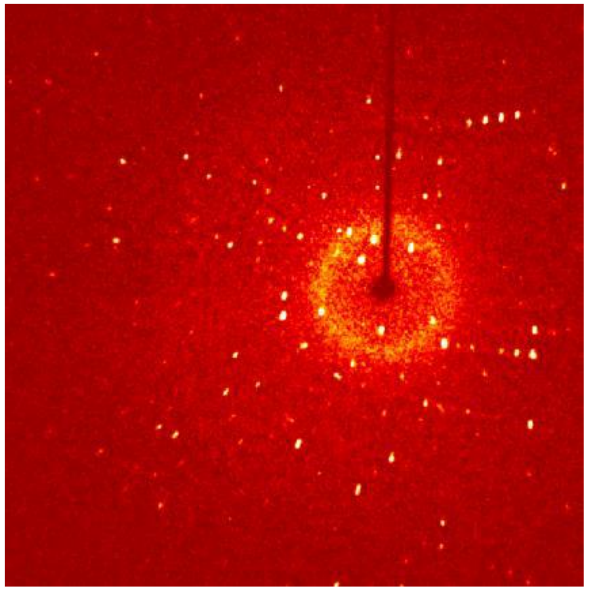
T6. Modeling disorder: Solvent masks

Slides and sample data from Dr. Brian Patrick, UBC

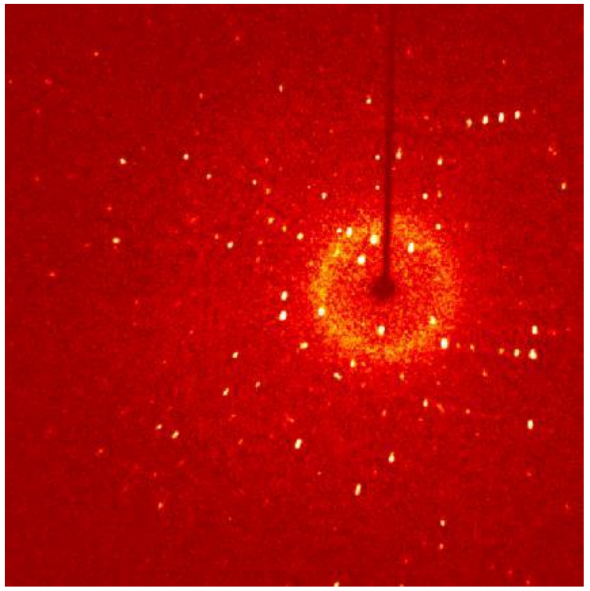
Edits and presentation by Dr. Louise Dawe, WLU

Based on: Spek, A.L. *Acta Cryst.* (2015). C**71**, 9–18.
https://journals.iucr.org/c/issues/2015/01/00/ln3172/ln3172.pdf



Integrate reflections

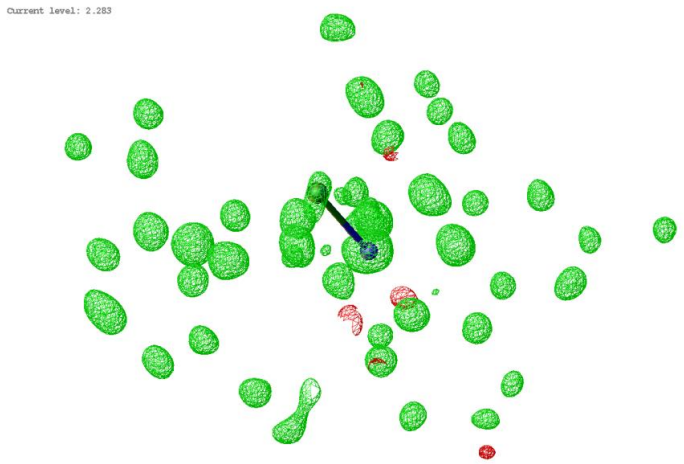
$$F_o = \sqrt{I / Lp}$$

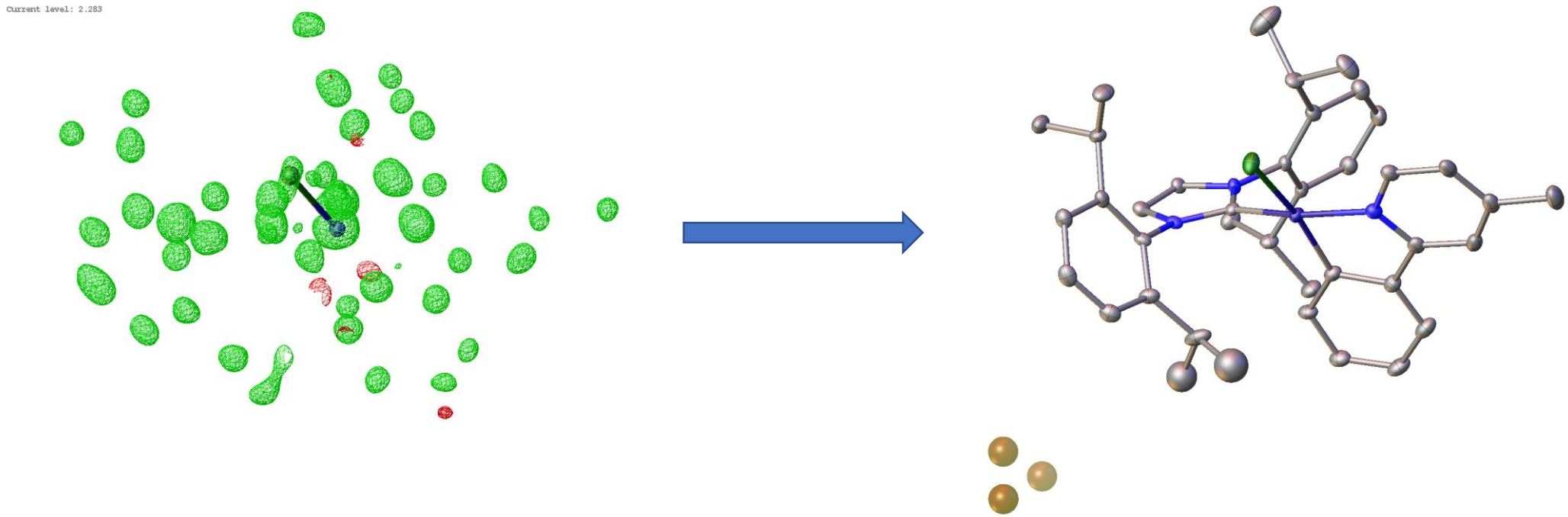


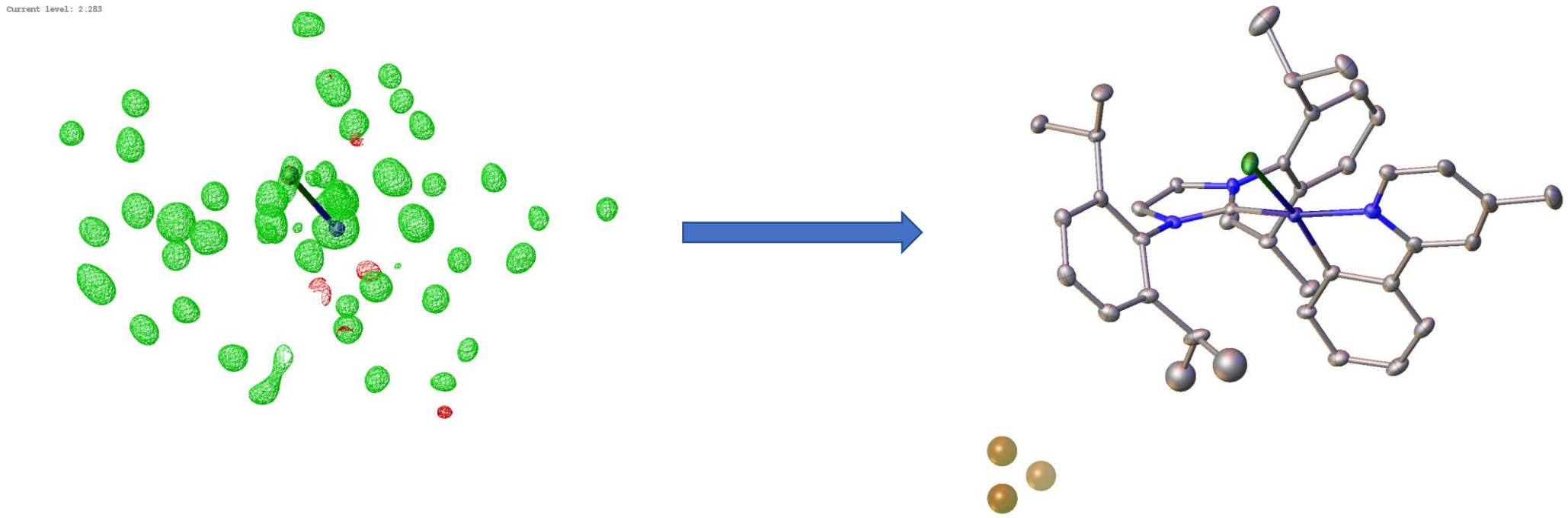
Integrate reflections

$$F_o = \sqrt{I / Lp}$$

$$\rho(xyz) = \frac{1}{V} \sum_{hkl} \vec{F}_{hkl} \cos 2\pi(hx + ky + lz)$$




Current level: 2.283

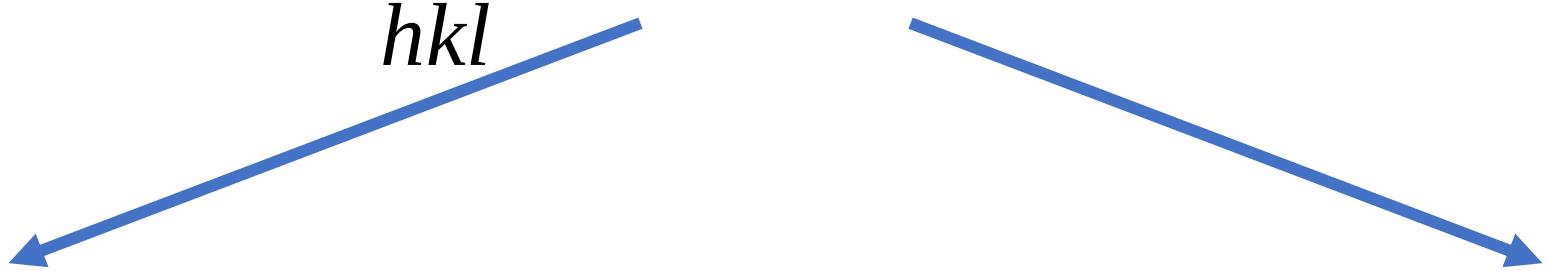


Use scattering factors and coordinates (x, y, z) for each atom (j) in the unit cell to calculate structure factors (F) for each reflection.

$$\vec{F}(hkl) = \sum f_j \exp[2\pi i(hx_j + ky_j + lz_j)]$$

Least-squares refinement tries to minimize

$$\sum_{hkl} w'(F_o^2 - F_c^2)^2$$



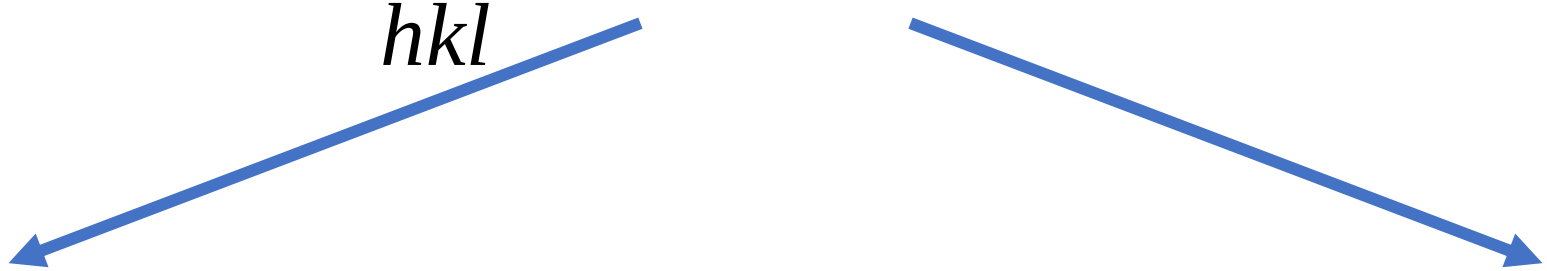
Your measured intensities

Calculated from your model

Each atom in your model contributes to the calculated structure factor.

Least-squares refinement tries to minimize

$$\sum_{hkl} w'(F_o^2 - F_c^2)^2$$



Your measured intensities

Calculated from your model

The closer your model approximates the electron density in your unit cell, the smaller the difference.

Solvent Masking via PLATON's SQUEEZE

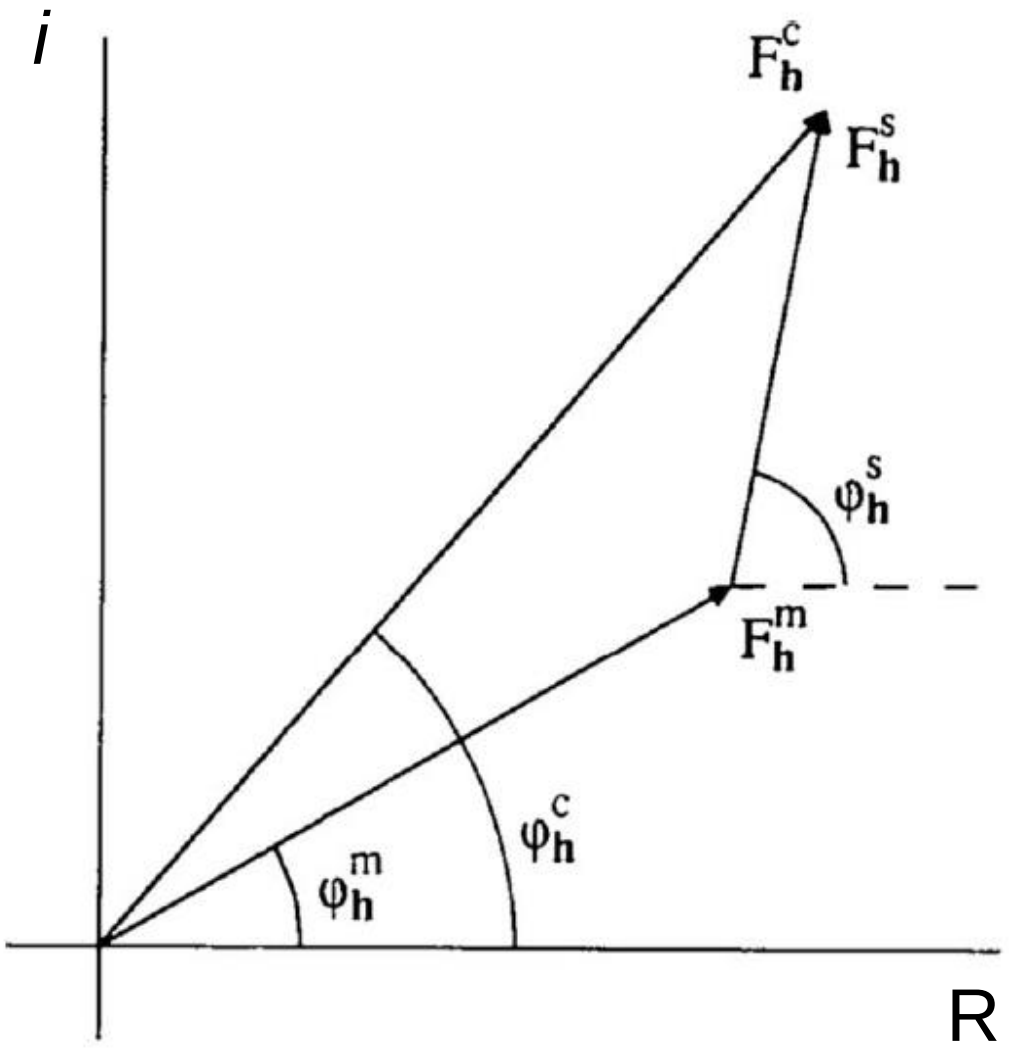
Identifies solvent accessible regions in the unit cell.

Calculates unresolved (unmodeled) electron density in that region.

SQUEEZE calculates the solvent contribution to the structure factors by back-Fourier transformation of the electron density found in the solvent-accessible region

$$F_{calc} = F_{Main} + F_{Solvent}$$

Solvent Masking via PLATON's SQUEEZE



$$F_{calc} = F_{Main} + F_{Solvent}$$

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- Refine everything in the main structure, including non-solvent disorder and hydrogen atoms.
- Refinement must include the ACTA and LIST 4 command in the .ins file. This will create the .cif and .fcf in a format that can be used by PLATON SQUEEZE.
- Twinned data with HKLF5 or TWIN/BASF data requires LIST 8 (not 4).

- Initiate PLATON/SQUEEZE. It will:

  - Search for solvent accessible voids

  - Calculate the electron density in each void

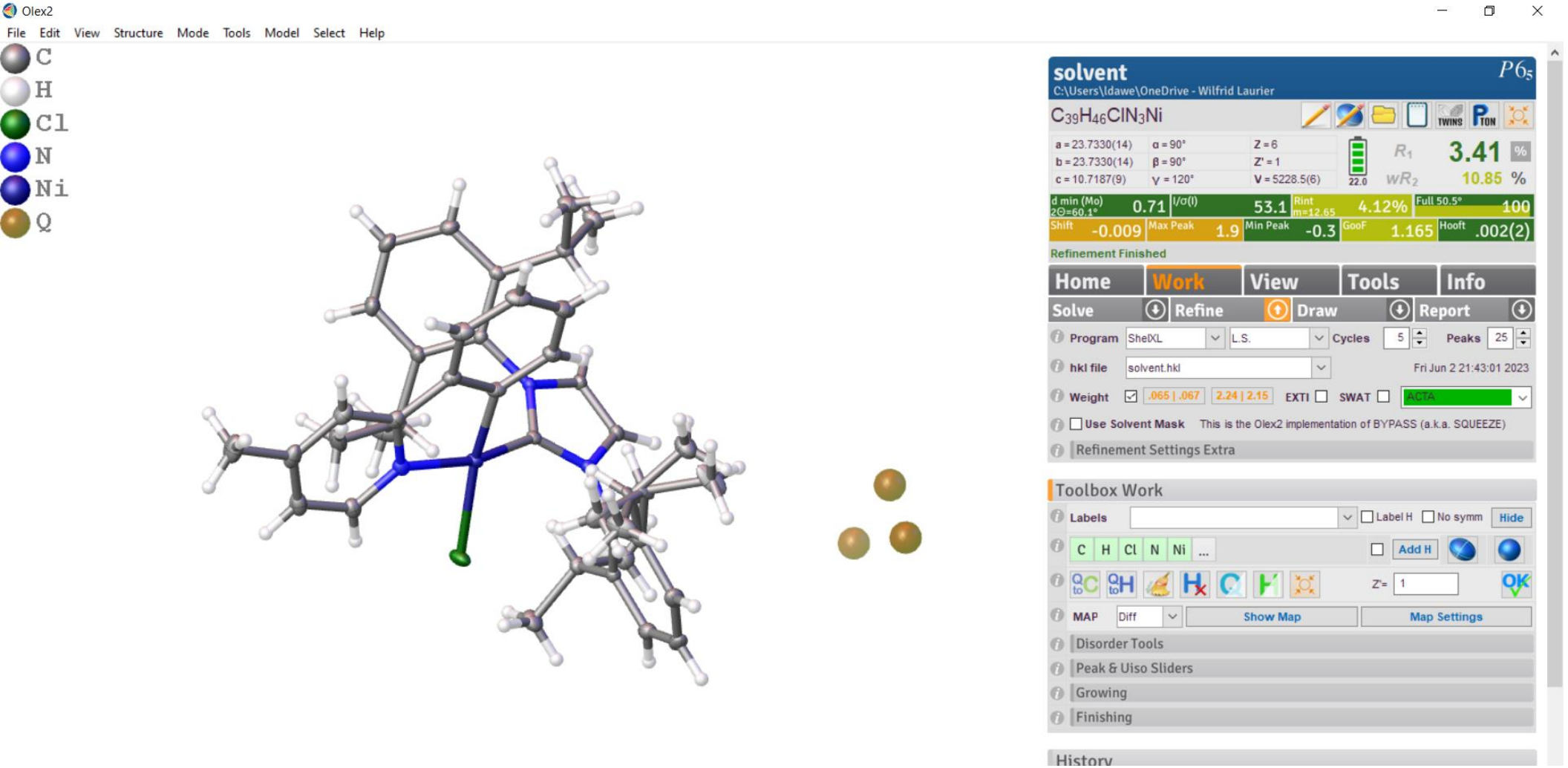
  - Calculate the correction (.fab file) required to each reflection to remove that electron density from the void.

- Initiate PLATON/SQUEEZE. It will:

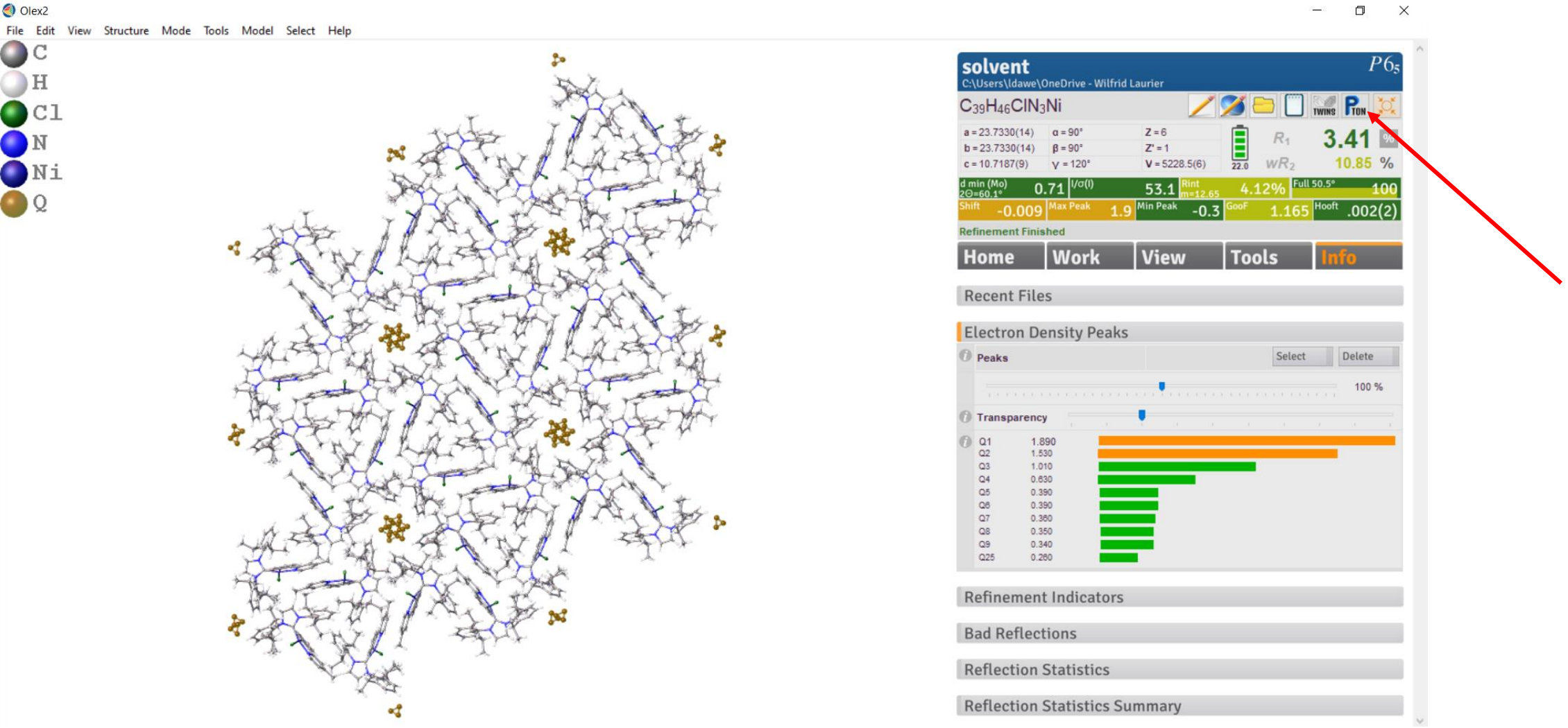
  - Creates new .ins file (name_sq.ins) containing the ABIN instruction

  - Creates new .hkl file (name_sq.hkl), identical to original .hkl file

  - Creates new .fab file (name_sq.fab), includes the solvent contribution to the calculated structure factors



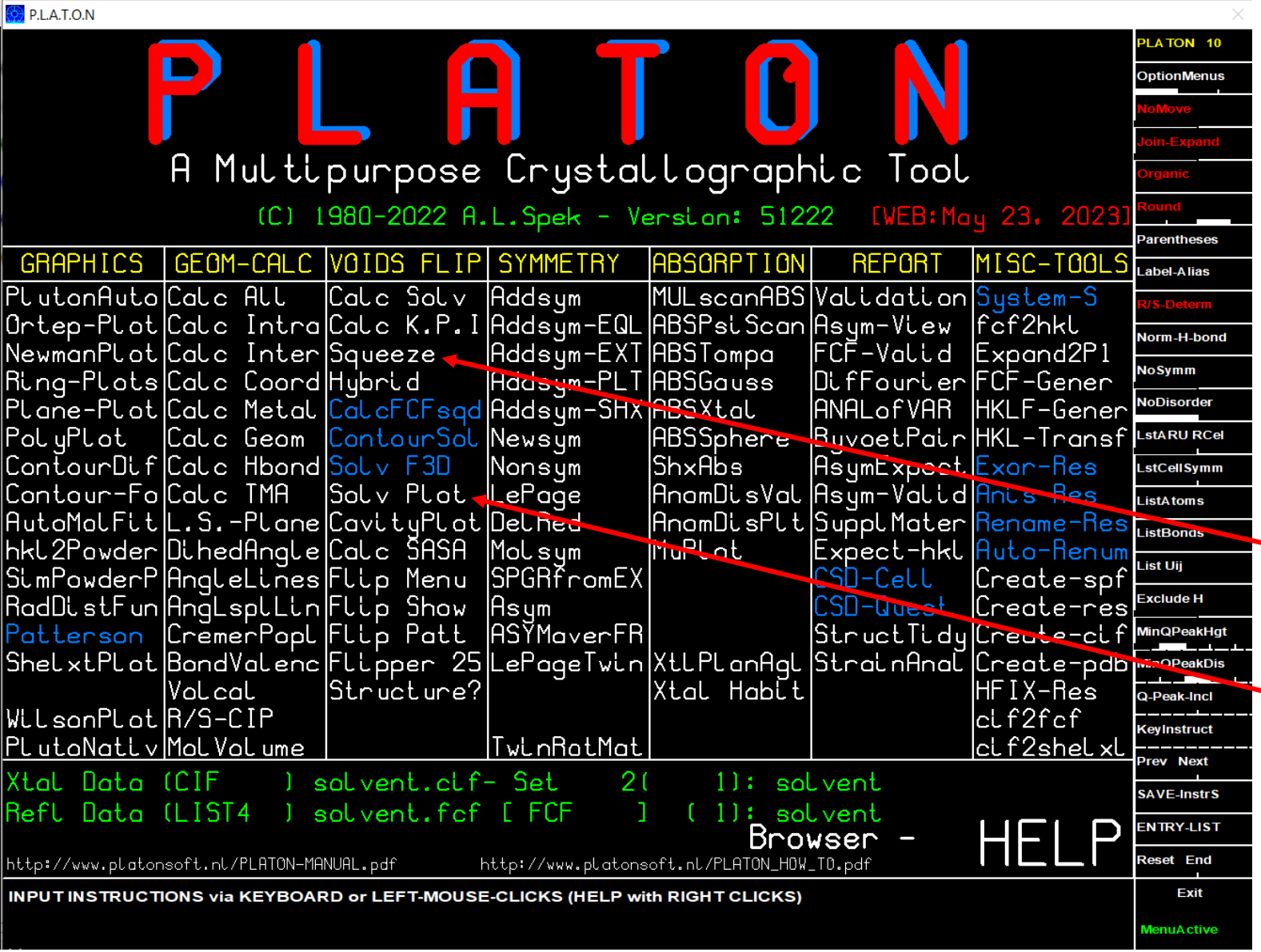
As a first step, you can explore the disordered solvent. Some suggestions:
CTRL + Q to connect the three largest peaks
GROW
PACK
MATR 3



As a first step, you can explore the disordered solvent. Some suggestions:
Navigate to INFO → Electron Density Peaks

FUSE

Launch PLATON (if you do not have the PTON button, you can launch from the folder that hosts pwt.exe).



2. Apply a solvent mask.

1. Visualize the solvent accessible region.

PLATON - Dialog Window

```
      Values below for gridpoints and volumes in []
      refer to areas where atom centers may reside.

Area #GridPoint VolPerc.  Vol(A^3)  X(av) Y(av) Z(av) Eigenvector(frac) Sig(Ang)
-------------------------------------------------------------------------------
 1  34650[  9462]  5   262[  71.6]  0.000 0.000-0.023 1 -0.058 0.055 1.000  3.16
                                                     2  0.979 1.000 0.000  1.53
                                                     3 -1.000 0.937-0.803  1.40

       x     y     z              Shortest Contacts within 4.5 Ang. (Excl. H)
===============================================================================
  1  0.000 0.000-0.023            C20  3.47; C21  3.94; C19  4.16;

:: Note: use CALC VOID (not CALC SOLV) for Packing Index.

     Report the Distance from VOID-CG to Boundary in EV-Directions

 Nr MinEV1 MaxEV1 MinEV2 MaxEV2 MinEV3 MaxEV3 MaxDist (Ang)
===============================================================================
  1  -6.78   6.38  -3.87   1.87  -2.85   2.86   6.78
:: ADP <C24A          0.005   0.020   0.072 - RATIO(MAX/MIN) =     15.1 prolate

:: TRMX = (  1.00  0.00  0.00/  0.00  1.00  0.00/  0.00  0.00  1.00)
:: CELL =    23.733    23.733    10.719    90.000    90.000   120.000

:: Reflection Data are READ from File : solvent.fcf - ( OBS-Data)
W: No Reflections Supplied !

:: END Statement Executed for this Entry
:: *** FILES for Final Refinement with SHELXL201n ***

:: SQUEEZE  ins on :solvent_sq.ins
:: SQUEEZE  hkl on :solvent_sq.hkl
:: SQUEEZE  fab on :solvent_sq.fab
:: SQUEEZE  cif on :solvent_sq.sqf
:: SQUEEZE  xyz on :solvent_sq.sqz
```

1. Return to OLEX2.
2. Open solvent_sq.ins.
3. Change your hkl file to solvent_sq.hkl.
4. Refine.
5. Examine the result (including the CIF).