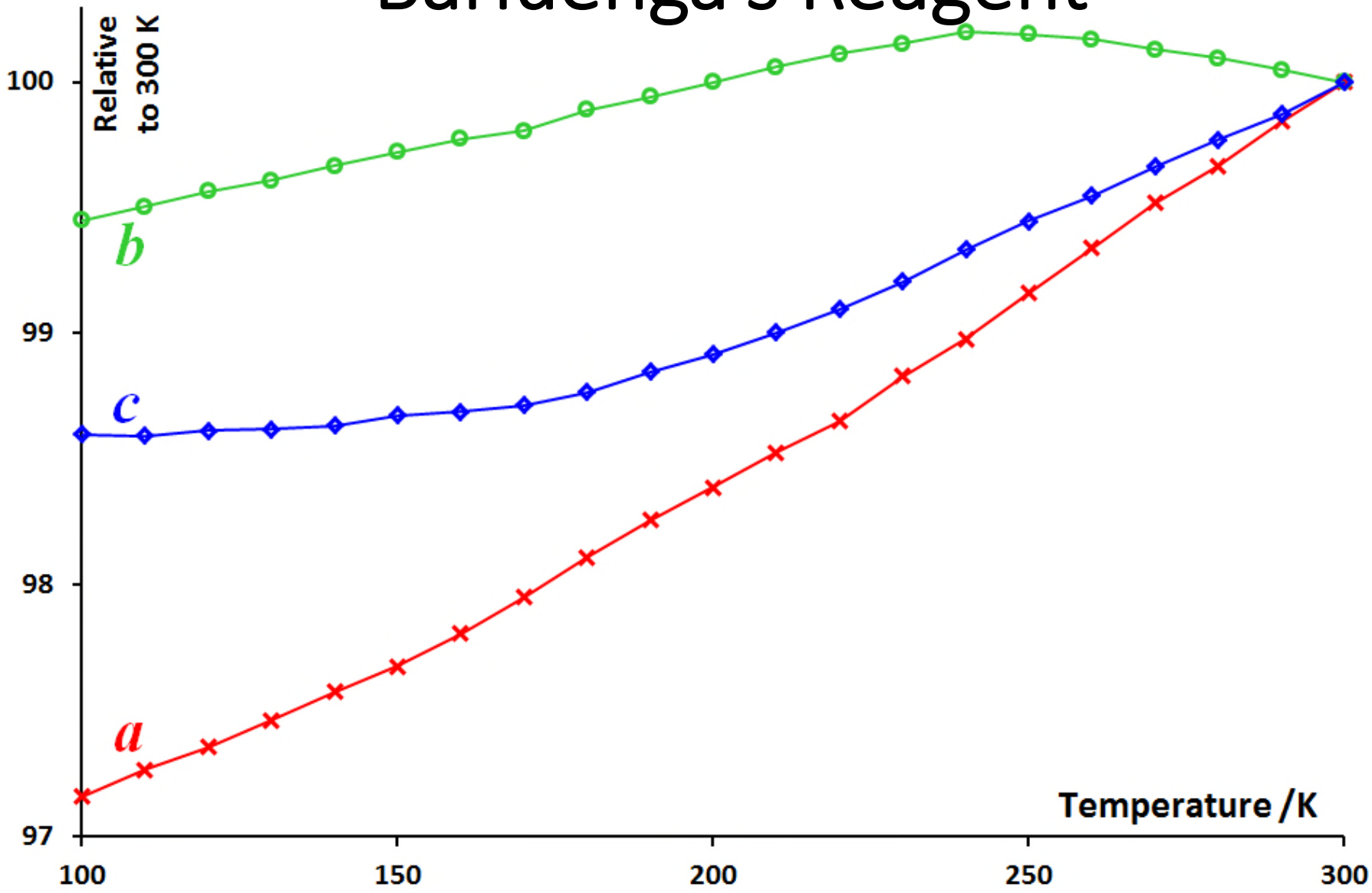
a= 12.1662 Å
b= 15.7653 Å
c= 14.6030 Å
β= 113.489˚

Barluenga's Reagent



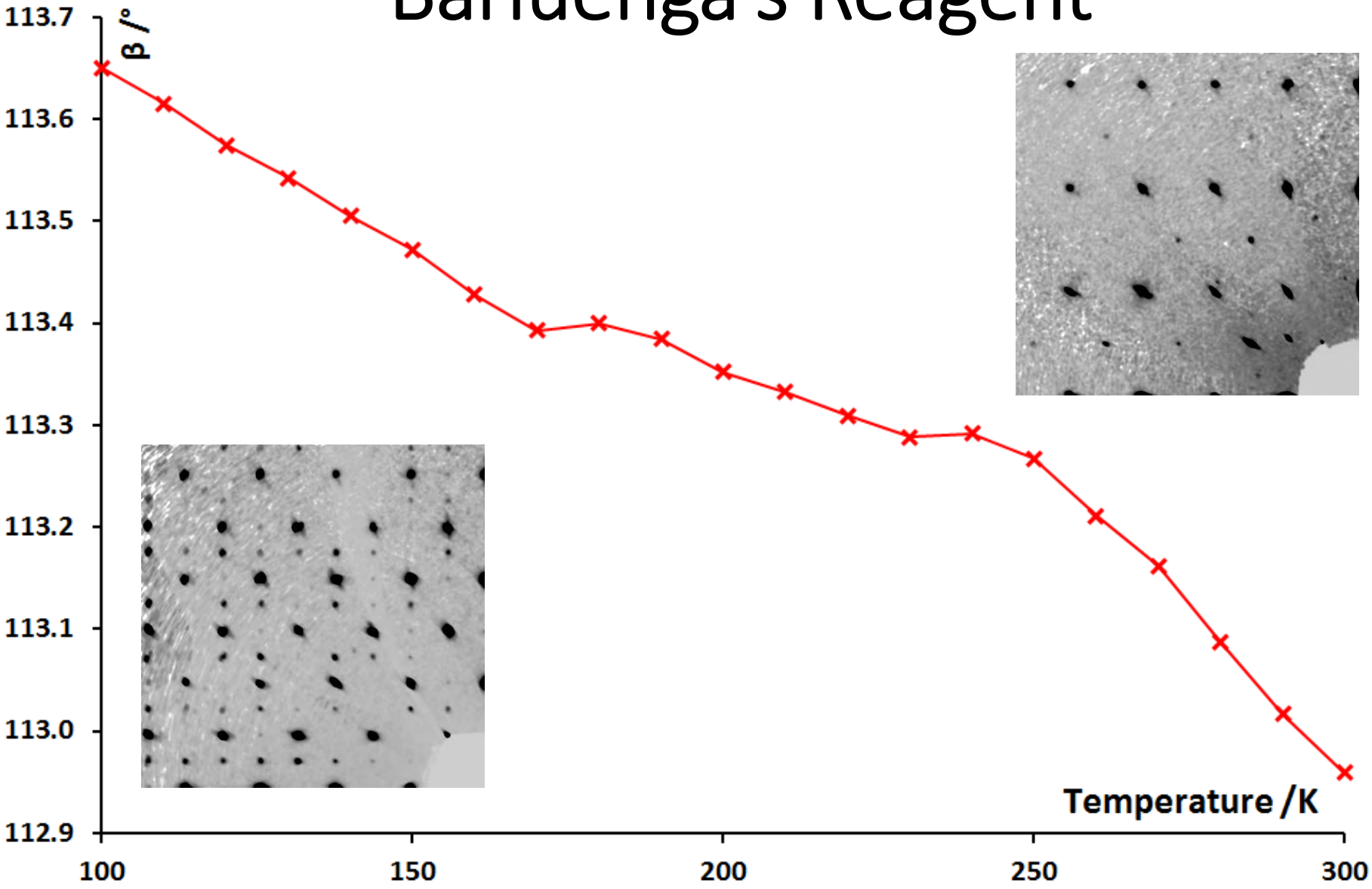
Kim, McKinley *et al.*, *Cryst. Growth Des*, 2014, **14**, 6294-6301.

Barluenga's Reagent



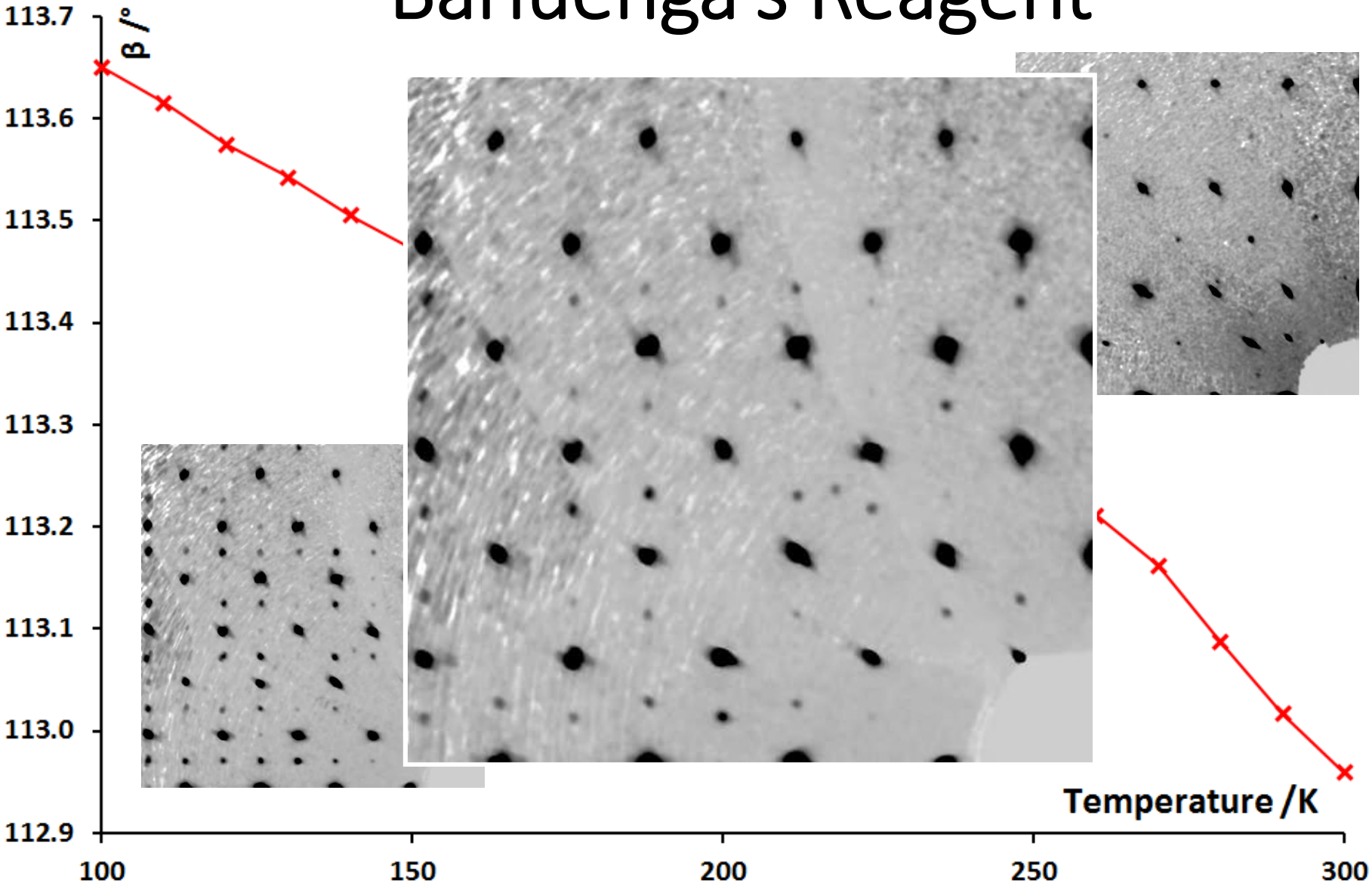
Kim, McKinley *et al.*, *Cryst. Growth Des*, 2014, **14**, 6294-6301.

Barluenga's Reagent



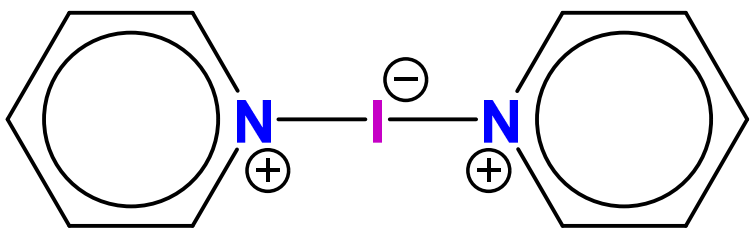
Kim, McKinley *et al.*, *Cryst. Growth Des*, 2014, **14**, 6294-6301.

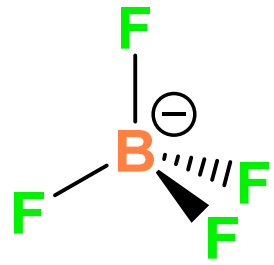
Barluenga's Reagent



Kim, McKinley *et al.*, *Cryst. Growth Des*, 2014, **14**, 6294-6301.

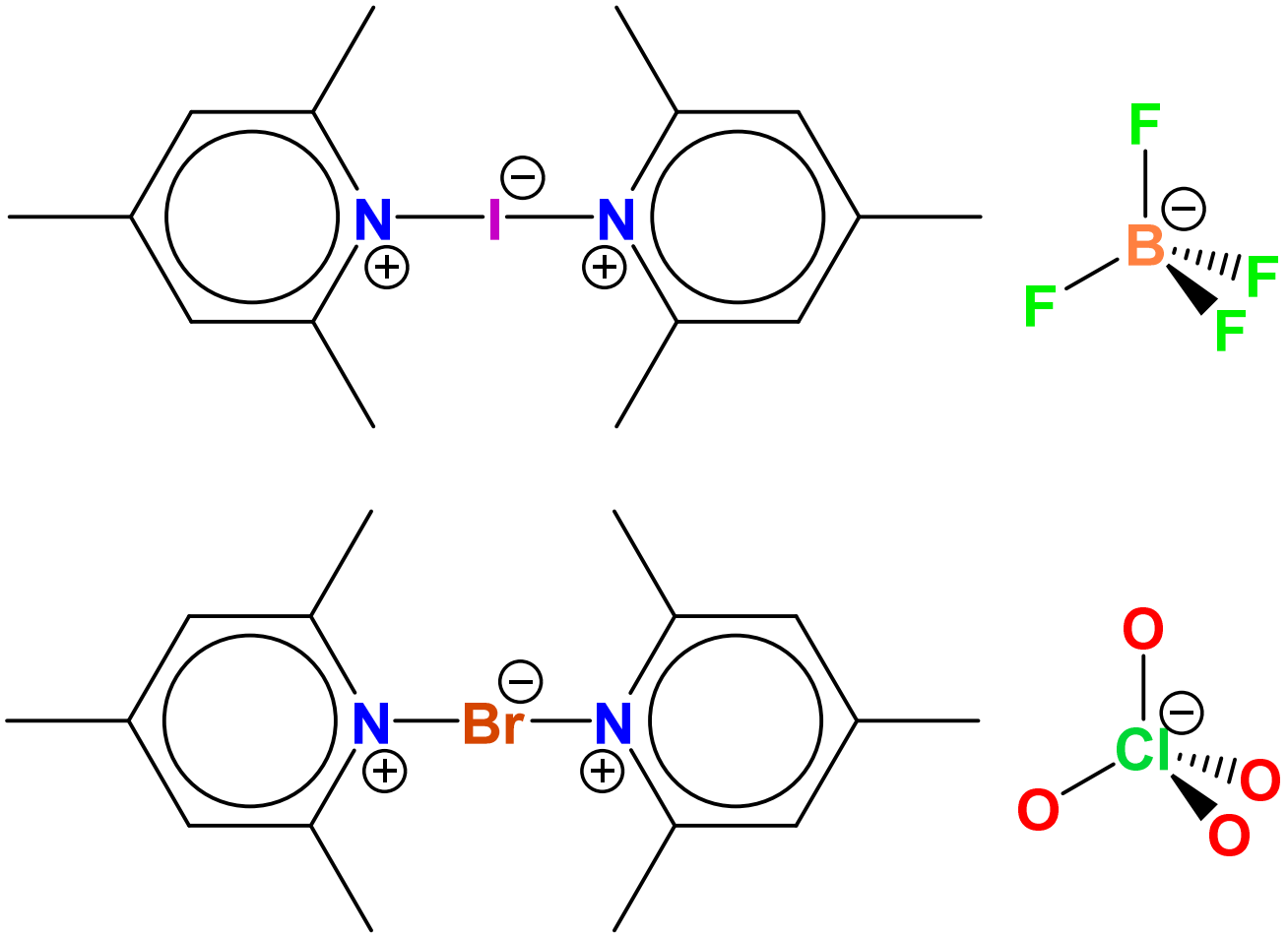
Barluenga's Reagent Derivatives





Kim, McKinley *et al.*, *Cryst. Growth Des,* 2014, **14**, 6294-6301.

Barluenga's Reagent Derivatives

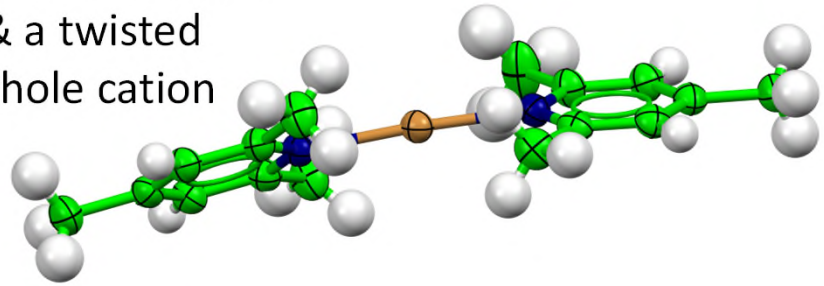


Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

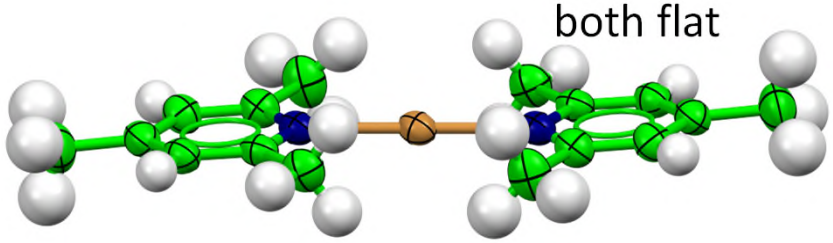
Barluenga's Reagent Derivatives

	Polymorph I		Polymorph II	
	$I(Coll)_2ClO_4$ (BAZNAR)	$Br(Coll)_2ClO_4$ (AKOXON)	$I(Coll)_2ClO_4$	$Br(Coll)_2ClO_4$
a /Å	27.844(5)	27.280(30)	29.1027(8)	28.8059(14)
b /Å	11.036(2)	11.028(11)	8.5667(2)	8.5296(3)
c /Å	23.412(7)	23.551(20)	15.9883(5)	15.8383(7)
β/°	126.47(2)	127.19(1)	100.804(3)	99.565(4)
Volume /Å	5785.7	5644(9)	3915.47(19)	3837.4(3)
Space Group	C2/c	C2/c	C2/c	C2/c

A flat half molecule & a twisted whole cation



2 half cations both flat

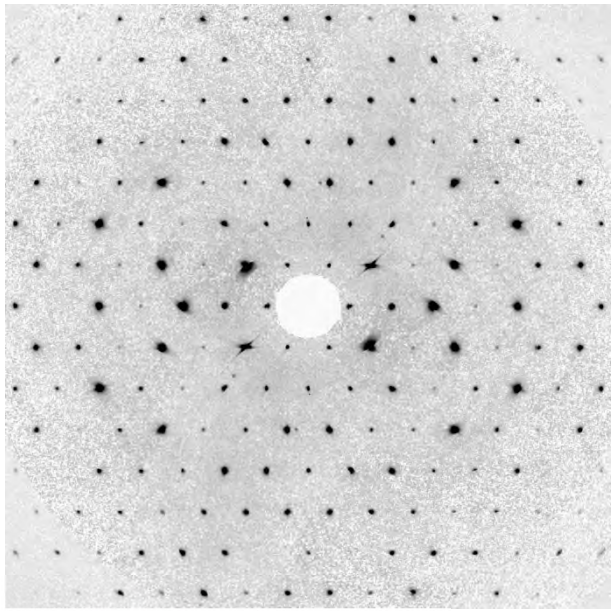


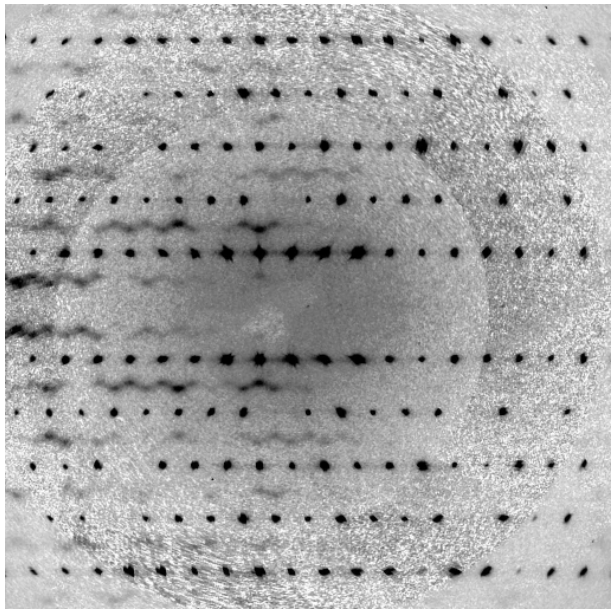
Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

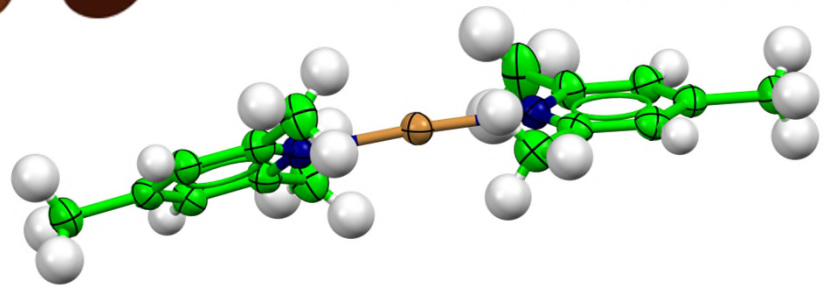
Barluenga's Reagent Derivatives

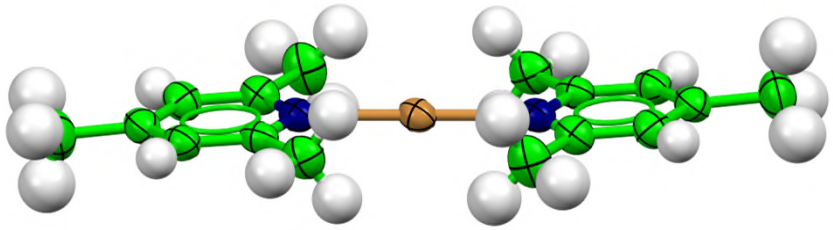
And $Br(Coll)_2BF_4$

	Polymorph I	Polymorph II
	$Br(Coll)_2ClO_4$	$Br(Coll)_2ClO_4$









Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

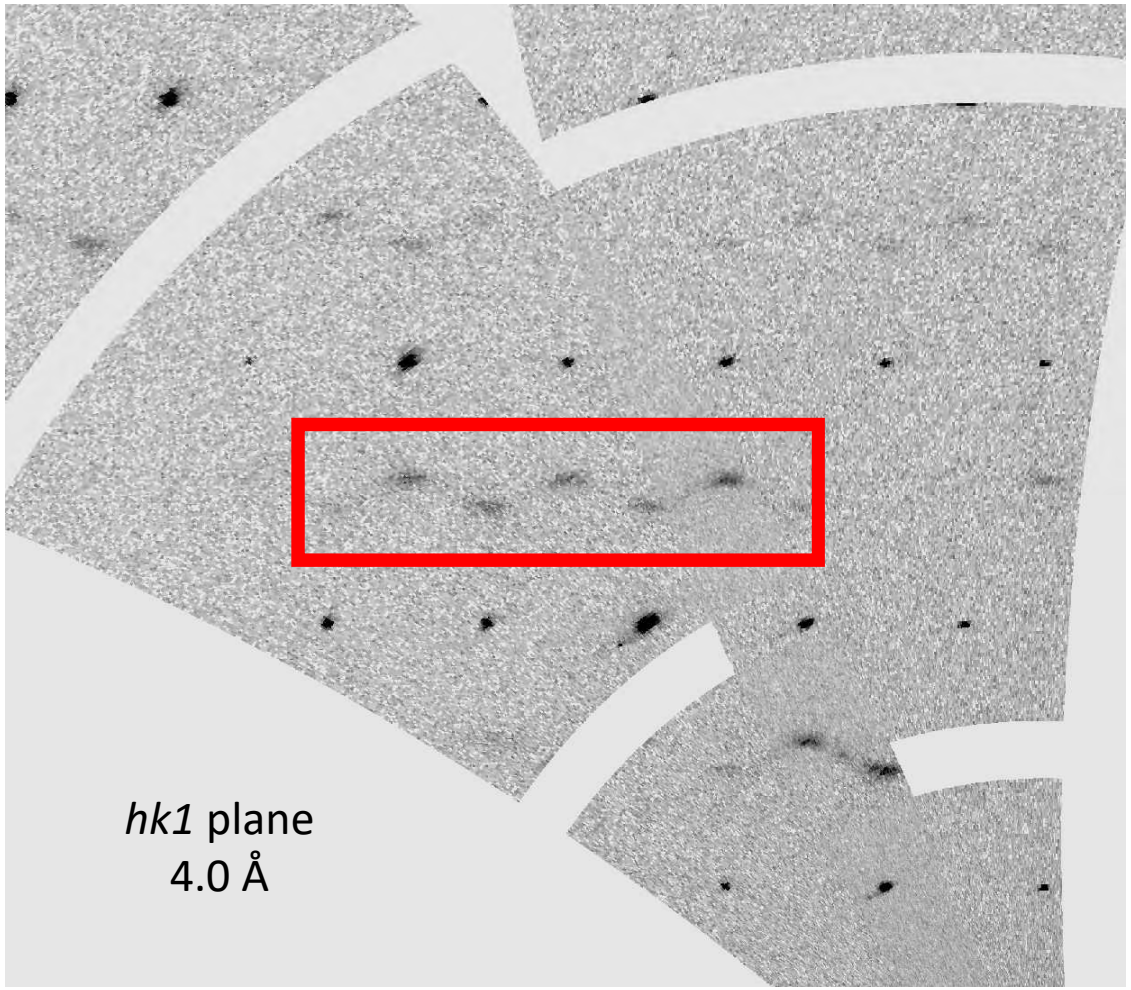
Cooling to 30 K

- Data at 30 K at Diamond
  - No diffuse or satellites
- Crystal at home
  - Diffuse features
- Same crystal at Diamond, at 100 K 24hrs later
  - No diffuse or satellites…

Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

Radiation Damage

- Repeatedly collect same measurement on the same crystal
- 45° phi scan
- 45 seconds of exposure per scan
- Look for change in peaks

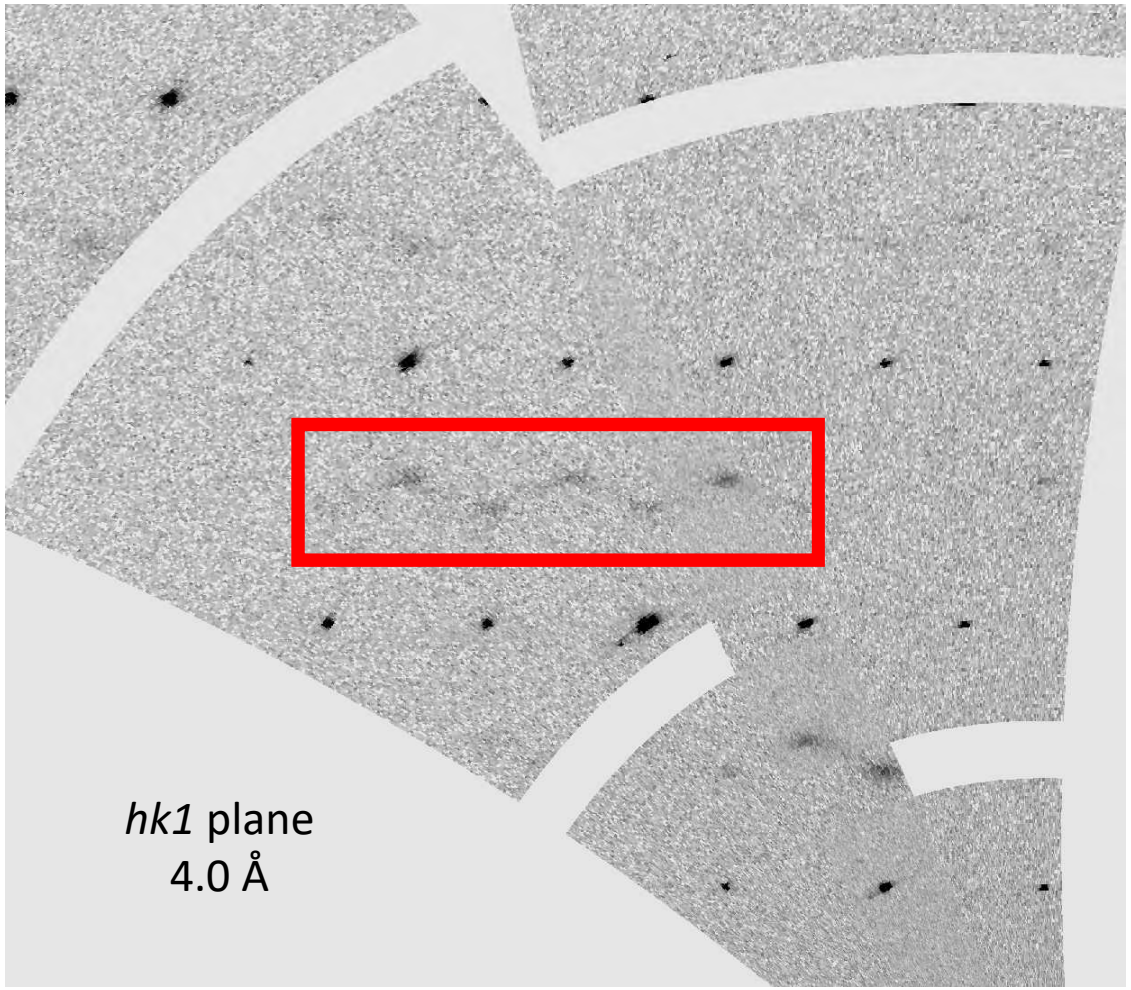


Scan 1

Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

Radiation Damage

- Repeatedly collect same measurement on the same crystal
- 45° phi scan
- 45 seconds of exposure per scan
- Look for change in peaks

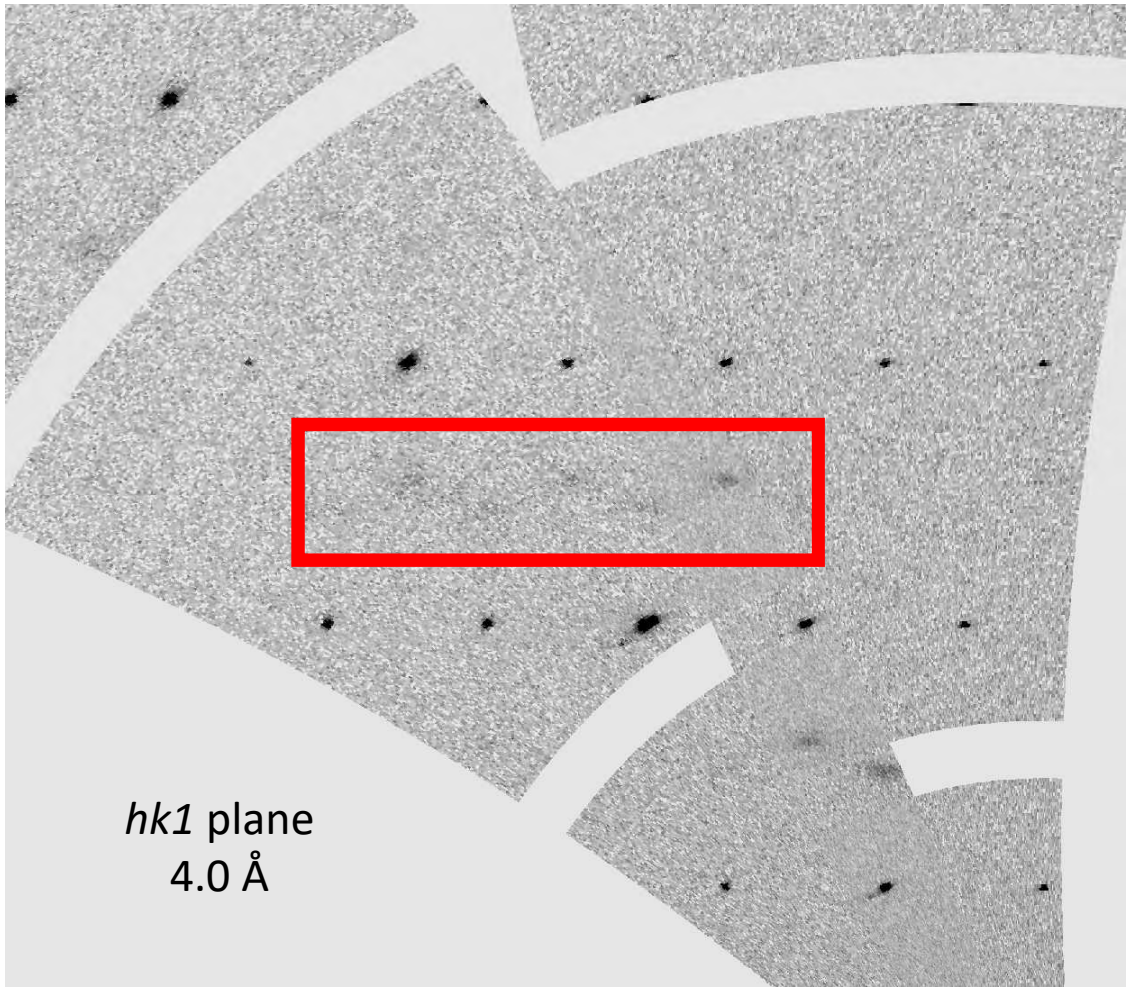


Scan 2

Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

Radiation Damage

- Repeatedly collect same measurement on the same crystal
- 45° phi scan
- 45 seconds of exposure per scan
- Look for change in peaks

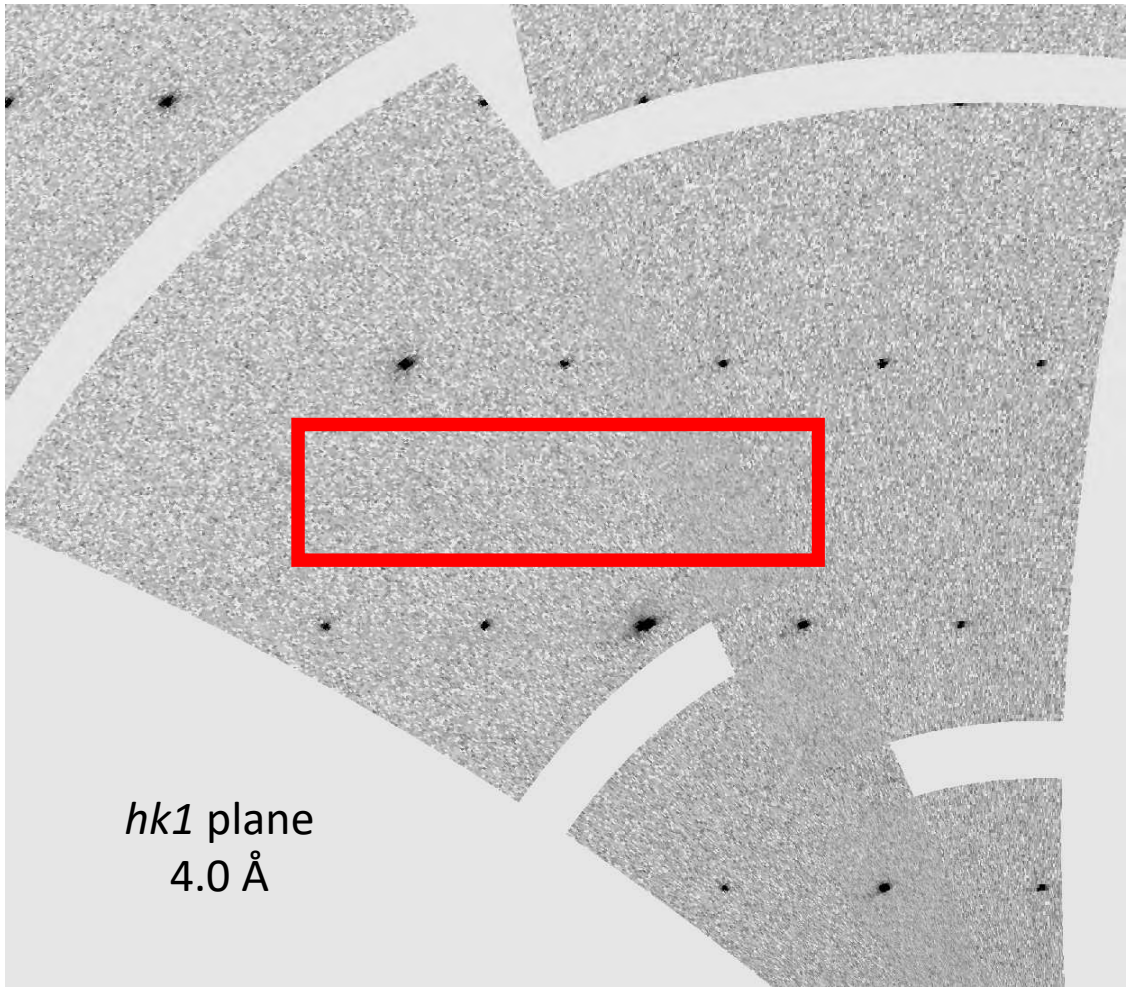


Scan 3

Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.

Radiation Damage

- Repeatedly collect same measurement on the same crystal
- 45° phi scan
- 45 seconds of exposure per scan
- Look for change in peaks

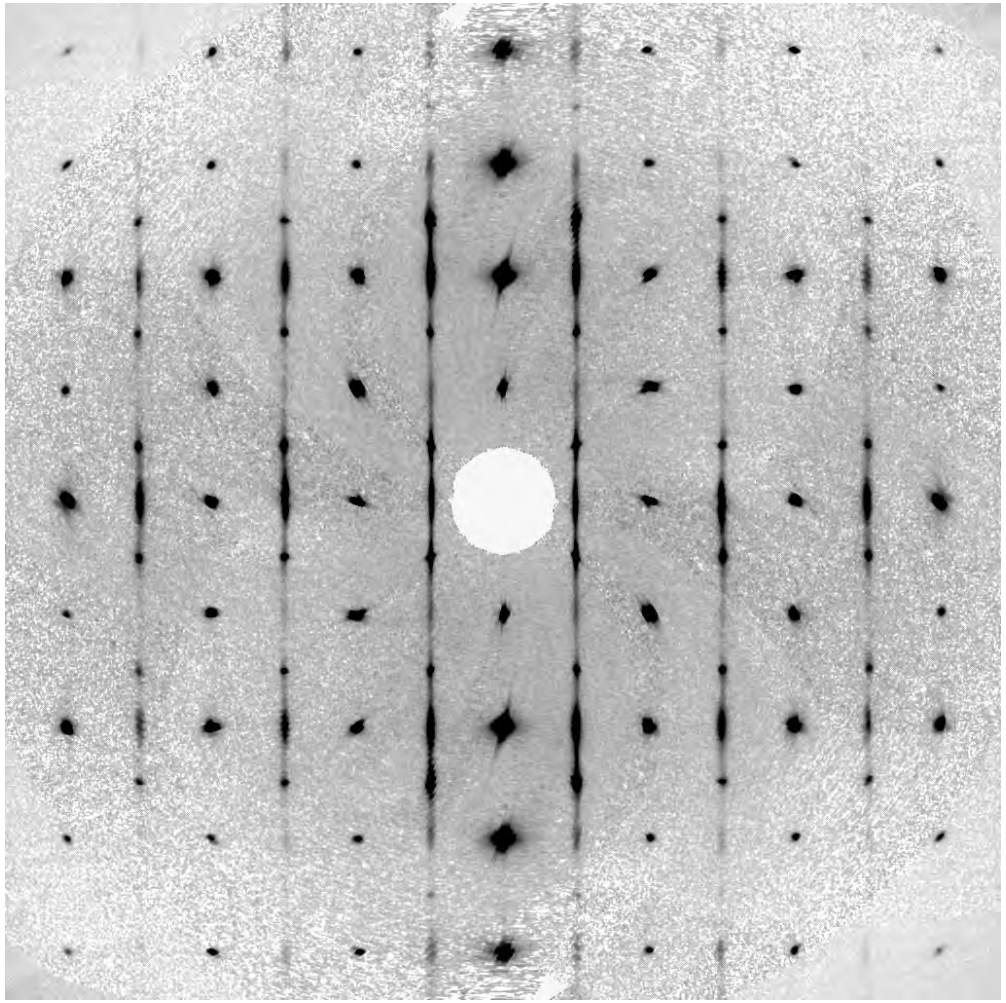


Scan 10

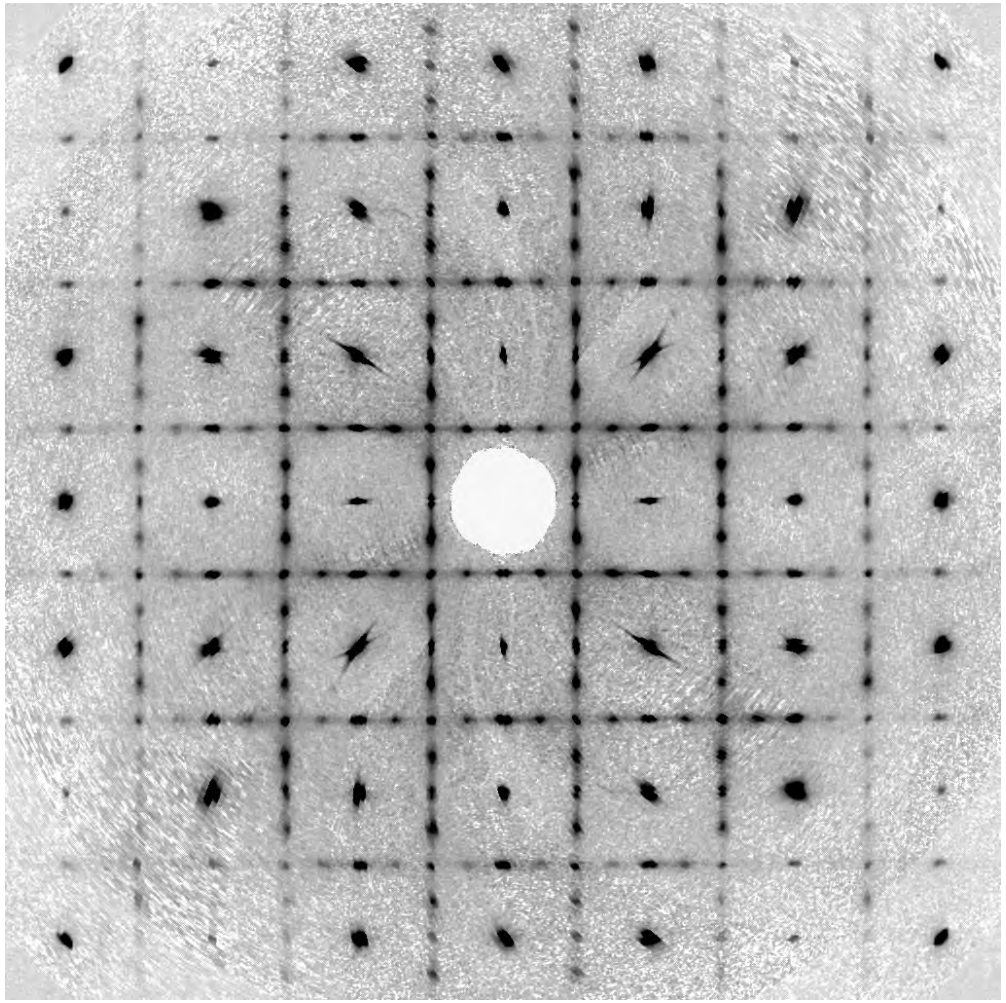
Morgan, Kim, *et al.*, *Chem. Comm.*, 2018, **54**, 9849-9852.


The Silk Purse…?

If the Structure is Known to be wrong…



0kl

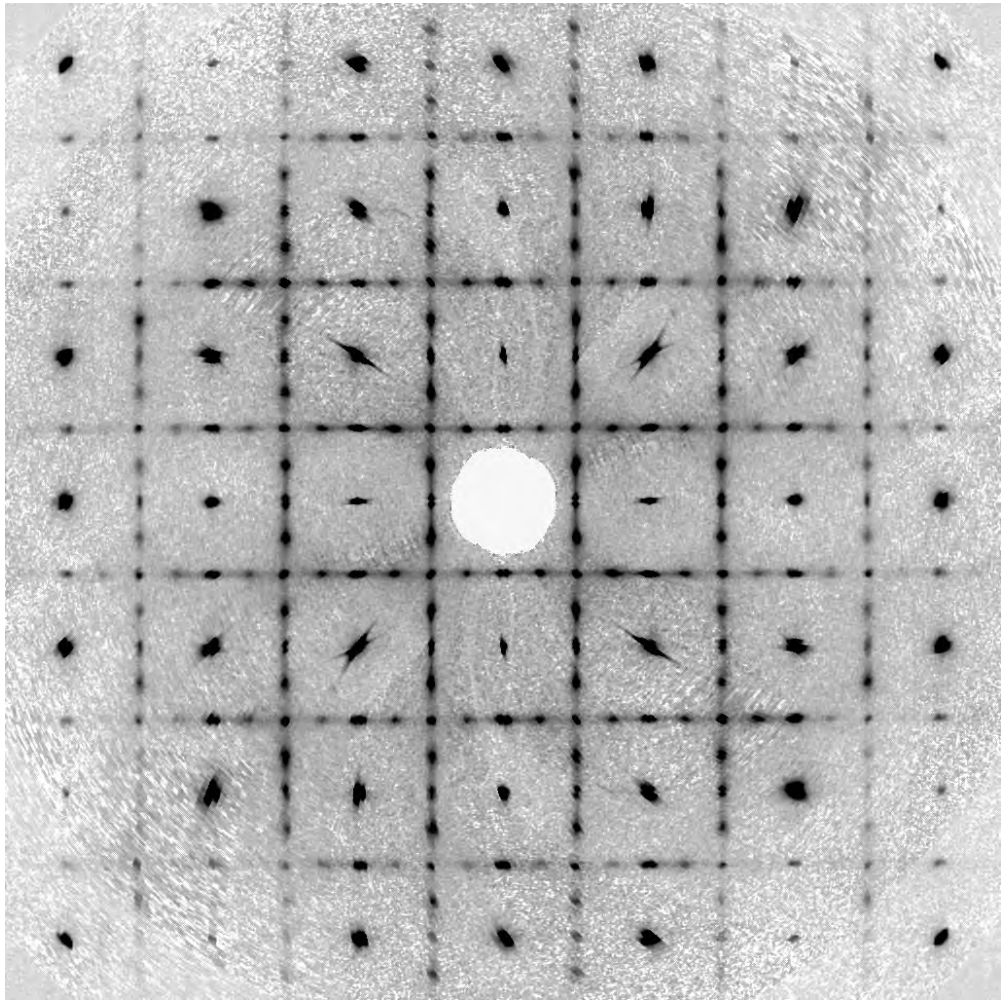


hk0

Fleming, *et al.*, *manuscript in preparation.*

If the Structure is Known to be wrong…

- Diffuse scattering ✓
- Uncertain (pseudo)symmetry ✓
- Twinning ✓
- Modulation (✓)

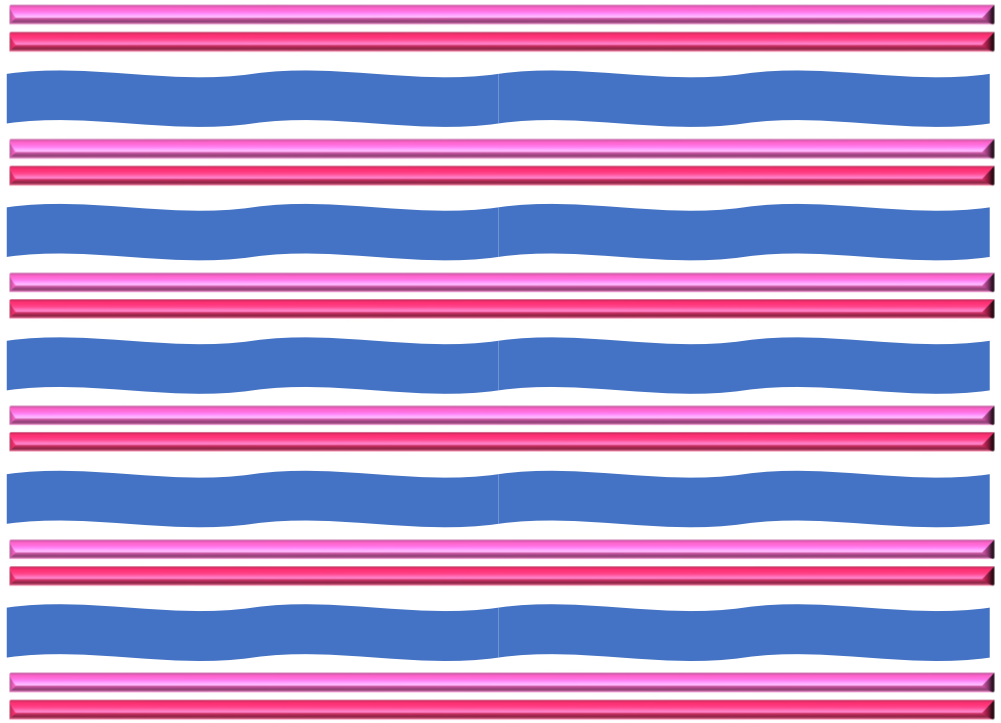


Fleming, *et al.*, *manuscript in preparation.*

If the Structure is Known to be wrong…

- Diffuse scattering ✓
- Uncertain (pseudo)symmetry ✓
- Twinning ✓
- Modulation (✓)

- Still useful…



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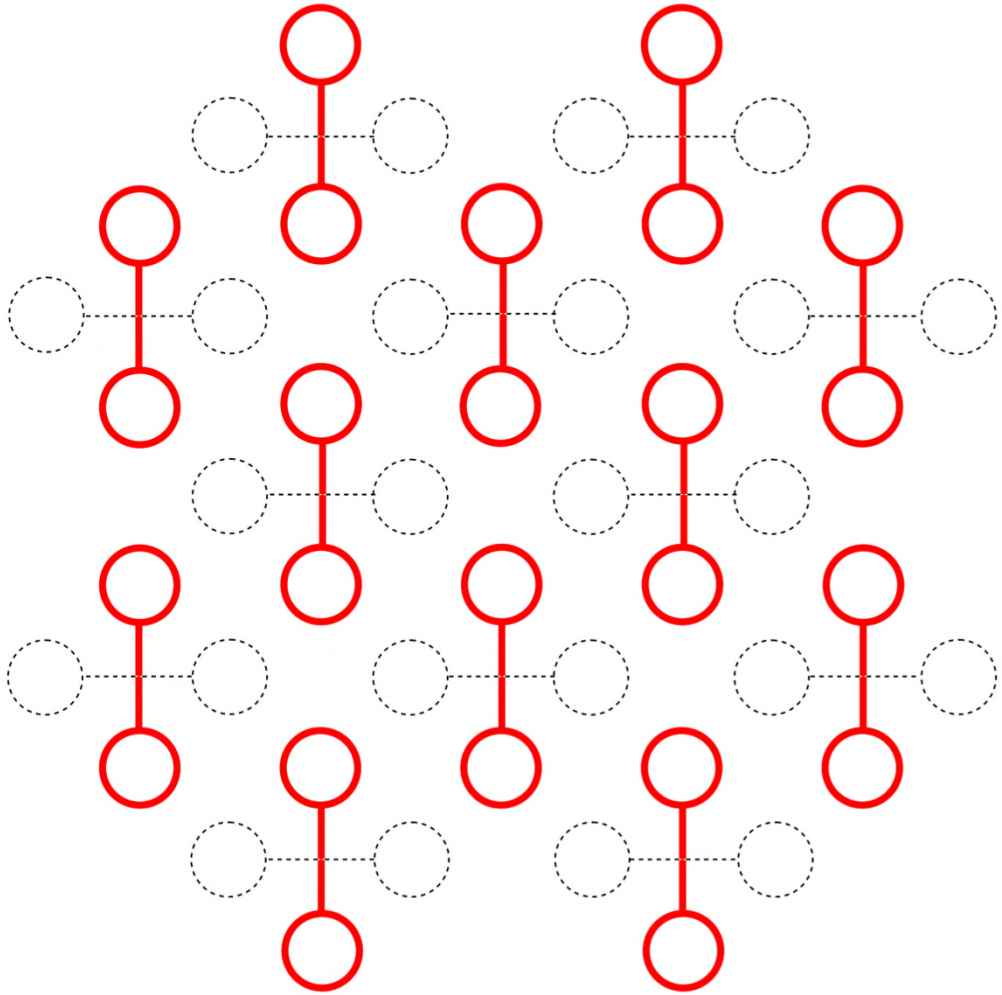
- Still useful…

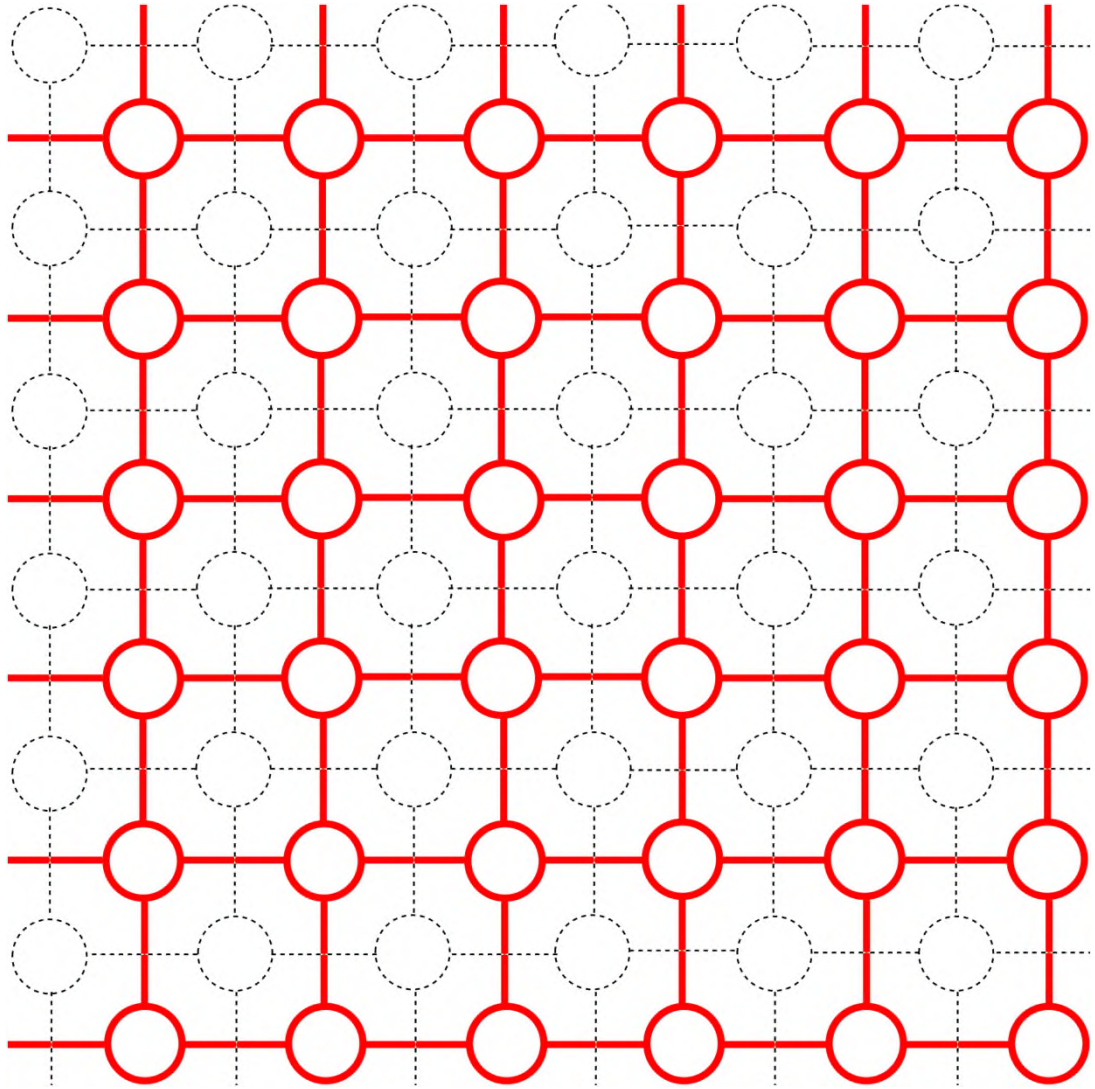


Fleming, *et al.*, *manuscript in preparation.*

If the Structure is Known to be wrong…

- Diffuse scattering ✓
- Uncertain (pseudo)symmetry ✓
- Twinning ✓
- Modulation (✓)

- Still useful…



Fleming, *et al.*, *manuscript in preparation.*

Publishing Difficult Data?

- “Real science” not “just a crystal structure”
- Use the ESI and include
  - Description/Discussion
  - Selected raw images
  - Reconstructed reciprocal lattice sections
- Raw data?
- A crystal structure is just a model…

The Other Little Piggies…

- Kirsten Christensen
  - Emma McKinley
  - Yejin Kim
  - Lewis Morgan
  - Georgia Scurfield
  - Tora Flemming

- Nick Funnel (ISIS)
- Claire Murray (I11, DLS)

- Alasdair French, Justin Chalker & Ben Davis
- Nick Evans, Chris Serpell & Paul Beer
- Nicola Davis & Harry Anderson