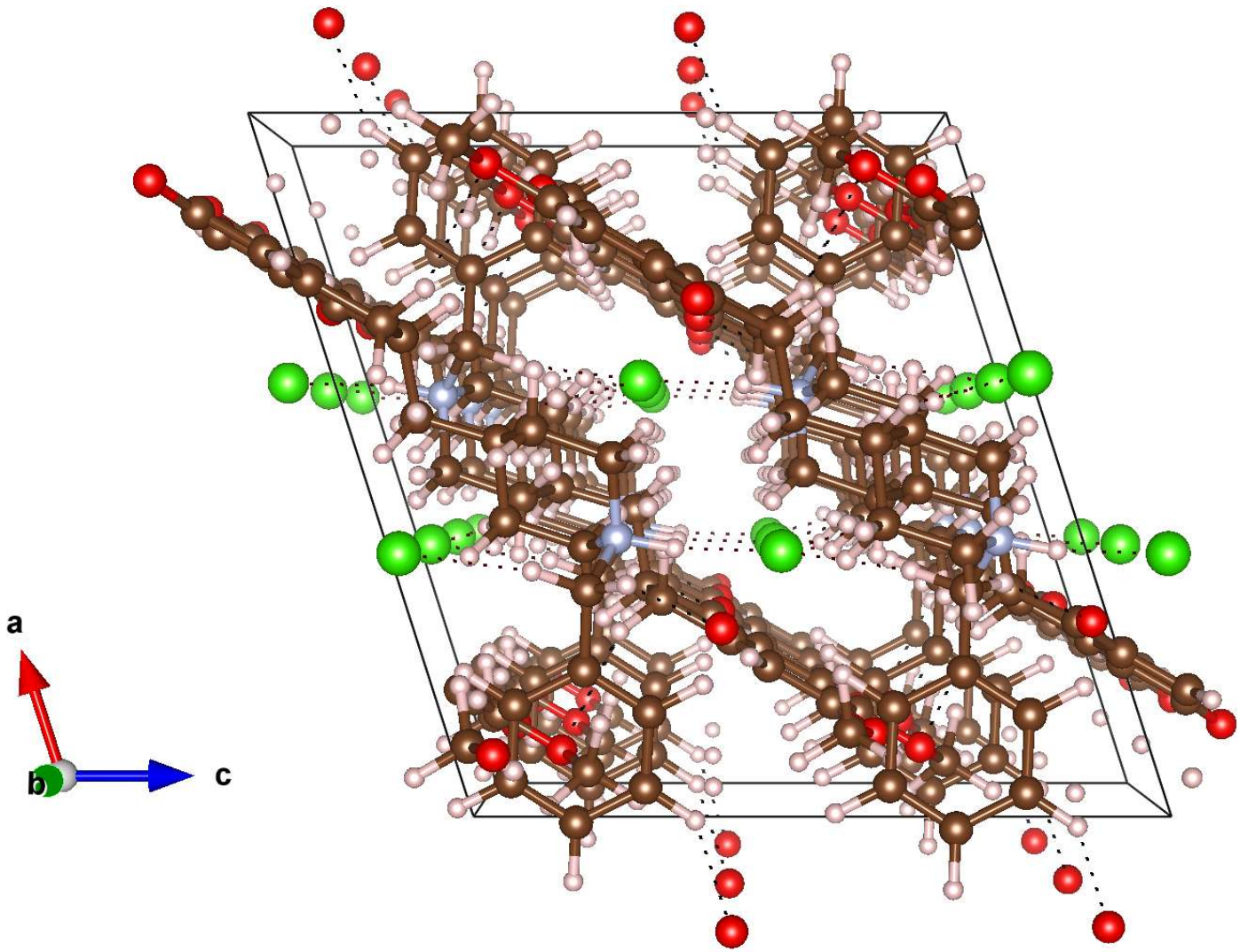
Structure Solution with Powder Diffraction



Joel Reid,
Industrial Scientist,
Canadian Light Source

2026 CLS XRD Summer School

Tuesday, July 14th, 2026

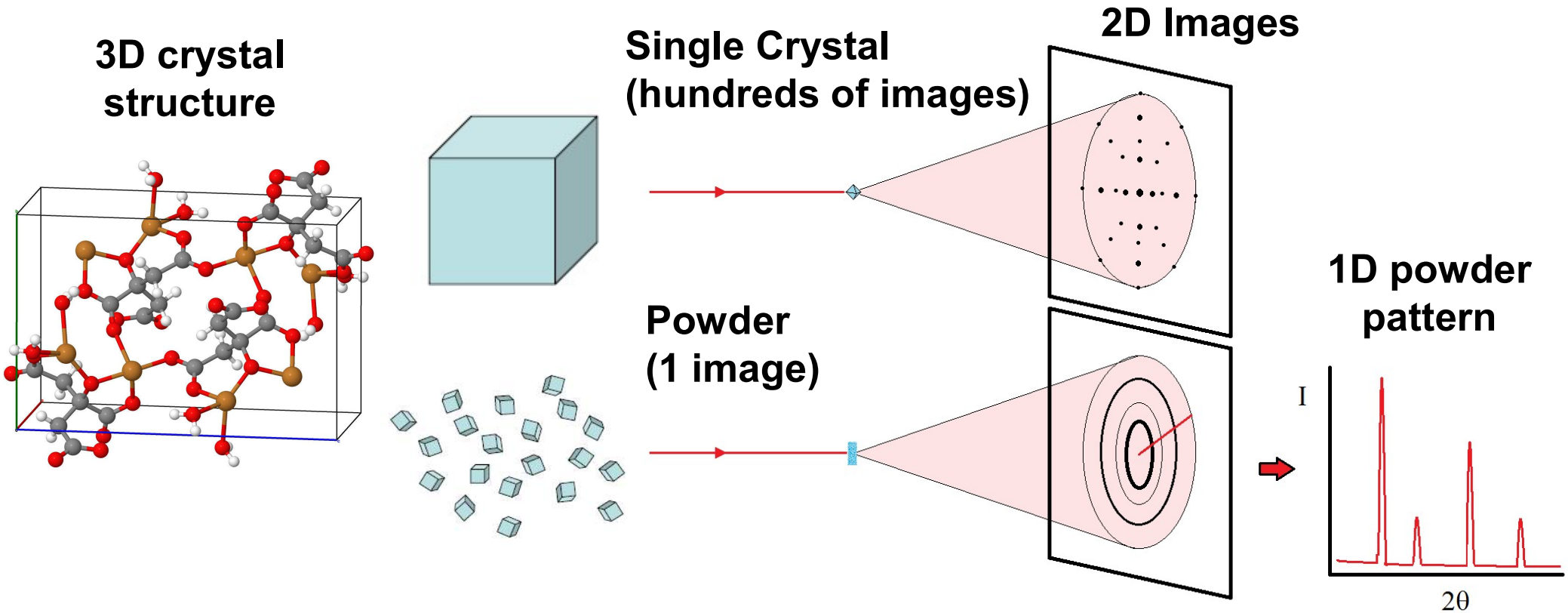
Why Solve Crystal Structures with Powder Data?

- To perform Rietveld refinement, and get the most information possible about your material from your powder data, a reasonable initial crystal structure is required. **What if you don't have a reasonable starting structure?**

- While single crystal XRD is the most used method (by far) for determining a crystal structure, many new functional materials are difficult, if not impossible, to synthesize as large single crystals.

- Powder diffraction structure solution is addictive. If you try it, you may have trouble stopping. But it can also be very challenging…

Why is Powder Structure Solution Challenging?



- Powder diffraction suffers an inherent loss of information, due to the compression of 3D crystalline structure information into 1D.
- Peak overlap means crystal structure solution with PXRD generally requires (1) detailed initial chemical/molecular information, (2) constraints and/or restraints to keep solutions chemically reasonable, and (3) validation with other techniques.

What is Required *Before* Structure Solution?

- Perform thorough search/match phase identification on your powder data with appropriate database(s) like the **Powder Diffraction File** (**PDF-4+**), **Cambridge Structural Database** (**CSD**), American Mineralogist Crystal Structure Database (AMCSD), etc., to make sure no one has already solved the structure. Look for analogous structures, where an element (often a metal) is substituted by a similar element.

- Before trying to solve the crystal structure, synthesize the best possible sample; either phase-pure, or with minimal impurities.

- Based on your chemical synthesis, figure out the probable chemical formula, and confirm it with elemental analysis (XRF, ICP-MS, etc.).

- Get high quality data, using an appropriate radiation source (lab X-rays, synchrotron, neutron) based on the type of sample.

Correlating Pattern Features to Crystal Structure

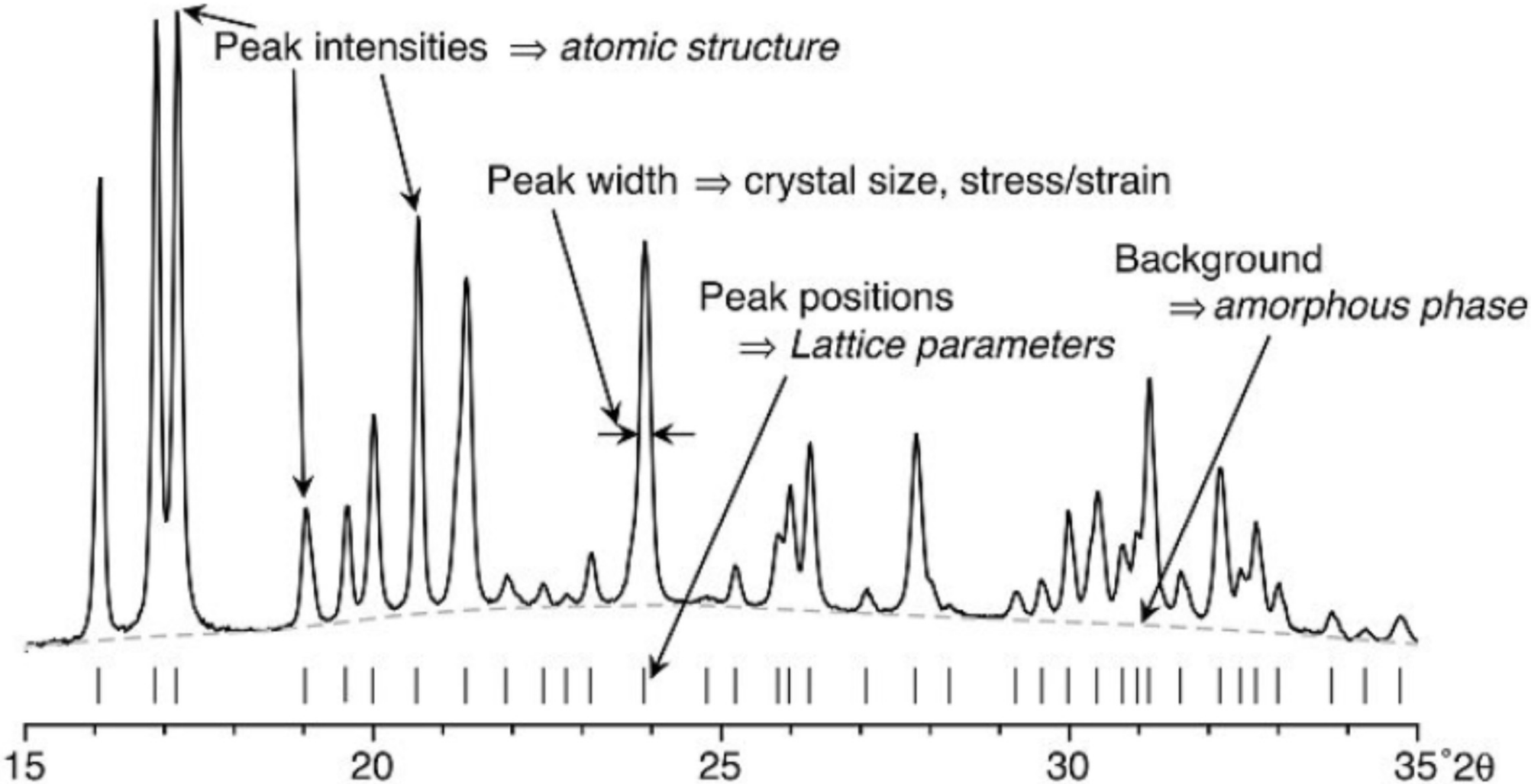


Figure taken from: McCusker, L. B. & Baerlocher, C., Chimia 68 (2014) 19-25

Structure Solution from Powder Diffraction Maze

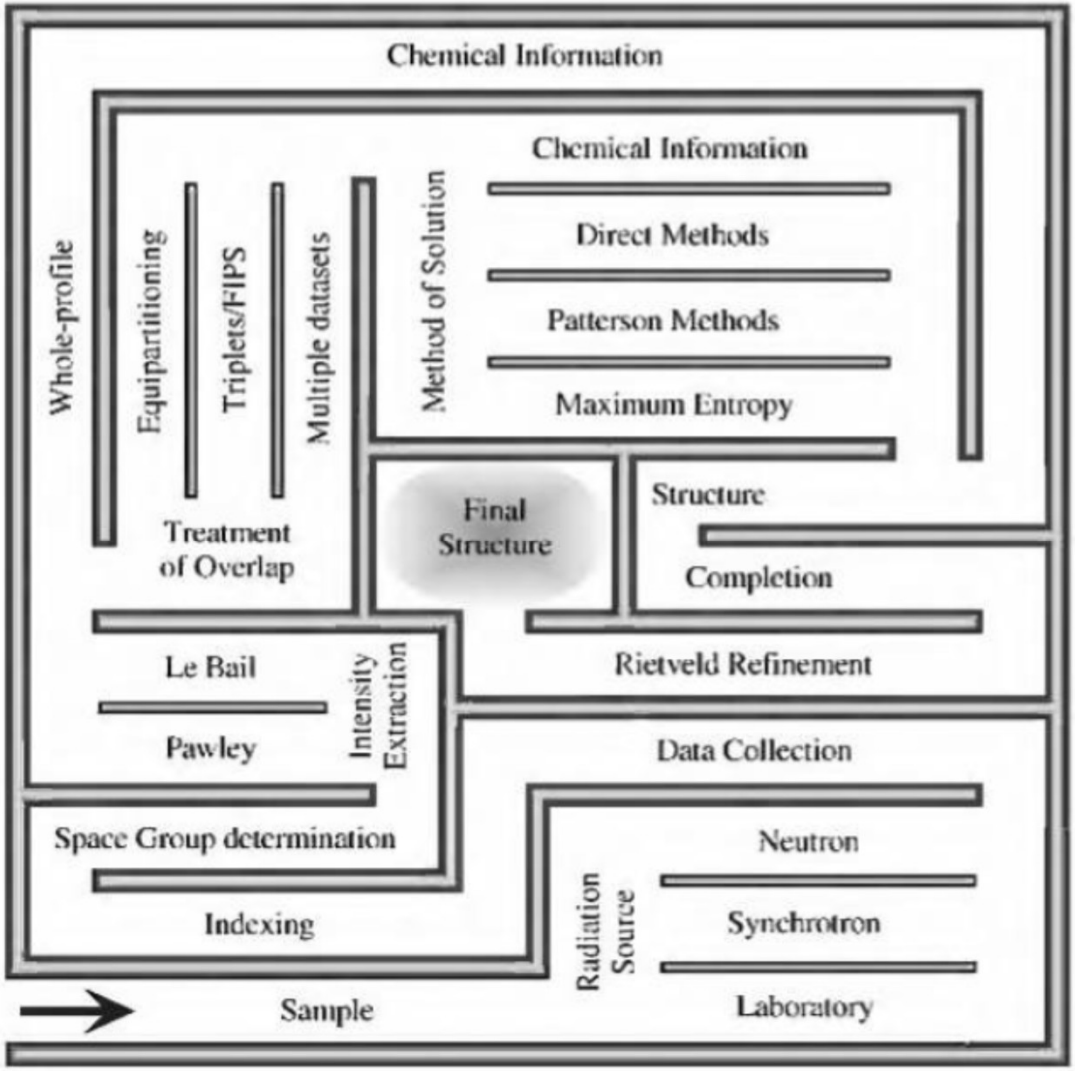


Figure taken from: David, W.I.F. *et al.*, Structure Determination from Powder Diffraction Data. (Oxford: New York, 2002).

Main Steps in PXRD Structure Solution

1. Collect high quality data, with source radiation (lab X-rays, synchrotron, neutron) suitable for the problem.
2. Perform *ab initio* indexing to determine the unit cell of the material. Use systematic reflection absences to determine the space group.
3. Use structure-less Le Bail or Pawley refinements to (i) test unit cells and space groups to confirm the combination providing the best fit, and (ii) to extract intensities for structure solution.
4. Choose an appropriate structure solution route for your material (simulated annealing/parallel tempering, direct methods, charge flipping, etc.) to solve the basic structure.
5. Use Rietveld refinement to refine the structure, while using difference Fourier maps and additional chemical information to complete the structure (often an iterative process).
6. If possible, confirm the final Rietveld-refined crystal structure is correct with density functional theory (DFT) or other experimental techniques.

What Type(s) of Data Should You Use?

Table 6.5 Summary of the issues which need to be addressed in the collection of diffraction data and recommendation of the most appropriate source to use

Issue	Conventional laboratory X-rays[a]	High-resolution laboratory X-rays[b]	Synchrotron X-rays	Low-resolution neutrons[c]	High-resolution neutrons[d]
Intensity at sample	✓	no	✓	no	some third generation instruments
Unit cell determination	fair	✓	✓	poor	✓
Heavy X-ray absorber	short λ reflection geometry	short λ reflection geometry	short λ	✓	✓
Light atom in presence of heavy atom	no	no	no	✓	✓
Hydrogen atoms	no	maybe	maybe	when deuterated	when deuterated
Large unit cell (complex structure)	no	✓	✓	no	✓
Magnetic structure	no	no	no	✓	✓
Thermal parameters	poor	✓	✓	poor	✓
Line-broadened sample	✓	compromised	compromised	✓	compromised
Availability at low cost	✓	✓	competitive access	competitive access	competitive access
Very small sample size	✓	✓	✓	✓	no
Non-ambient environment	maybe	maybe	✓	✓	✓

Table taken from:
Hill, R.J..& Madsen, I.C. in Structure Determination from Powder Diffraction Data. Edited by W. I. F. David, K. Shankland, L. B. McCusker & Ch. Baerlocher. (Oxford: New York, 2002)

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Indexing & Space Group Determination

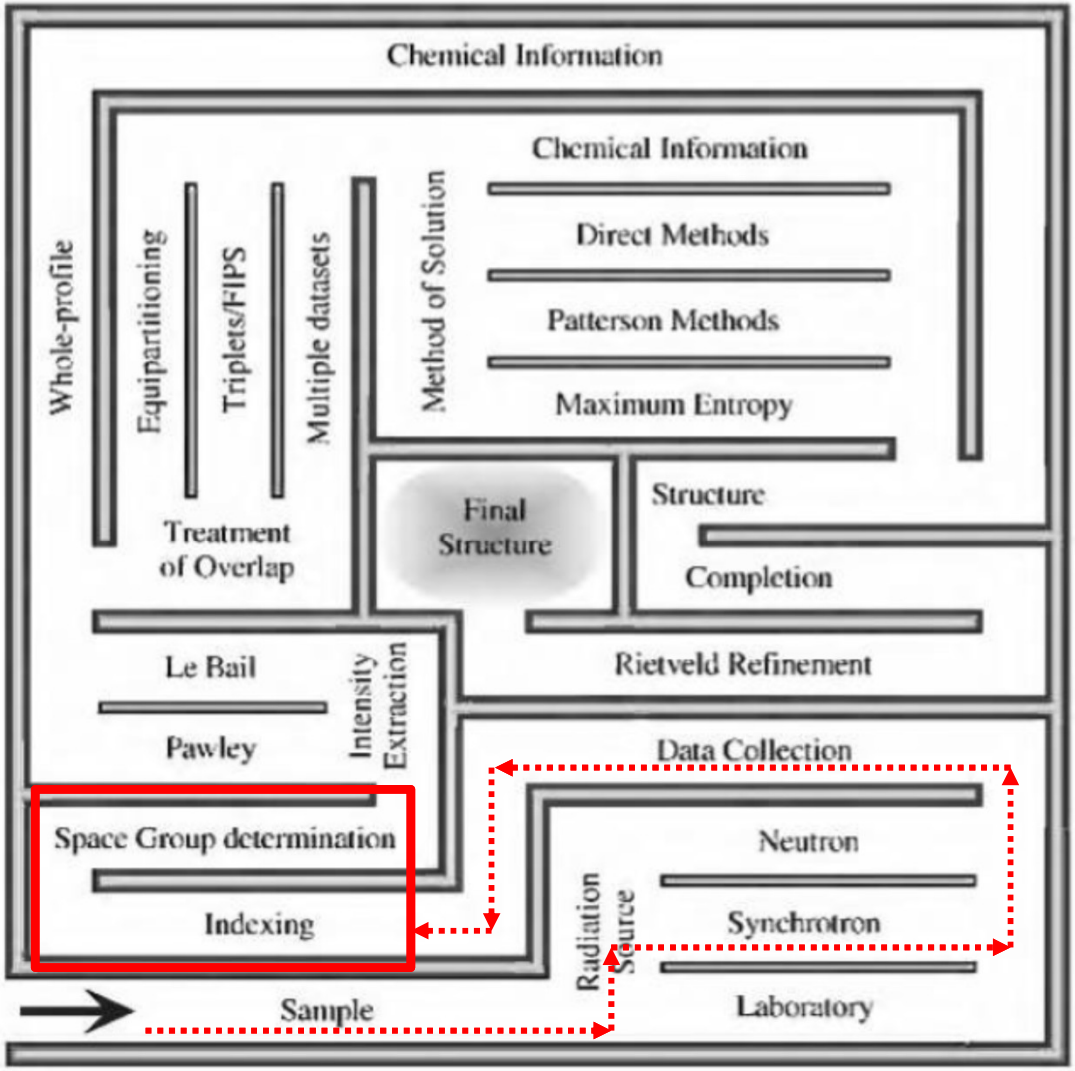


Figure taken from: David, W.I.F. *et al.*, Structure Determination from Powder Diffraction Data. (Oxford: New York, 2002).

Indexing & Space Group Determination

- *Ab initio* indexing is finding the correct unit cell (and space group) description for your crystalline phase.

- Depending on the data quality and size/symmetry of the unit cell, *ab initio* indexing can be the easiest or hardest step.

- There are many programs for indexing powder patterns (**DICVOL**, **TREOR**, **GSAS2**, **McMaille**, **FOX**, ITO, X-Cell, etc.), which use a variety of different algorithms and methodologies.

- Indexing is **always** most straightforward with a single-phase powder pattern. Some indexing programs allow for small numbers of impurity reflections (typically 2-4 peaks maximum in the low angle reflections), but the purer the sample is, the better. If you know peaks are impurity peaks, you should exclude them from indexing.

DICVOL: Boultif, A. & Louer, D.,
J. Appl. Cryst. 37 (2004) 724-731.

Indexing & Space Group Determination

- Most (but not all) indexing programs work optimally using approximately the 20 largest d-spacing (lowest 2θ angle) Bragg reflections.

- Two figures-of-merit (FOM) are typically used for evaluating *ab initio* indexing:

  - The de Wolff FOM:

$$M_{20} = \frac{Q_{20}}{2 \cdot \langle Q \rangle \cdot N_{20}}$$

$$Q_{hkl} = \frac{10^4}{d_{hkl}^2}$$

  - The Smith-Snyder FOM:

$$F_N = \frac{1}{\langle \Delta 2\theta \rangle} \cdot \frac{N}{N(\theta_G)}$$

Werner, P. E. 'Autoindexing',' in: Structure Determination from Powder Diffraction Data. Ed: David, W.I.F. *et al.* (Oxford: New York, 2002).

Indexing & Space Group Determination

- Some programs work better in different circumstances, but it is always helpful to compare results with more than one program.

- A few software platforms facilitate access to multiple indexing programs, like **WinPLOTR/FullProf Suite** (**DICVOL**, **TREOR**, ITO) and **EXPO** (**DICVOL**, **TREOR**, **McMaille**).

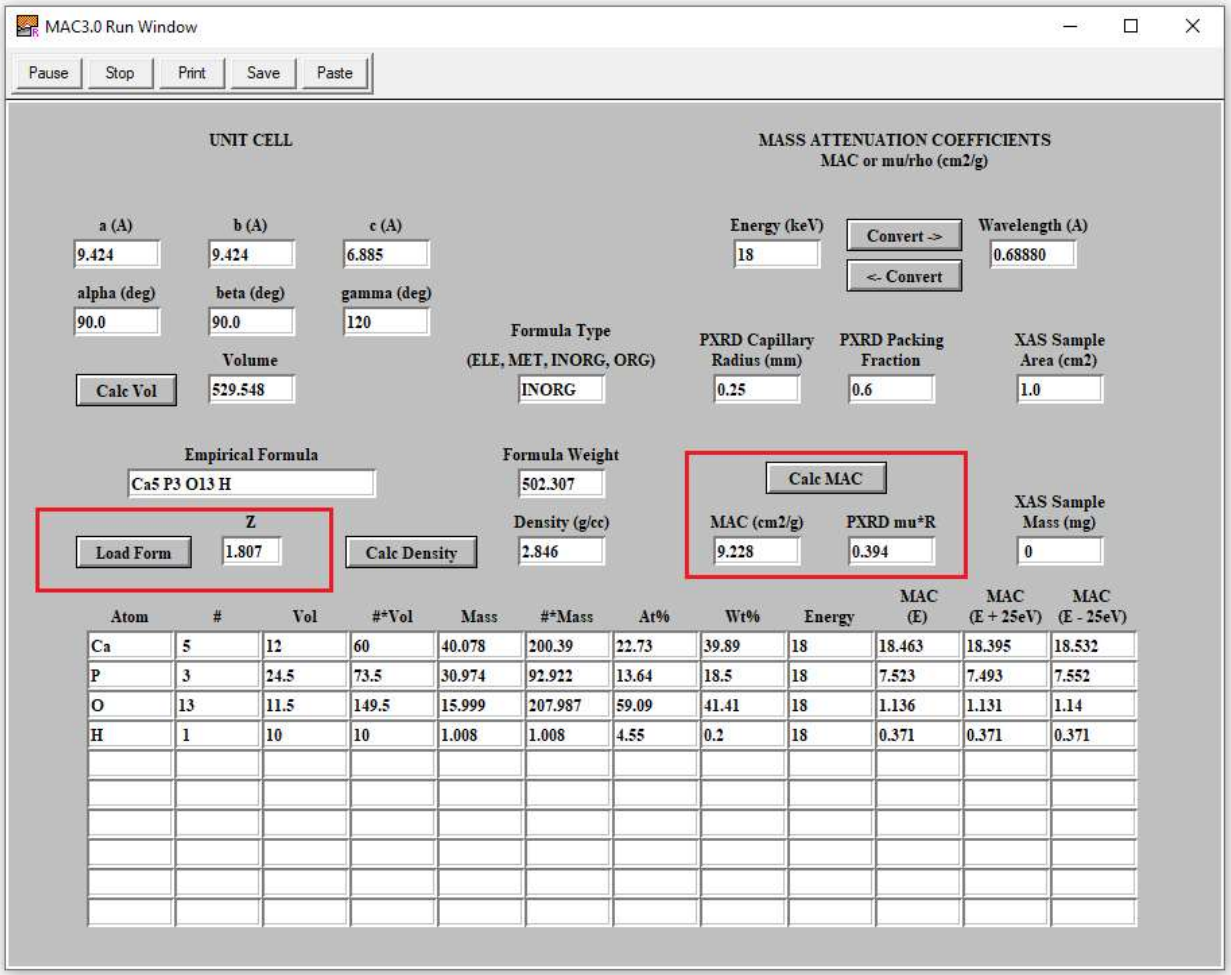
- Once you have a tentative unit cell, a number of programs can examine the observed reflections for systematic absences, to determine the most likely space groups (**ChekCell**, **EXPO**, **GSAS2**, **FOX**, Topas, etc.).

- You always need to make sure your unit cell volume and the general multiplicity of your space group are compatible with the formula. **The cell contents (formula, Z) need to make sense with the unit cell.**

Estimating Cell Contents (Z) from Formula, Volume

- The number of non-hydrogen atoms in the unit cell for an organic compound can be estimated by dividing the volume by 15 to 20 (typically 17 or 18, often called the ‘Rule of 18’).

- Some software packages like **EXPO** have calculators for estimating the unit cell volume based on the formula and Z value.



For a ‘typical’ hexagonal hydroxylapatite cell, Z = 2.

Le Bail & Pawley Refinements

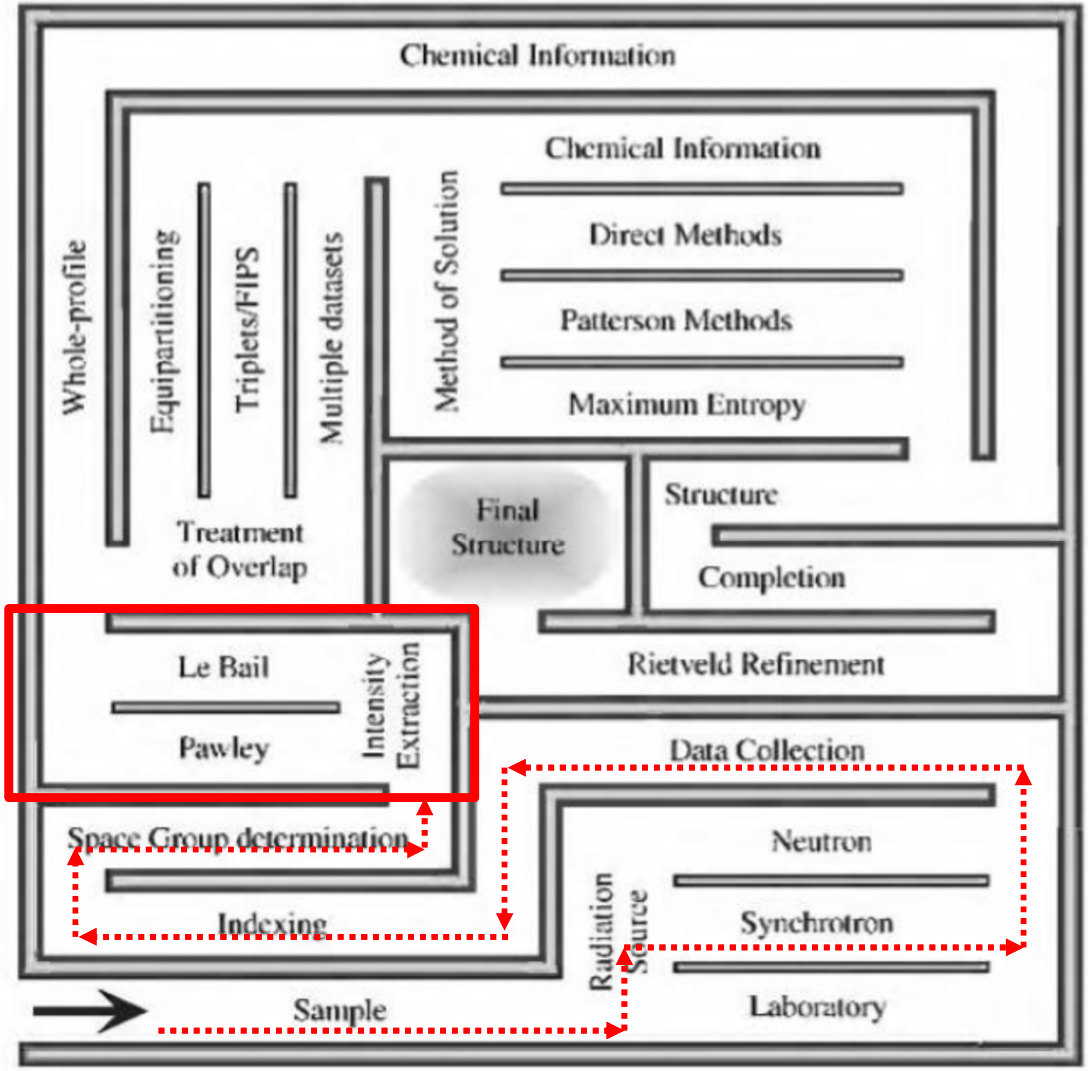


Figure taken from: David, W.I.F. *et al.*, Structure Determination from Powder Diffraction Data. (Oxford: New York, 2002).

Le Bail & Pawley Refinements

- The Le Bail and Pawley methods were developed to allow full profile fitting of powder patterns using (1) a unit cell (with space group) to define the peak positions and (2) the peak shape/width parameters, **but without requiring a crystal structure.**

- The peak intensities are optimized to best fit the experimental data without any structural model constraints (i.e. atomic coordinates).

- These methods are critical for getting estimates of the integrated reflection intensities (or structure factor amplitudes, $|F_{hkl}|$) from powder patterns for the solution of unknown crystal structures.

Pawley, G. S.
J. Appl. Cryst. 14 (1981) 357-361.

Le Bail, A, Duroy, H. & Fourquet, J. L.
Mater. Res. Bull. 23 (1988) 447-452.

Le Bail & Pawley Refinements

- The Pawley method treats individual reflection intensities as least-squares parameters to be refined (resulting in a very large least-squares matrix for large, low symmetry unit cells).

- The Le Bail method uses an iterative profile intensity partitioning method to estimate the calculated intensities from the observed pattern.

- The Le Bail method is more similar mathematically to the Rietveld method, therefore it has been more widely incorporated in Rietveld programs than the Pawley method. But one or both methods are available in all major Rietveld programs (**GSAS2**, **FullProf**, **JANA**, Topas etc.).

Pawley, G. S.
J. Appl. Cryst. 14 (1981) 357-361.

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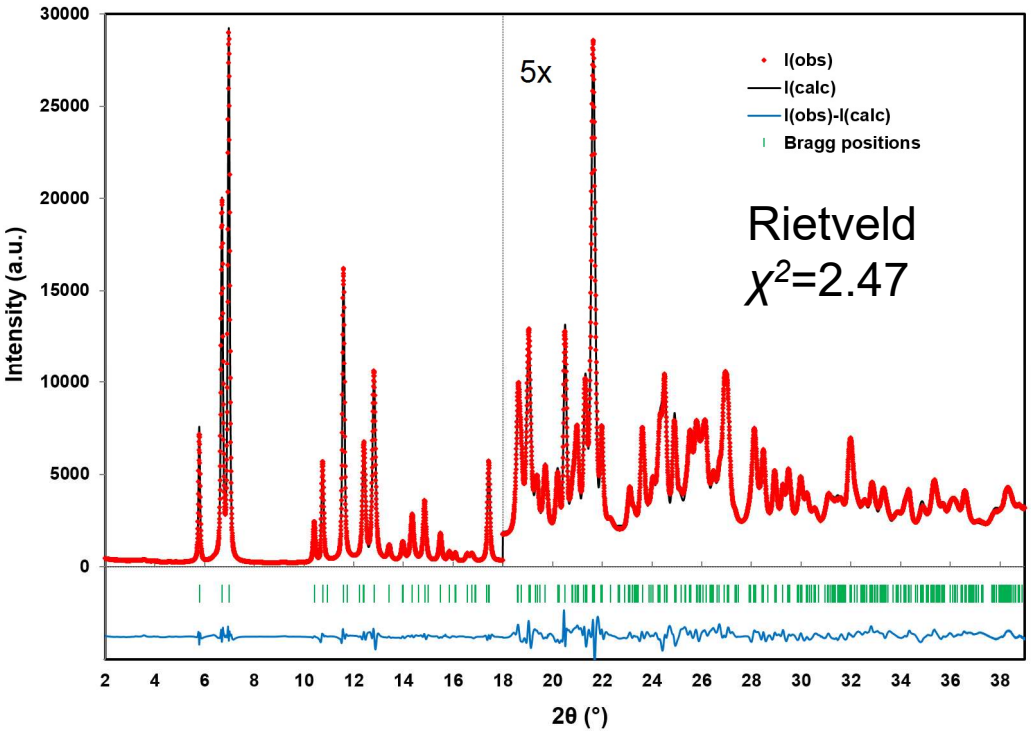
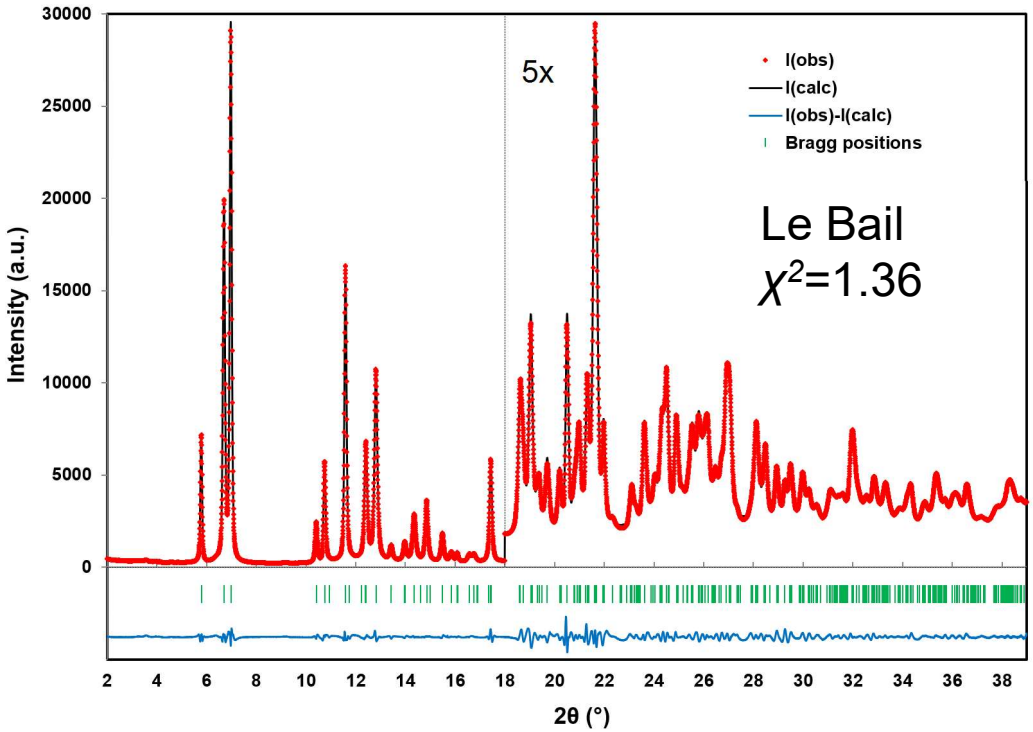
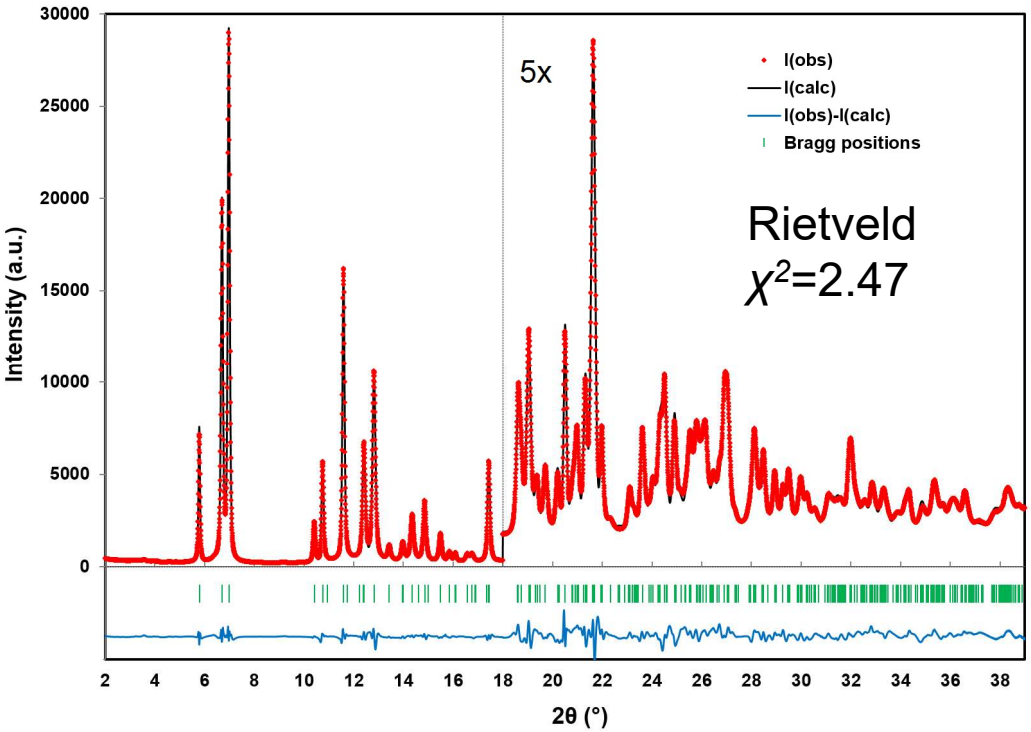
Comparison of Rietveld & Le Bail Refinements

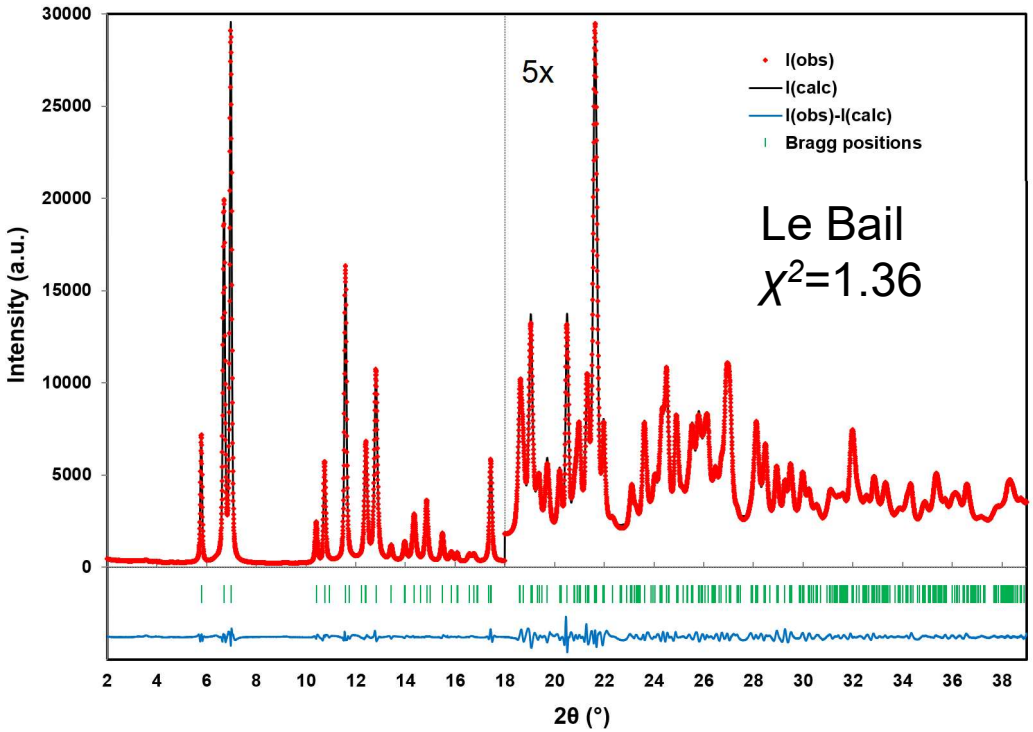


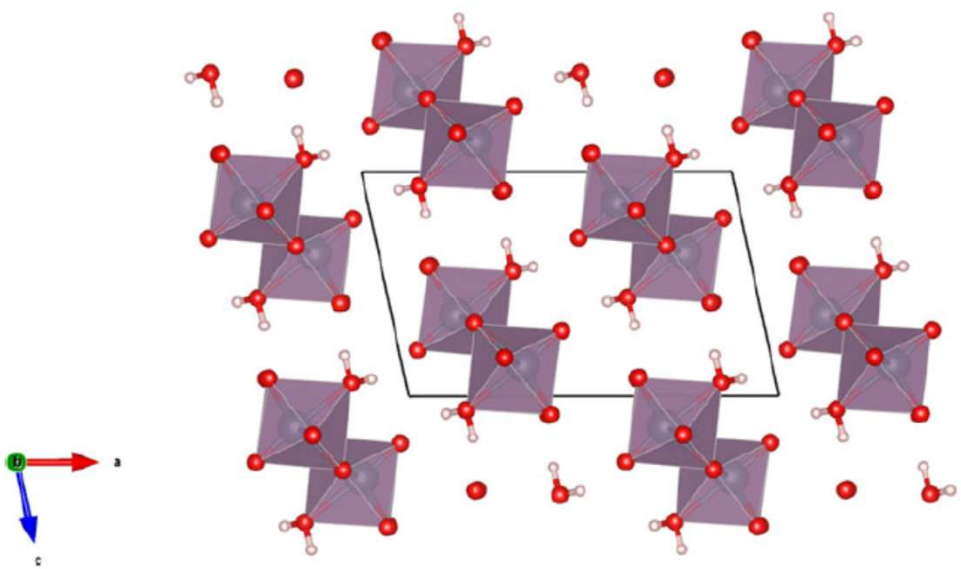


- The Le Bail refinement sets a lower limit on the achievable reduced chi-squared (χ^2), and provides a reference for how good your Rietveld refinement is.
- This can be helpful for deciding if your final refinement and structural model are reasonable.

Comparison of Rietveld & Le Bail Refinements







$MoO_2(O_2)H_2O$

Reid, J. W., Kaduk, J. A. & Matei, L.
Powder Diffraction 33 (2018) 49-54.

Crystal Structure Solution

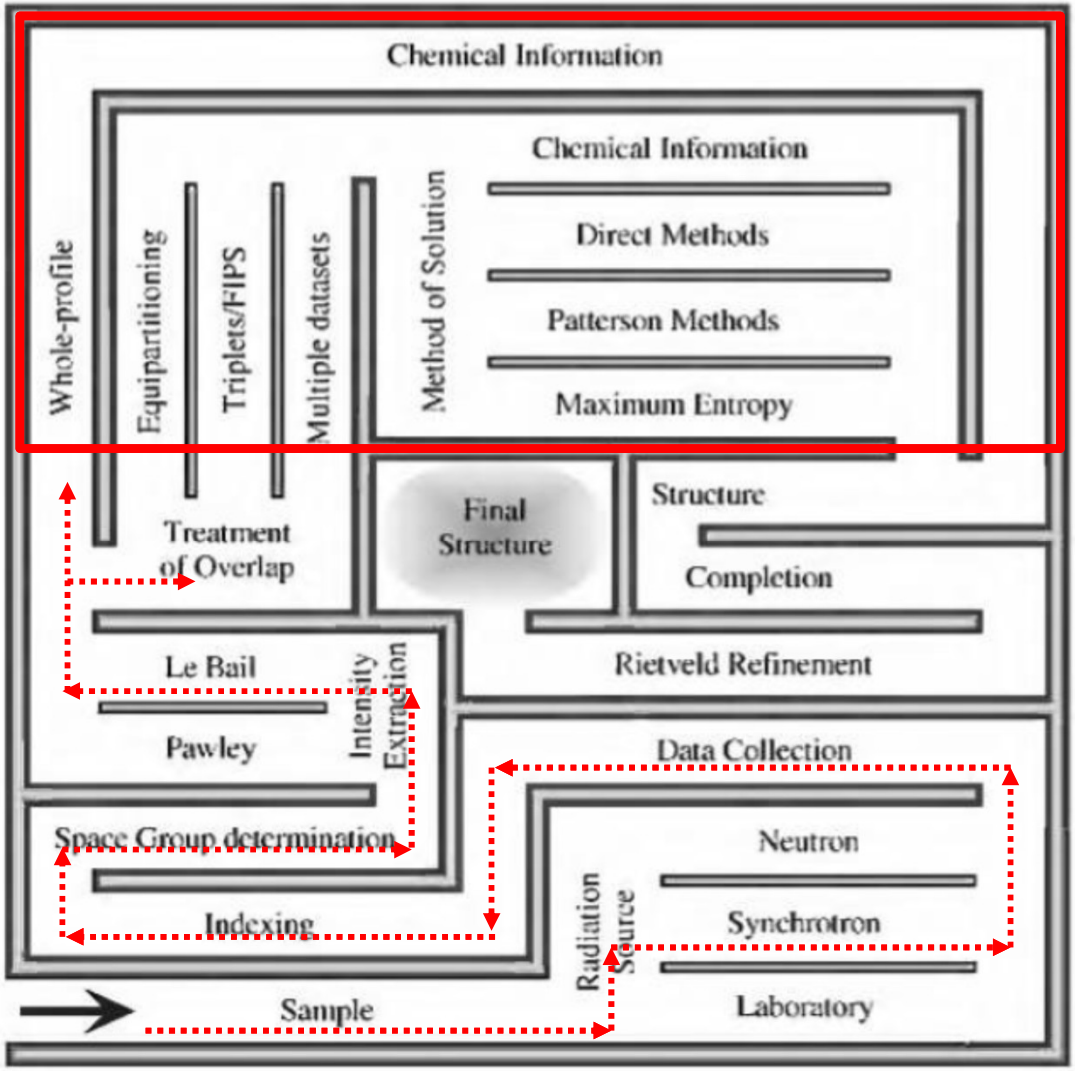
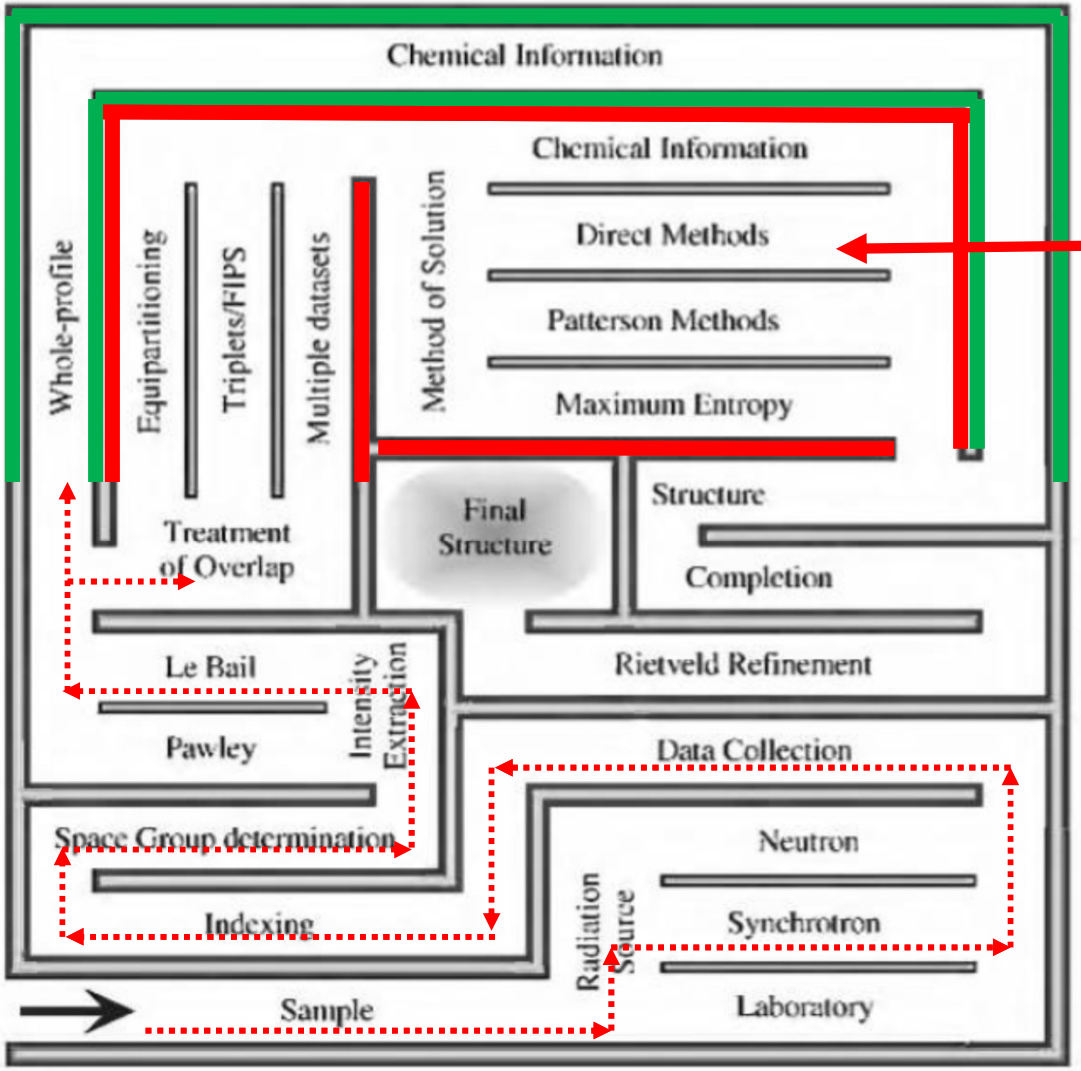


Figure taken from: David, W.I.F. *et al.*, Structure Determination from Powder Diffraction Data. (Oxford: New York, 2002).

Crystal Structure Solution

Direct-Space (DS) or 'Real-Space' Methods



Reciprocal-Space (RS) Methods

Figure taken from: David, W.I.F. *et al.*, Structure Determination from Powder Diffraction Data. (Oxford: New York, 2002).

Crystal Structure Solution

- Crystal structure solution strategies for powder data can generally be divided into two types of methods:

1. Reciprocal-space methods:
   - Direct methods/Patterson methods (**EXPO**, Topas, XLENS)
   - Charge flipping (**GSAS2**, **SUPERFLIP**, JANA2006)
   - Maximum entropy methods (MICE)

2. Direct-space (real-space) methods:
   - Simulated annealing (**GSAS2**, DASH, Topas, PSSP, FOX)
   - Parallel tempering (**FOX**)
   - Monte Carlo and hybrid methods (ESPOIR, POWDERSOLVE)

Structure solution review article: Meden, A. & Radosavljevic Evans, I., Cryst. Res. Technol. 50 (2015) 747-758.

Reciprocal-Space Methods

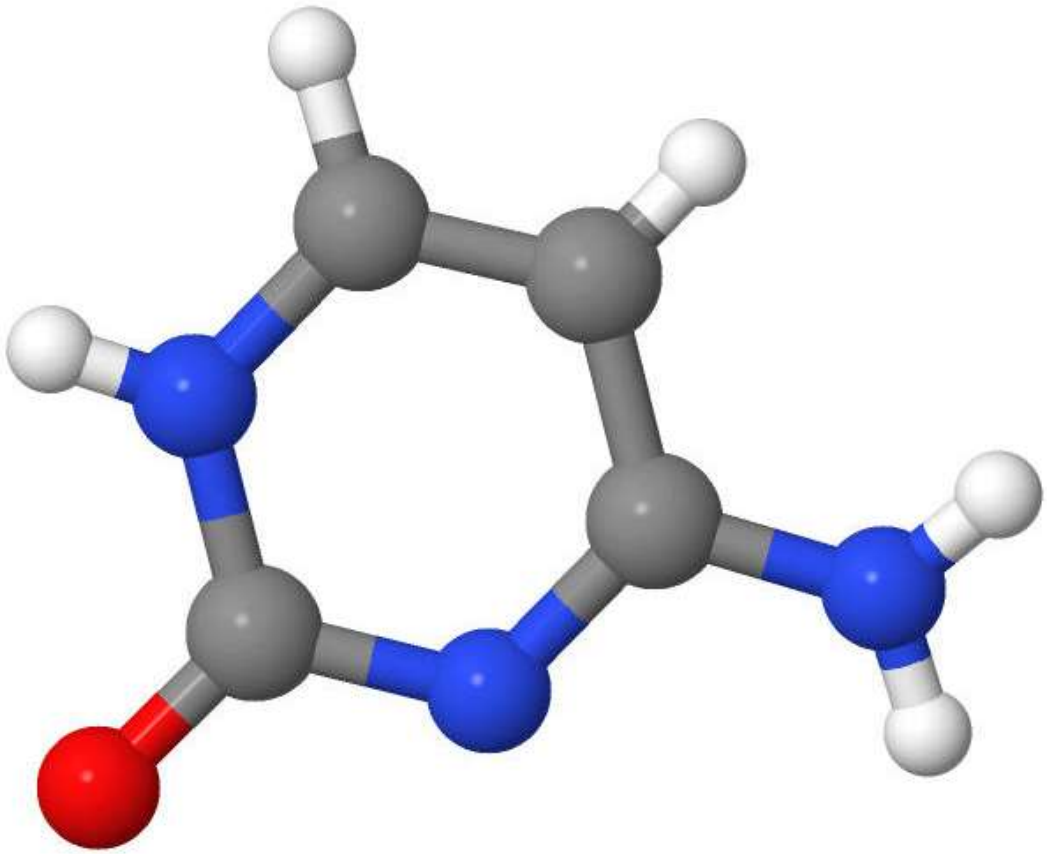
- Reciprocal-space (RS) methods are based on single-crystal structure solution methods, which over time have been optimized for powder data, and use individual Bragg reflection integrated intensities ($|F_{hkl}|$) extracted from the full PXRD pattern.

- The key to success with these methods is getting a sufficient number of accurate (non-overlapped) intensities with data content out to atomic resolution (d = ~1 Å). These methods tend to need minimal to moderate reflection overlap, which makes them more successful for smaller, higher symmetry unit cells (and higher resolution data).

- RS methods generally require minimal *a priori* chemical information to find a basic solution, but you still want to know the chemical information, if possible, to confirm (or adjust) the fine details of the solution.

Indexing and Solution Demonstration: EXPO2014

- Indexing synchrotron powder data (E = 18 keV, λ = 0.68908 Å) for cytosine ($C_4H_5N_3O$) using **DICVOL**, via **EXPO2014**, and solution with direct methods **(EXPO2014**).



EXPO: Altomare, A. *et al.*,
J. Appl. Cryst. 46 (2013) 1231-1235.

Direct-Space Structure Solution Methods

- Direct-space (DS) methods tend to use the whole pattern, rather than extracted integrated intensities. They follow a global optimization strategy, where molecules or multiple structural fragments are moved around the unit cell in 'random walk' processes.

- The entire powder pattern is calculated at every step, to compare with the observed pattern, and the best fit(s) are preserved.

- DS methods tend to be less dependent on resolution (peak breadth) and data content (minimum d-spacing) than reciprocal-space methods, because they use the entire pattern.

- But to use DS methods, **you need to have reasonable chemical information like the molecule(s) present in the structure (organics) or the approximate cell contents and polyhedral fragments (inorganics).**

DS review: Cerny, R. & Farve-Nicolin, V., Z. Kristallogr. 222 (2007) 747-758.

Considerations: Symmetry, Cell Volume & Content

- You always need to make sure your unit cell volume and the general multiplicity of your space group are compatible with the formula. **The cell contents (formula, Z) need to make sense with the unit cell.**

- Consider the mineral gypsum ($CaSO_4 \cdot 2H_2O$):
  - Unit Cell: a = 6.284 Å, b = 15.200 Å, c = 6.523 Å, β = 127.41°, V = 494.90 $Å^3$
  - Formula: $Ca(SO_4) \cdot 2H_2O$, **Z = 4** → **Total cell contents: 4 Ca, 4 SO_4, 8 H_2O**
  - Space group: **C2/c (#15)** → **The general multiplicity of the space group is 8.**

- All our component contents (Ca, SO_4, H_2O) are either half the general multiplicity (4 Ca, 4 SO_4) or equal to the general multiplicity (8 H_2O) . Therefore we probably only need 1 Ca atom, 1 SO_4 tetrahedron, and one H_2O to completely describe the structure. Since Ca and SO_4 are half the general multiplicity, they must be located on special positions with reduced multiplicity.

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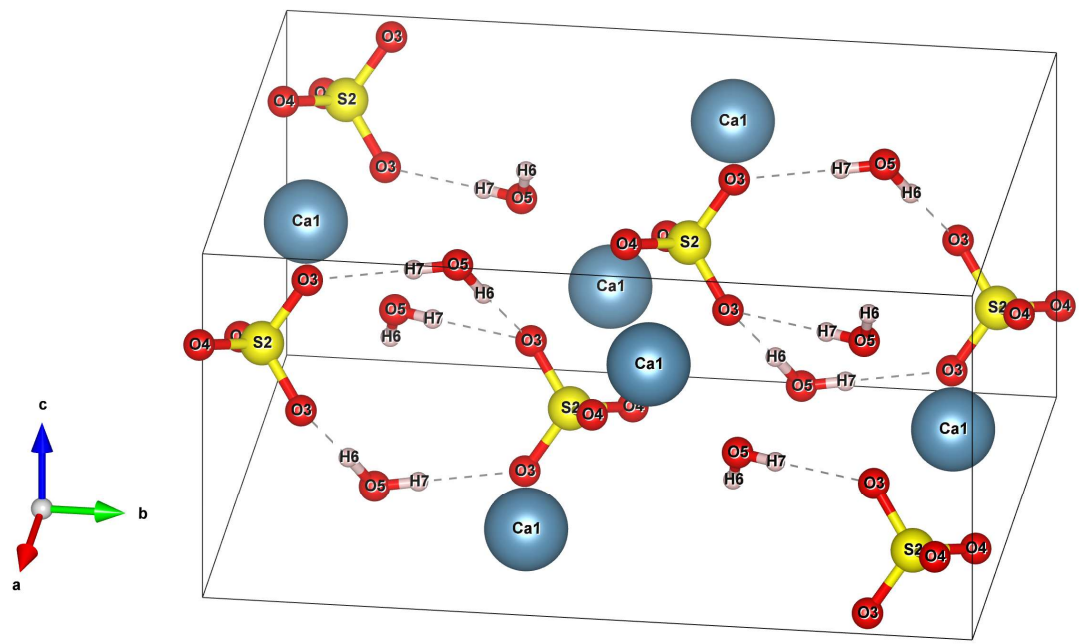
Label	Atom	x/a	y/b	z/c	Mult.	Wyckoff
Ca1	Ca	0.5	0.0796	0.75	4	4e
S2	S	0	0.0772	0.75	4	4e
O3	O	0.0372	0.1320	0.5872	8	8f
O4	O	0.2424	0.0221	0.9092	8	8f
O5	O	0.6202	0.1820	0.0784	8	8f
H6	H	0.7490	0.1620	0.2510	8	8f
H7	H	0.5840	0.2350	0.0730	8	8f

Special position (site multiplicity of 4)
General position (site multiplicity of 8)



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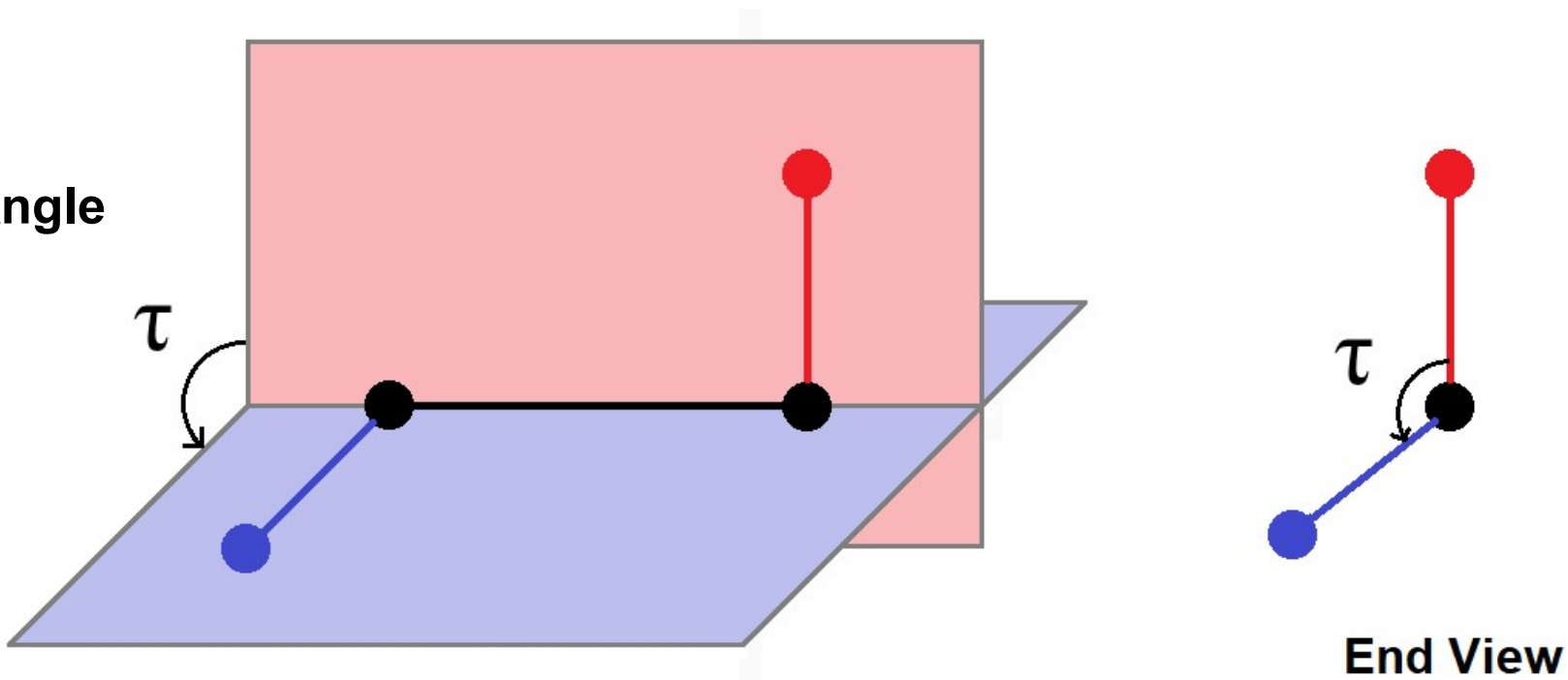
Considerations: Model Size

- The difficulty of a structure solution with powder data depends on (among other things) the **number of atoms** and the **number of degrees of freedom (DoF)** in the model.
- A single atom has 3 DoF (3 translations).
- A rigid body molecule has 6 DoF (3 translations, 3 rotations).
- Molecules with *n* torsion angles generally have (6+*n)* DoF.

Torsion Angle



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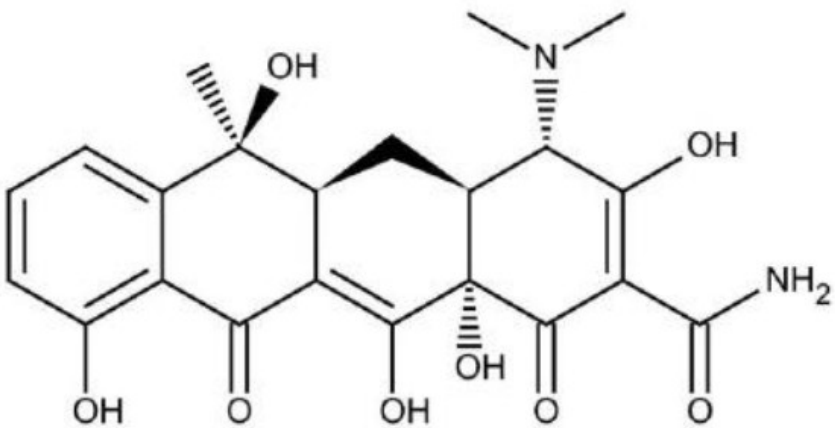
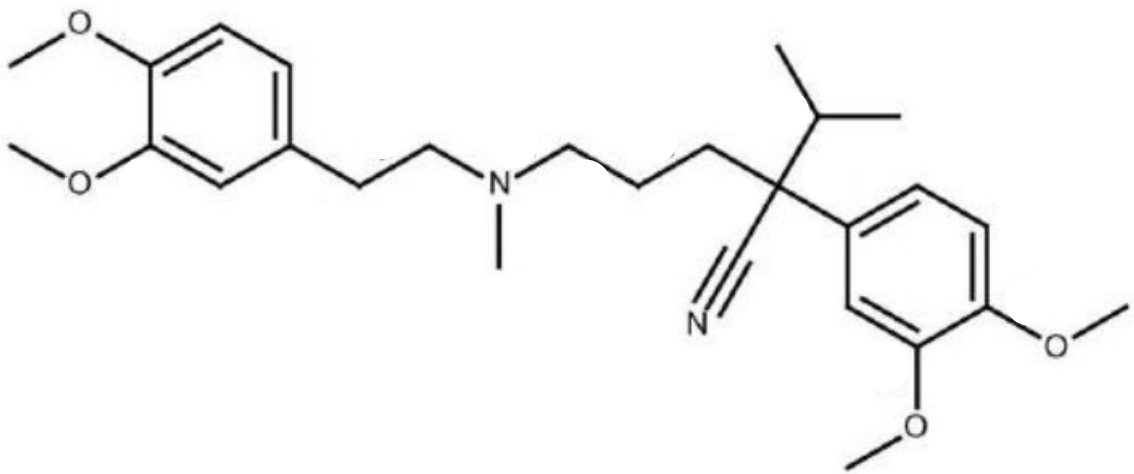


Tetracycline



Verapamil

Shankland, K. *et al.*,
Acta Cryst. C 69 (2013) 1251-1259.

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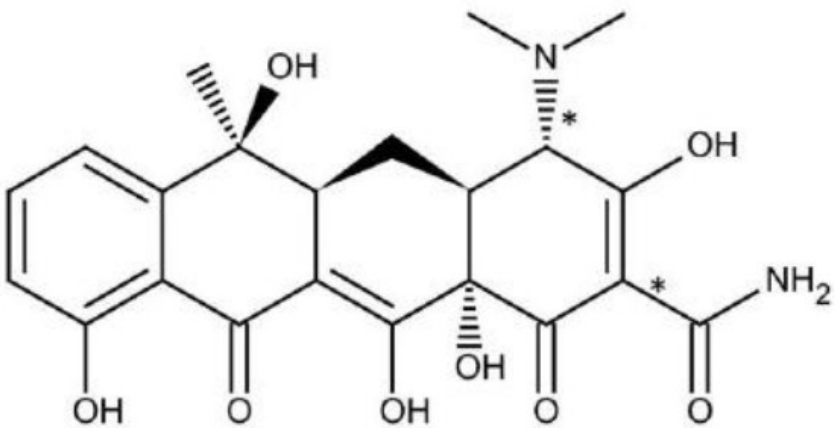

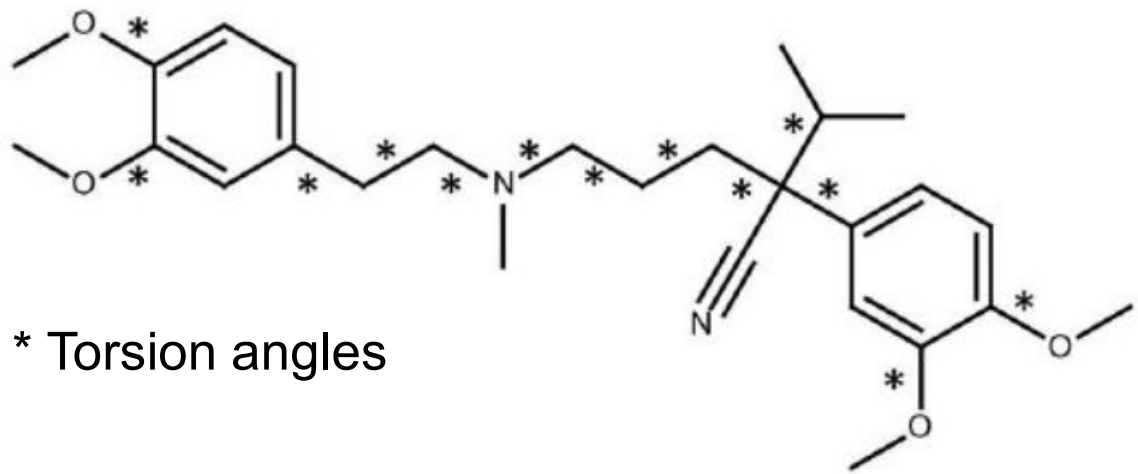

* Torsion angles

Tetracycline 32 non-H atoms
n = 2, **DoF = 8**

Verapamil 33 non-H atoms
n = 13, **DoF = 19**

Shankland, K. *et al.*,
Acta Cryst. C 69 (2013) 1251-1259.

Considerations: Model Size

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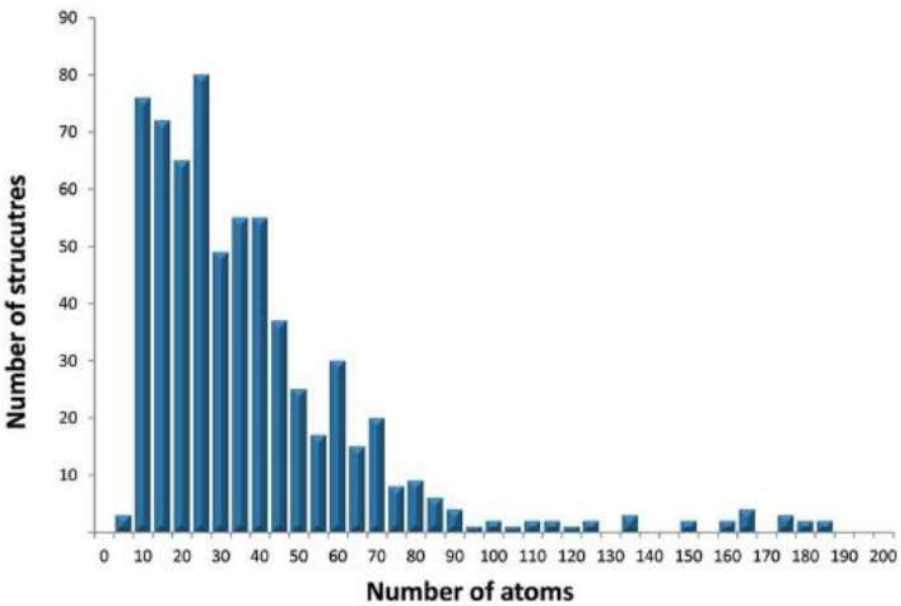
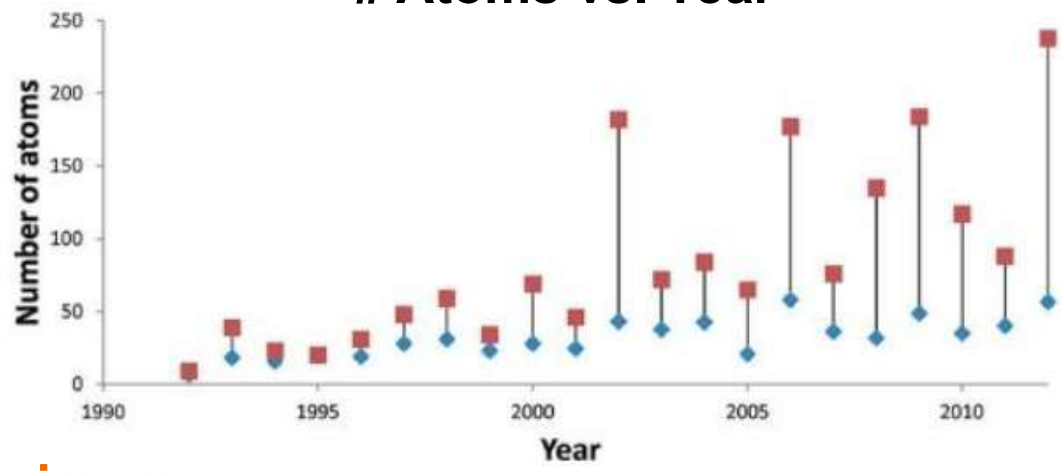
CSD - # of Atoms in PXRD structures

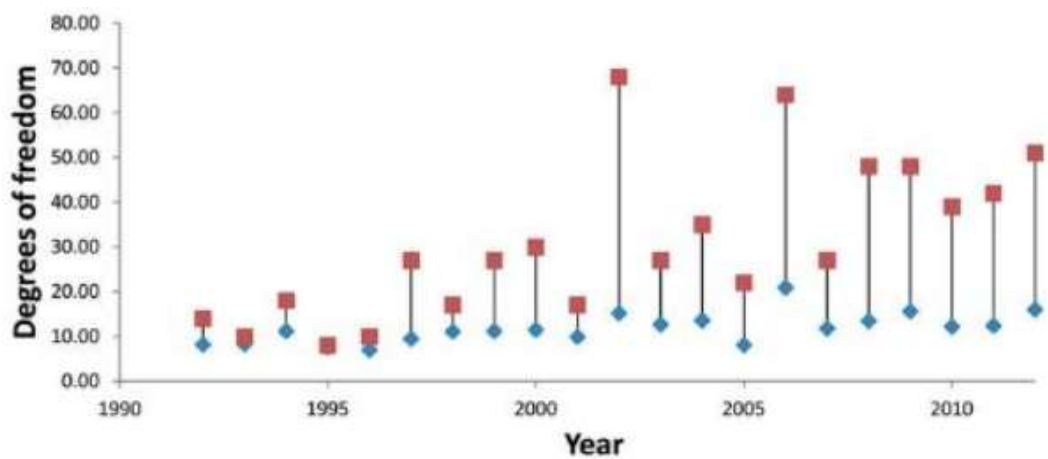


 # Atoms vs. Year



Maximum
Mean

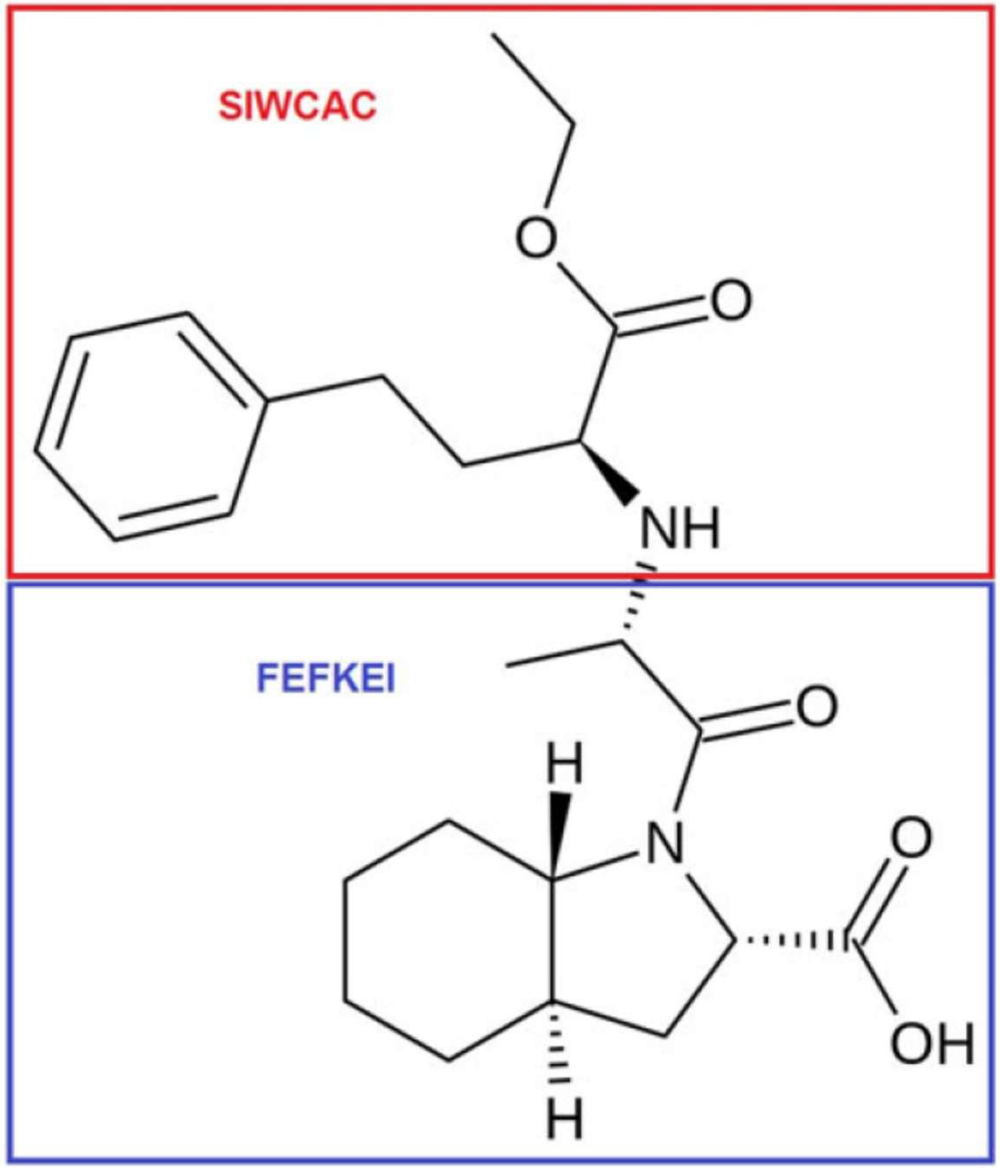
DoF vs. Year



Shankland, K. *et al.*,
Acta Cryst. C 69 (2013) 1251-1259.

Structure Solution Example: FOX

- Laboratory powder data (Cu Kα1, λ = 1.54059 Å) structure solution for trandolapril ($C_{24}H_{34}N_2O_{56}$) using **FOX** with parallel tempering.
- Indexing performed with **DICVOL** determined an orthorhombic cell (a = 19.7145, b = 15.0499, c = 7.6534 Å).
- The space group was determined to be $P2_12_12_1$ using **ChekCell**.
- A trandolapril molecule was built from two entries in the **Cambridge Structural Database (CSD)** using **Avogadro**.



FOX: Farve-Nicolin, V. & Cerny, R., J. Appl. Cryst. 35 (2002) 734-743.

Structure Solution Example: FOX

- Laboratory powder data (Cu Kα1, λ = 1.54059 Å) structure solution for trandolapril ($C_{24}H_{34}N_2O_{56}$) using **FOX** with parallel tempering.
- Indexing performed with **DICVOL** determined an orthorhombic cell (a = 19.7145, b = 15.0499, c = 7.6534 Å).
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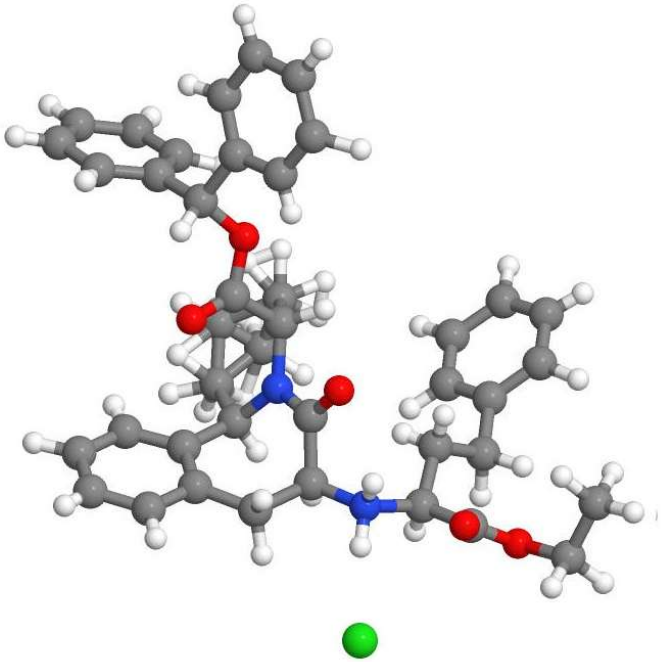
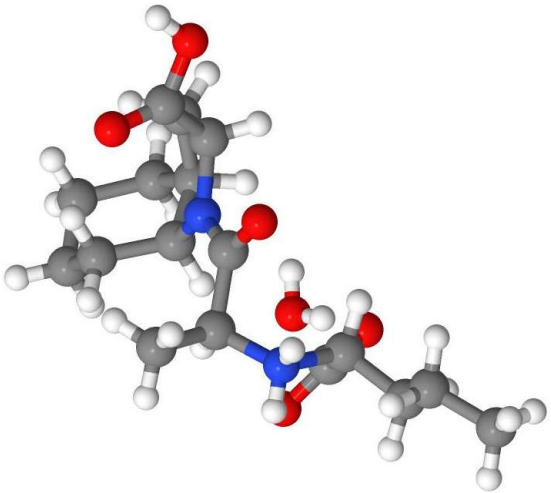
SIWCAC



FEFKEI



CSD: Groom, C. R. *et al.*, Acta Crystallogr. B 72 (2016) 171-179.

Structure Solution Example: FOX

- Laboratory powder data (Cu Kα1, λ = 1.54059 Å) structure solution for trandolapril ($C_{24}H_{34}N_2O_{56}$) using **FOX** with parallel tempering.

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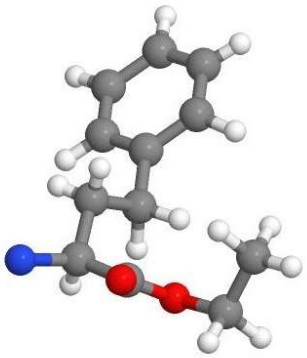
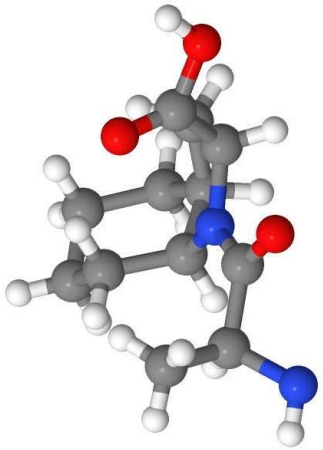
SIWCAC



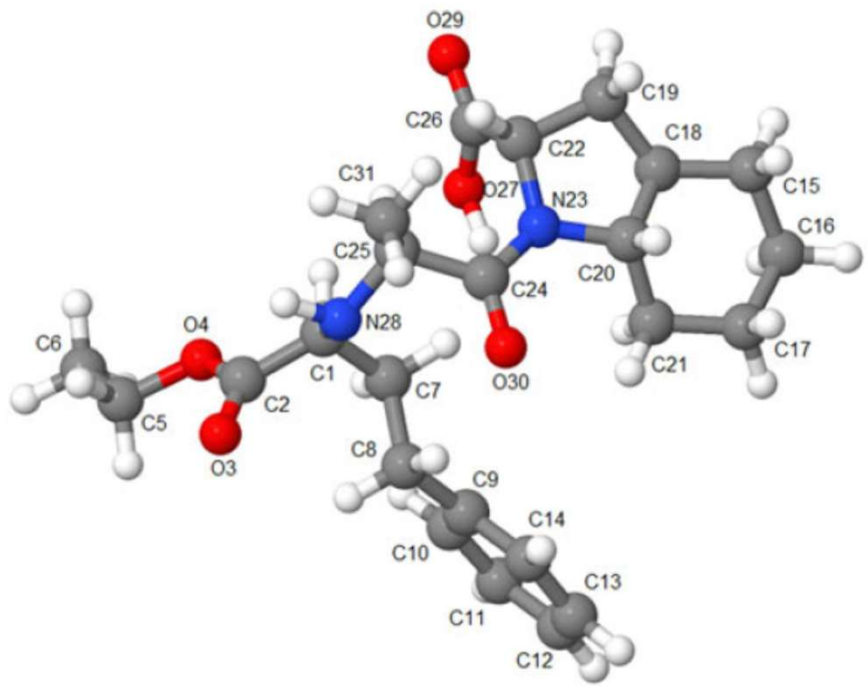
FEFKEI



Avogadro: Hanwell, M. D. *et al.*, J. Cheminform. 4 (2012) 17.

Structure Solution Example: FOX

- Laboratory powder data (Cu Kα1, λ = 1.54059 Å) structure solution for trandolapril ($C_{24}H_{34}N_2O_{56}$) using **FOX** with parallel tempering.
- A trandolapril molecule was built from two entries in the **Cambridge Structural Database (CSD)** using **Avogadro**.
- A crystallographic information file (CIF) of the molecule was converted to a Fenske-Hall Z-matrix (FHZ file) using **Open Babel**.
- The FHZ file was used to load the molecule into **FOX** for structure solution.



Reid, J. W., Kaduk, J. A. & Vickers, M. Powder Diffraction 31 (2016) 205-210.

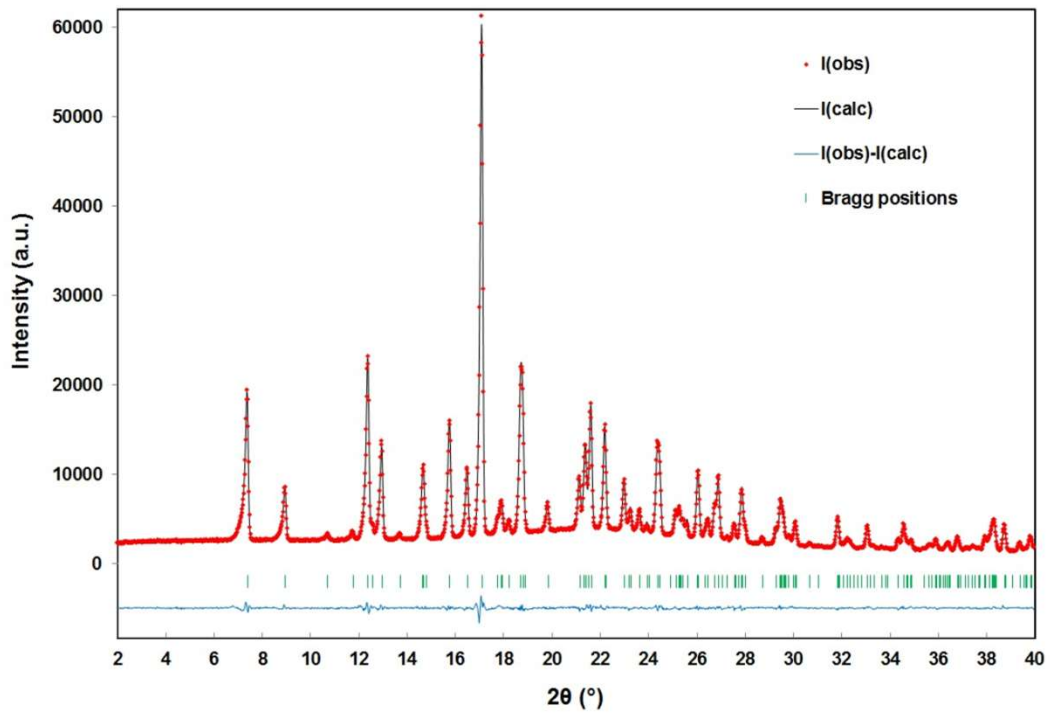
Open Babel: O'Boyle, N. *et al.*, J. Chem. Inform. 3 (2011) 1-14.

Structure Solution Example: FOX

- Laboratory powder data (Cu Kα1, λ = 1.54059 Å) structure solution for trandolapril ($C_{24}H_{34}N_2O_{56}$) using **FOX** with parallel tempering.

- A trandolapril molecule was built from two entries in the **Cambridge Structural Database (CSD)** using **Avogadro**.

- After solving the crystal structure with **FOX**, the structure was Rietveld refined with **GSAS**, using restraints on the bond distances and angles generated by **Mogul (CSD)**.

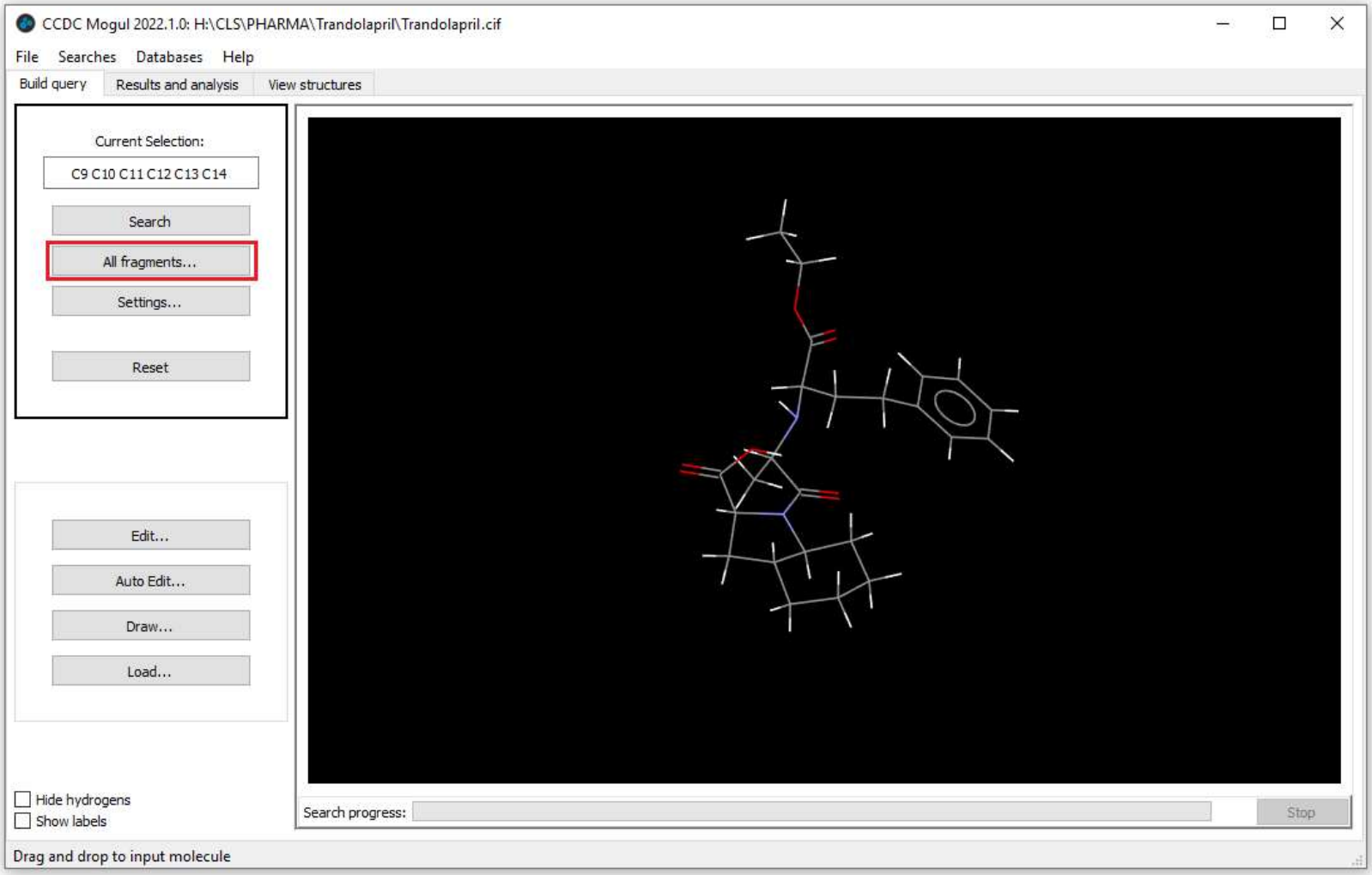


Reid, J. W., Kaduk, J. A. & Vickers, M. Powder Diffraction 31 (2016) 205-210.

GSAS-II: Toby, B. H. & von Dreele, R. B. J. Appl. Cryst. 46 (2013) 544-549.

Examining Bond Distances and Angles with Mogul

- **Mogul** allows you to upload a molecule and compare bond distances and angles to geometries from structures in the **CSD**.



Mogul: Bruno, I. J. *et al.*
J. Chem. Inf. Comput. Sci. 44 (2014) 2133-2144.

Examining Bond Distances and Angles with Mogul

- **Mogul** allows you to upload a molecule and compare bond distances and angles to geometries from structures in the **CSD**.

All fragments: Results

Bond | Angle | Torsion | Ring

Fragment	Number	Minimum	Maximum	Mean	Median	Std. dev.	| z-score |	Query value
C11 C10	20000	0.755	1.685	1.385	1.385	0.019	0.188	1.388
C12 C11	20000	0.588	1.723	1.377	1.379	0.023	0.127	1.374
C13 C12	20000	0.588	1.723	1.377	1.379	0.023	0.070	1.379
C13 C14	20000	0.755	1.685	1.385	1.385	0.019	0.124	1.382
C10 C9	14022	0.618	1.734	1.384	1.386	0.021	0.054	1.385
C14 C9	14022	0.618	1.734	1.384	1.386	0.021	0.151	1.387
C17 C16	12510	0.705	1.844	1.511	1.518	0.038	0.247	1.521
C16 C15	12163	0.991	1.929	1.528	1.528	0.026	0.180	1.532
C17 C21	12163	0.991	1.929	1.528	1.528	0.026	0.553	1.513
C6 C5	11354	0.635	2.028	1.466	1.485	0.074	0.040	1.469
O4 C5	10569	0.920	1.871	1.462	1.458	0.031	0.341	1.472
O3 C2	10562	0.976	1.688	1.208	1.203	0.025	0.005	1.208
C8 C9	10390	1.104	1.930	1.513	1.510	0.029	0.164	1.517
O30 C24	10237	1.014	1.407	1.219	1.217	0.018	0.029	1.220
O29 C26	8079	1.025	1.679	1.216	1.211	0.030	0.192	1.222
O27 C26	8077	1.042	1.662	1.302	1.309	0.032	0.378	1.290
C15 C18	5952	1.001	1.789	1.527	1.528	0.023	0.392	1.517

Select row to view search results

Export...

Mogul: Bruno, I. J. *et al.*
J. Chem. Inf. Comput. Sci. 44 (2014) 2133-2144.

Crystal Structure Validation

- Once you have a tentative structure, it's important to take multiple steps to confirm it is (likely) correct:
  - Make sure you can obtain a quality final Rietveld refinement (reasonable statistical metrics, and more importantly, a decent visual fit to the pattern).
  - Make sure the structure is consistent with the available elemental and chemical analysis (XRF, ICP-MS, NMR, IR, etc.).
  - Check the structure to make sure it is chemically reasonable.
  - See how closely the Rietveld-refined structure compares with a density functional theory (DFT) optimized structure, if possible.

Evaluating statistical metrics:
Toby, B. Powder Diffr. 21 (2006) 67-70.

Chemical reasonableness - organics:
Kaduk, J. A. Powder Diffr. 22 (2007) 74-82.

Chemical reasonableness - inorganics:
Kaduk, J. A. Powder Diffr. 22 (2007) 268-278.

Structure Validation - Chemical Reasonableness

- For organic structures, run the structure through the **Mogul** module of the **Cambridge Structural Database** (**CSD**) to check the bond distances and angles.

- For inorganic structures, perform bond valence sum calculations to see if the bond lengths and valences are reasonable **(BondStr** in the **FullProf Suite**).

- For either type of structure, run your final CIF through **CheckCIF** (https://checkcif.iucr.org/).

Bond Valence Model:
Brown, I. D. The Chemical Bond in Inorganic Chemistry: The Bond Valence Model. (Oxford: New York, 2002).

Chemical reasonableness - organics:
Kaduk, J. A. Powder Diffr. 22 (2007) 74-82.

Chemical reasonableness - inorganics:
Kaduk, J. A. Powder Diffr. 22 (2007) 268-278.

What is Density Functional Theory (DFT)?

- DFT is a quantum mechanical computation method that models electronic (atomic) structure and related properties in terms of the electron density in the system.

- Some excellent introductory resources and primers exist regarding the theory and application of DFT:
  - Burke, K., & Wagner, L.O. 'DFT in a Nutshell,' Int. J. Quantum Chem. (2012) 24259.
  - Fiolhais, C., Nogueira, F., & Marques, M. (Eds.). A Primer on Density Functional Theory. Springer: Berlin (2003).
  - Thakkar, A. J. Quantum Chemistry: A concise introduction for students of physics, chemistry, biochemistry and materials science (2nd Ed). Morgan and Claypool: San Rafael (2017).

- There are many software packages for DFT (**CRYSTAL**, VASP, ORCA, CASTEP, Quantum Expresso, Gaussian, Hyperchem, etc.).

Why Use DFT with Powder Diffraction?

- Of the numerous types of quantum mechanical calculations, DFT is one of the most used, successful and versatile methods available.

- DFT can help you:
  1. Validate your results and determine if your interpretation of your data is correct.
  2. Provide more accurate structural details (i.e. hydrogen positions and bonding) where the experimental data is ambiguous or completely lacking.
  3. Gain additional insights into properties of your material that might not be clear with the experimental data alone.

Why use DFT with PXRD Structure Solution?

- For small molecule crystal structures, PXRD provides the basic packing arrangement of the molecules, but DFT can improve the details:

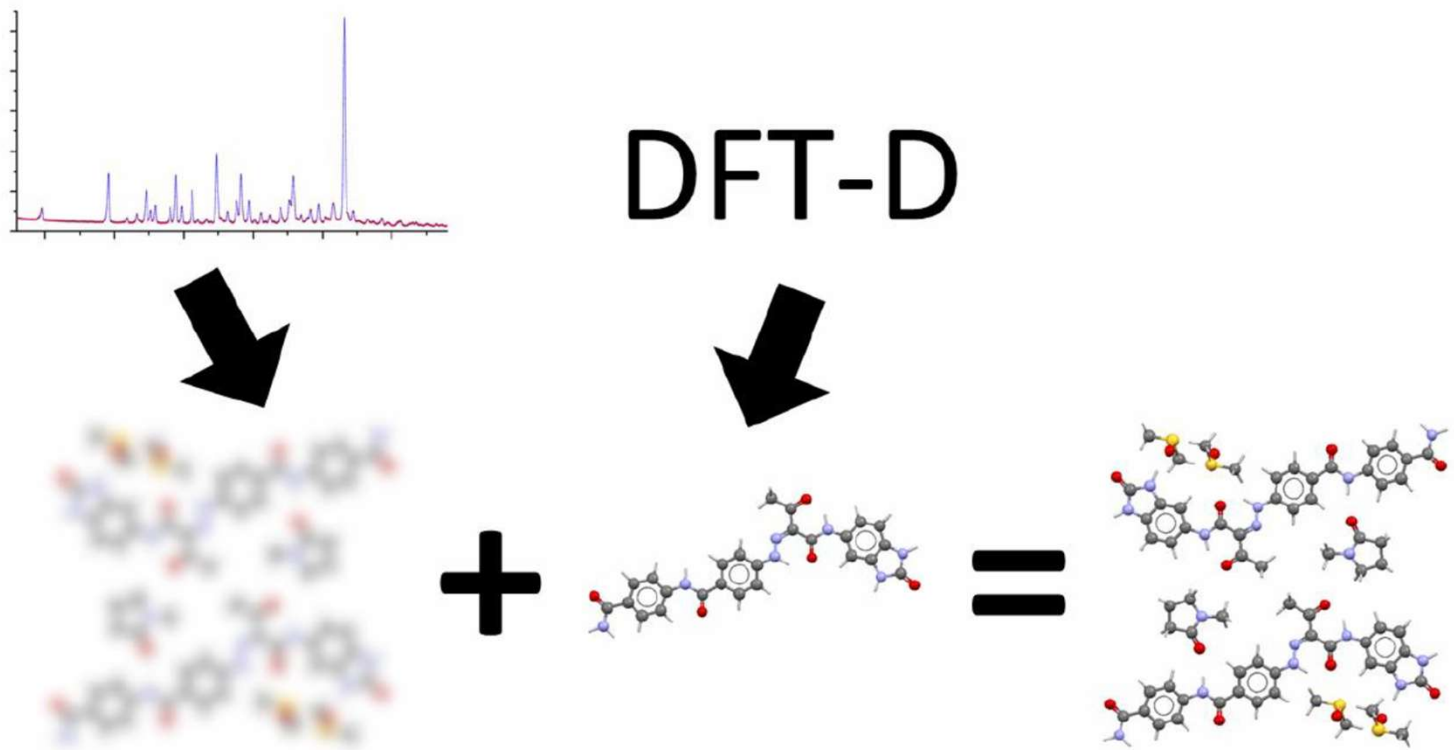
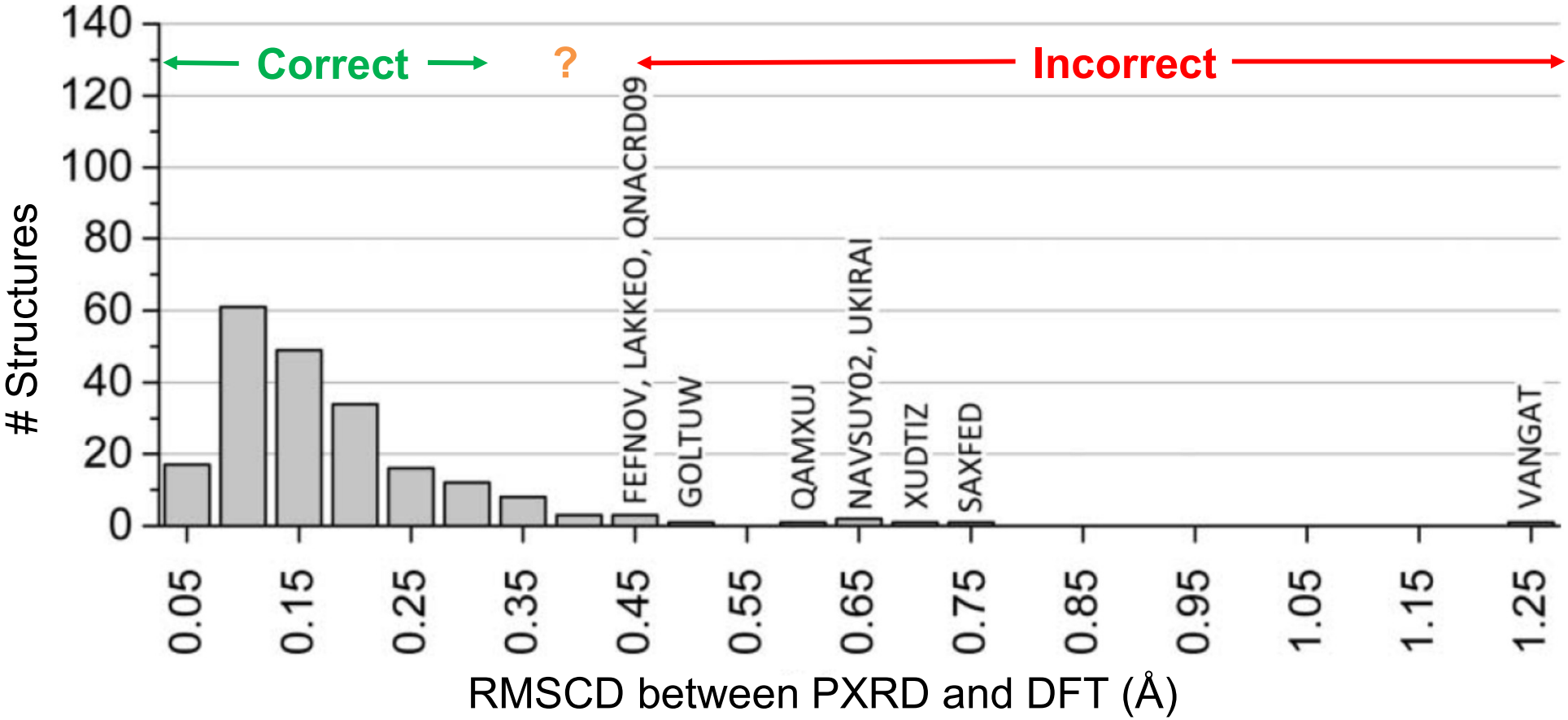


Figure taken from:
van de Streek, J. ‘Reliable and Highly Accurate Molecular Crystal Structures from a Combination of XRPD and DFT-D.’ Presented at Accuracy in Powder Diffraction IV (2013).

Benchmarking PXRD Structure Validation with DFT

- The average root-mean-square Cartesian displacement (RMSCD) between the non-hydrogen atoms of the PXRD and DFT structures can be used to validate the correctness of the PXRD structure.

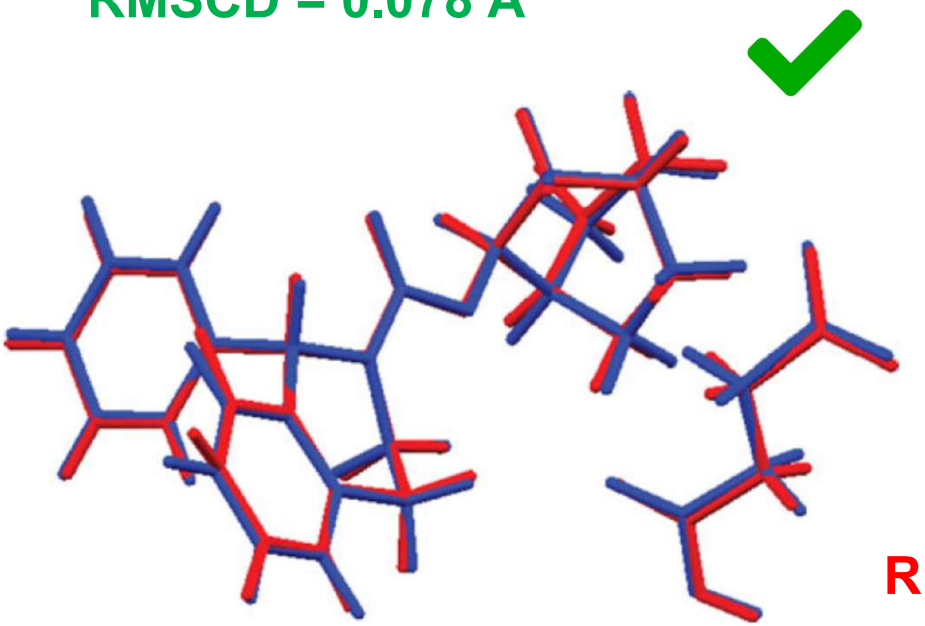


van de Streek, J. & Neumann, M. A. Acta Cryst B70 (2014) 1020-1032.

Benchmarking PXRD Structure Validation with DFT

- The average root-mean-square Cartesian displacement (RMSCD) between the non-hydrogen atoms of the PXRD and DFT structures can be used to validate the correctness of the structure.

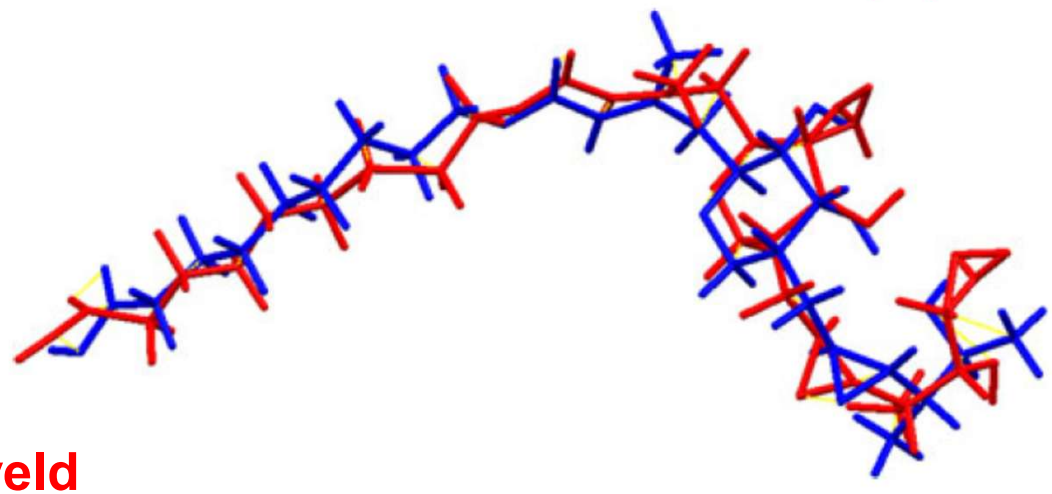
Solifenacin hydrogen succinate
RMSCD = 0.078 Å



Kaduk, J.A. *et al.* Powder Diffraction 30 (2015) 211-217

Rietveld
DFT

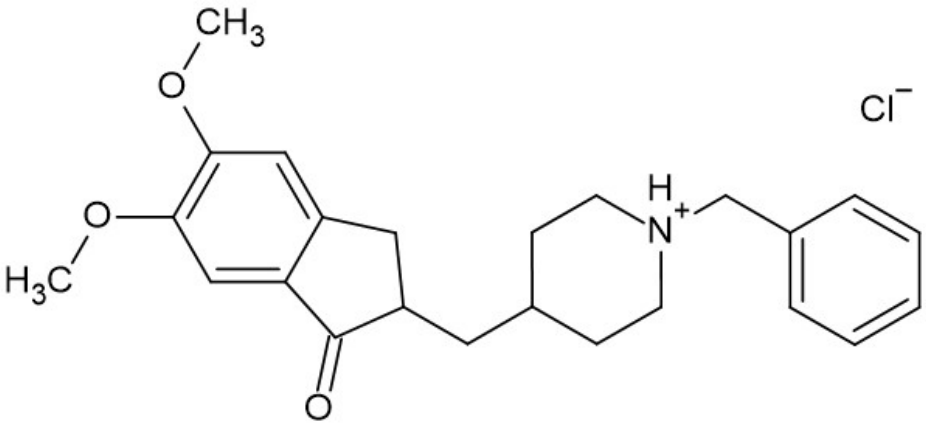
Mupirocin form I,
RMSCD = 1.02 Å

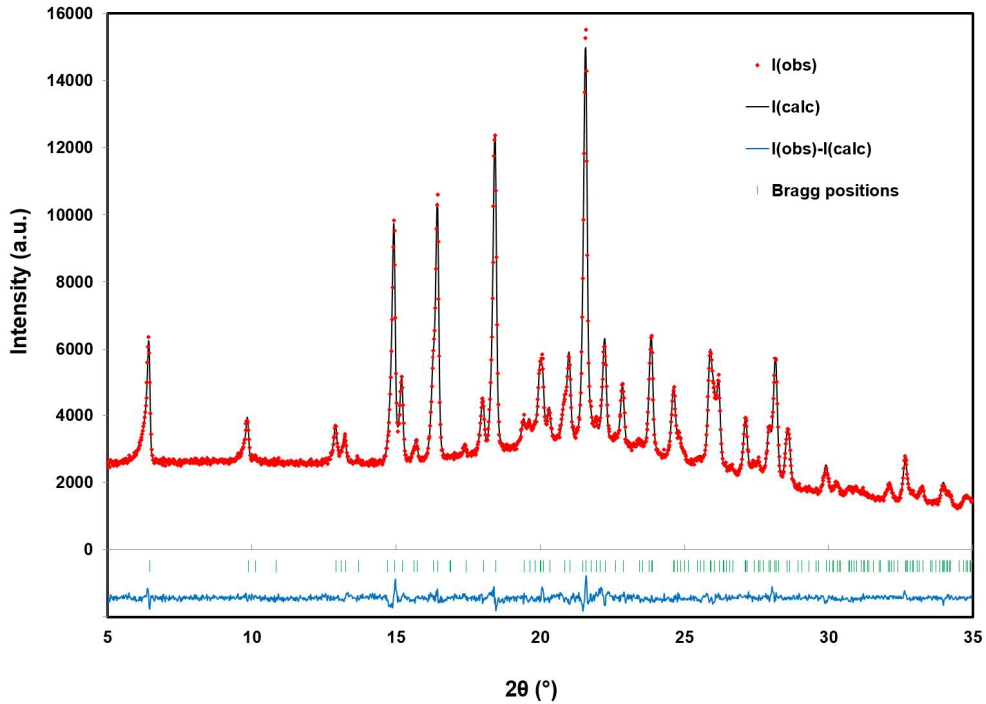


Kaduk, J.A. *et al.* Powder Diffraction 31 (2016) 118-125

DFT Example: Donepezil Hydrochloride

Donepezil HCl, form III
$C_{24}H_{29}NO_3 \cdot HCl$



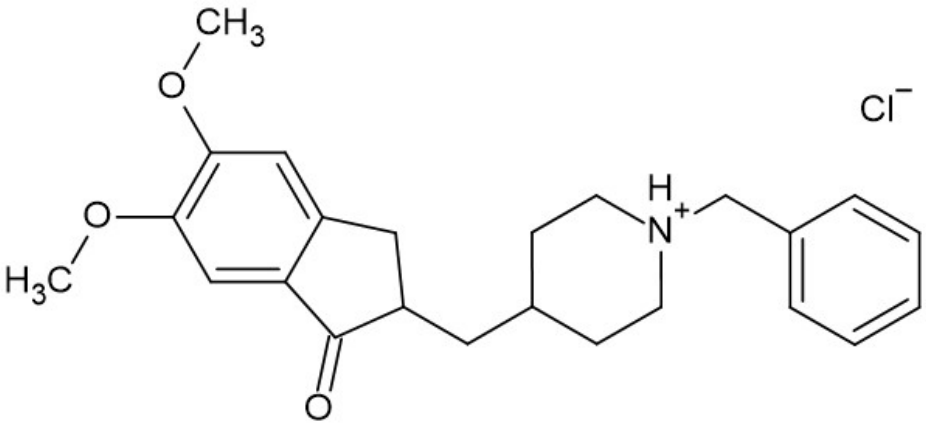


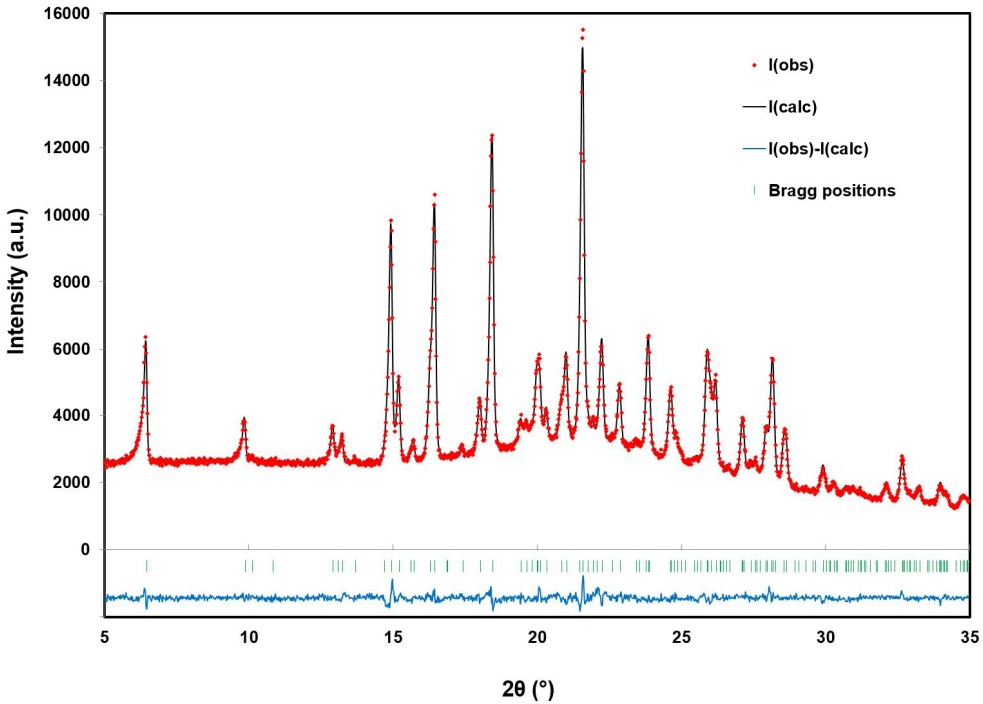
Lab PXRD, Cu Kα1 (λ = 1.54059 Å)
Upper Limit: 2θ = 35° (d_{min} = ~ 2.5 Å)

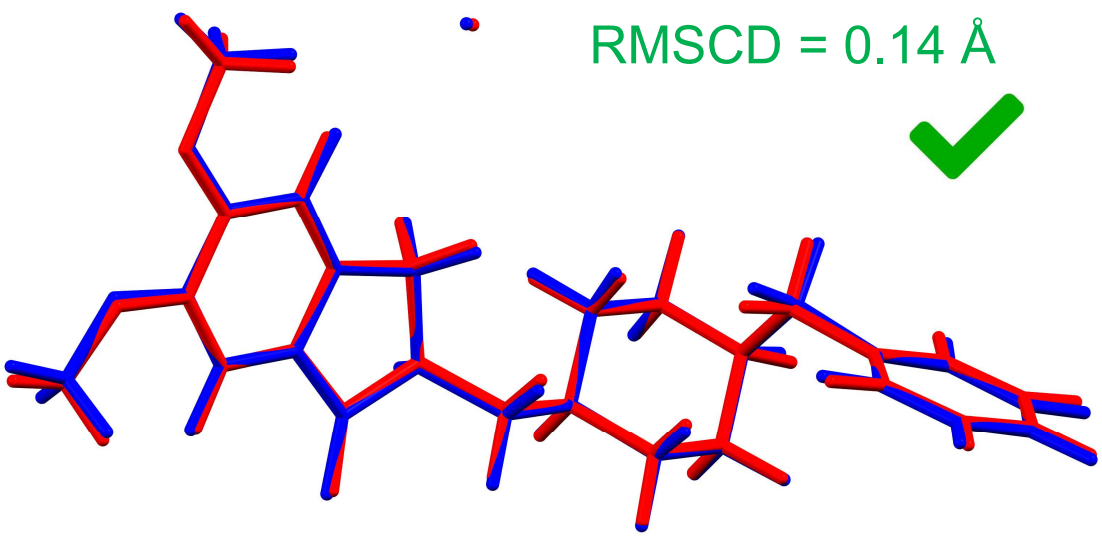
Can we actually solve the correct structure with this data range?

DFT Example: Donepezil Hydrochloride

Donepezil HCl, form III
$C_{24}H_{29}NO_3 \cdot HCl$







RMSCD = 0.14 Å

Rietveld
DFT

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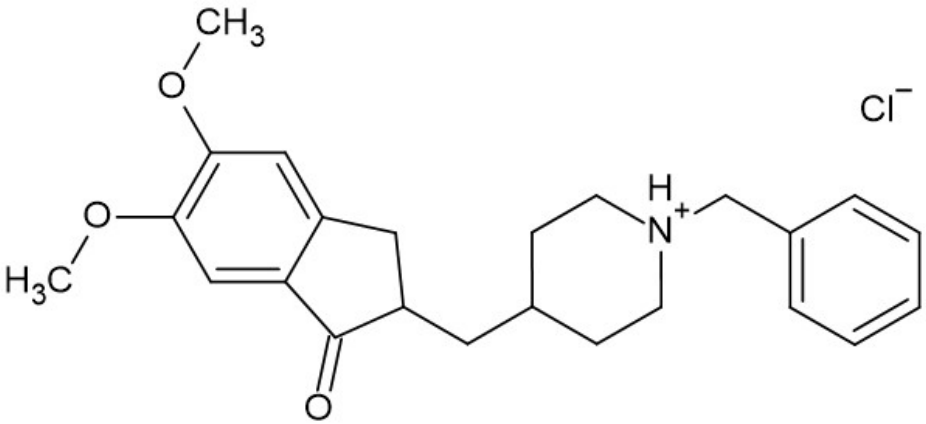
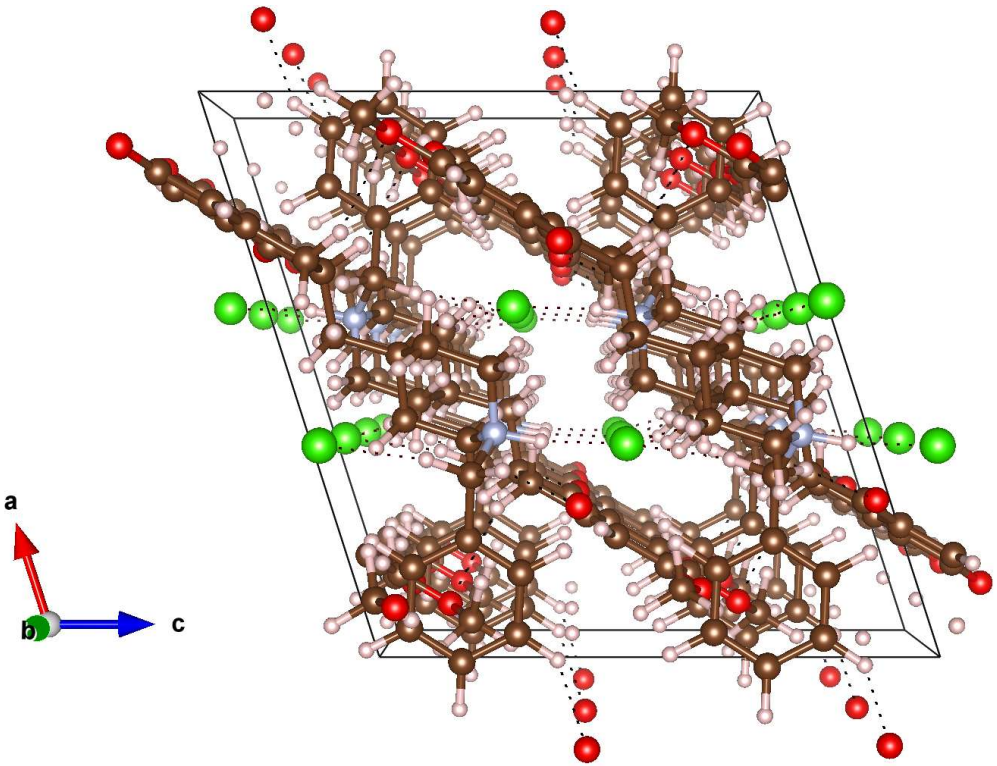
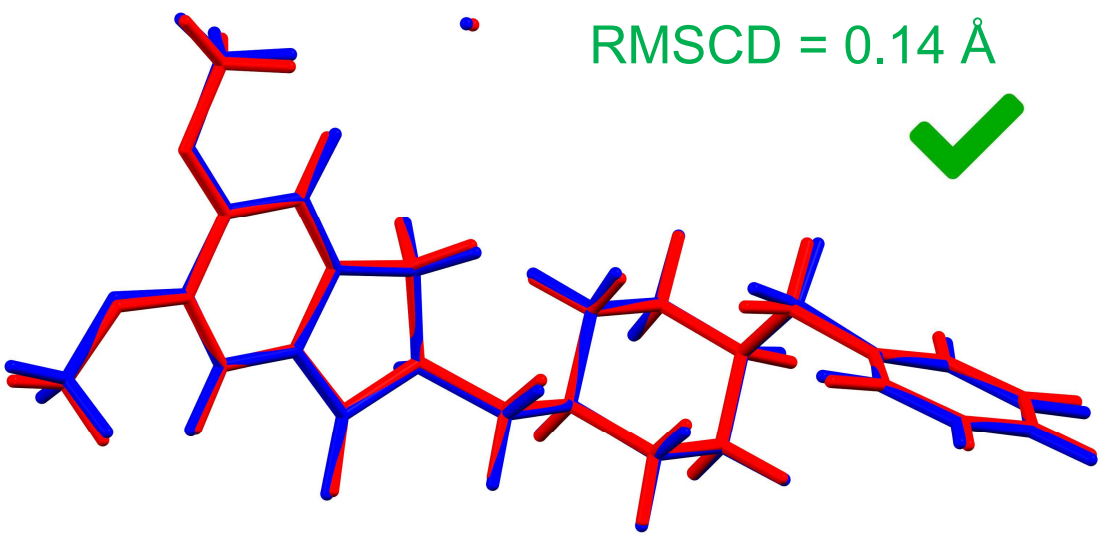
Canadian Light Source
Centre canadien de rayonnement synchrotron

DFT Example: Donepezil Hydrochloride

Donepezil HCl, form III
$C_{24}H_{29}NO_3 \cdot HCl$







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Reid, J.W. & Kaduk, J.A.,
Powder Diffraction 36 (2021) 233-240

Canadian Light Source
Centre canadien de rayonnement synchrotron

Software for Structure Solution

Step	Software
Search/Match and Data Mining Databases	**Powder Diffraction File** (**PDF-5+**), **Cambridge Structural Database** (**CSD**)
Ab initio indexing	**DICVOL**, **TREOR** (via **FullProf Suite**), **McMaille** (via **EXPO2014**), **GSAS2**, **FOX**
Space Group Determination	**ChekCell**, **EXPO2014**, **GSAS2**, **FOX**
Le Bail & Rietveld Refinement	**GSAS2**, **FullProf**, **JANA2006**, **FOX**, Topas
Reciprocal-Space Structure Solution	**EXPO2014**, **SuperFlip** (via **JANA2006**), **GSAS2**, XLENS
Direct-Space Structure Solution	**FOX**, **GSAS2**, GALLOP
Structure Building, Editing, Plotting & File Conversion	**Avogadro**, **VESTA**, **JMOL**, **Mercury** (**CSD**), **Open Babel**
Bond Distances, Angles, Valences and Structure Validation	**Mogul** (**CSD**), **BondStr**, **CheckCIF**
Density Functional Theory (DFT)	**CRYSTAL17**, VASP, Quantum Expresso, CASTEP, Gaussian

Summary

- Structure solution from powder diffraction data is a powerful tool that comes with challenges. But for some materials, it may be the only viable route to a crystal structure.

- Much more so than single-crystal XRD, structure solution from powder data generally requires:
  - Significant knowledge of the molecular structure and chemistry of the material.
  - Constraints and restraints during both structure solution and refinement to keep solutions chemically reasonable.
  - Validation and verification of the structure using additional chemical and physical data and/or DFT optimization, if possible.

Acknowledgements

- Many thanks are due to Jim Kaduk for multiple collaborations, many helpful discussions regarding aspects of structure solution and density functional theory, and for providing a constant source of inspiration.

Further Reading

- David, W.I.F., Shankland, K., McCusker, L. & Baerlocher, C. (ed.) Structure Determination from Powder Diffraction Data.
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- Pecharsky, V.K. & Zavalij, P.Y. Fundamentals of Powder Diffraction and Structural Characterization of Materials, 2nd Ed.
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  RSC: Cambridge, 2009.