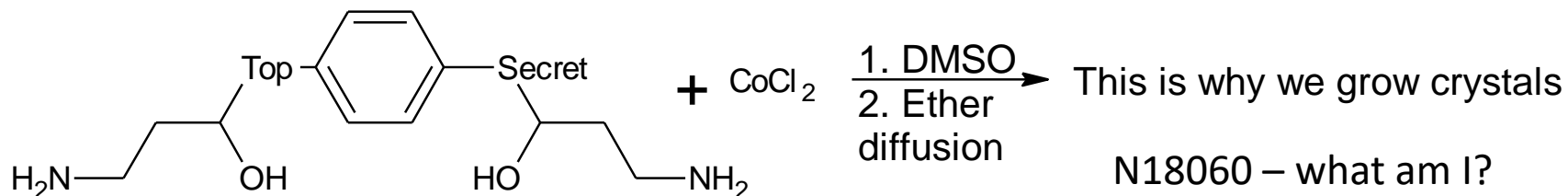


Canadian National Committee  
for Crystallography  
<http://xtallography.ca/>

## OLEX2 – Getting Started

Louise Dawe<sup>a</sup>, Christina Rodriguez<sup>a</sup>, Paul Boyle<sup>b</sup>

a. Wilfrid Laurier University; b. Western University



Need a file with basic information and a reflection file. Hkl

Navigate to your data folder and select your .ins (sometimes .p4p)

Careful about putting your data in the subfolder of a subfolder with a long pathname



The screenshot displays the Olex2 software interface. On the left, the 'File' menu is open, showing options like 'Open', 'Save (Ctrl+S)', 'Save With Sorting', 'Save model as .. (Ctrl+Shift+S)', 'Close', and 'Exit'. The main window on the right features a 'Start' section with a 'Welcome to Olex2!' message, a 'CHANGELOG' link, and an 'Open' button. Below this are buttons for 'Sucrose', 'THPP', 'Co110', 'ZP2', 'Water', '183', and 'Timmy'. A 'Documentation' section provides links for 'Online', 'Static PDF', and 'All Inline Help'. Further down are sections for 'Tutorials', 'Extension Modules', 'Settings', and 'News'. The 'News' section includes a citation for Dolomanov et al. (2009) and a large graphic titled 'Setting up Olex2' with the text '... and everything else you need to get going' and the OlexSys logo.

File Edit View Structure Mode Tools Model Select Help

Open

C:\Users\Lou\Desktop\CCCW17\Olex2 Workshop\Example 0 - Structure Solution\Example0  
C:\Users\Lou\Desktop\Maly\_June\_2017\Maly\_June\_2017\n16053 Report Stuff\n16053\_CIF.cif  
C:\Users\Lou\Desktop\Old Data\b16146\June\_2017\Crystal\_1\b16146\_crystal1  
C:\Users\Lou\Desktop\Old Data\b16146\June\_2017\Crystal\_1\b16146\_crystal1.p4p  
C:\Users\Lou\Desktop\Old Data\b16146\June\_2017\Crystal\_2\b16146\_crystal2  
C:\Users\Lou\Desktop\Old Data\b16146\June\_2017\Crystal\_2\b16146\_crystal2.p4p  
C:\Users\Lou\Desktop\Old Data\b16146\twin\b16146  
C:\Users\Lou\Desktop\Old Data\b16146\twin\b16146.p4p

Save (Ctrl+S)  
Save With Sorting  
Save model as .. (Ctrl+Shift+S)  
Close  
Exit

Loading AutoChem\_40 (Version Sun May 20 16:49:29 2018)Welcome to Olex2

We are grateful to our users for testing and supporting Olex2  
Please find the link to credits in the About box

Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H.,  
OLEX2: A complete structure solution, refinement and analysis program (2009).  
J. Appl. Cryst., 42, 339-341.

Loading HARP (Version Sun May 20 16:49:43 2018)OK.  
File  
is  
closed  
>>

Olex2

Start

Welcome to Olex2! [CHANGELOG](#) [Open](#)

[Sucrose](#) [THPP](#) [Co110](#) [ZP2](#) [Water](#) [183](#) [Timmy](#)

Documentation: [Online](#) | [Static PDF](#) | [All Inline Help](#)

Tutorials

Extension Modules

Settings

News

Please cite us in your publications:  
Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H., *OLEX2: A complete structure solution, refinement and analysis program* (2009). J. Appl. Cryst., 42, 339-341.

Setting up Olex2  
... and everything else you need to get going

OlexSys



# OLEX<sup>2</sup>

Home **Work** View Tools Info

## Start

Welcome to Olex2! [CHANGELOG](#) [Open](#)

[Sucrose](#) [THPP](#) [Co110](#) [ZP2](#) [Water](#) [183](#) [Timmy](#)

Documentation: [Online](#) | [Static PDF](#) | [All Inline Help](#)

Tutorials

Extension Modules

Settings

## News

**Please cite us in your publications:**

Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H., *OLEX2: A complete structure solution, refinement and analysis program* (2009). *J. Appl. Cryst.*, **42**, 339-341.

## Setting up Olex2

... and everything else you need to get going



Select the program that you want to use to solve your structure. ShelxT has become very popular, and we will use it today, but there are other powerful ways to solve structures, especially if you are encountering problems.

Sometimes you may want to look at other possible spacegroups (you won't have Rigaku's Explain as a possibility; Bruker users could also do this in XPREP)

Loading AutoChem\_40 (Version Sun May 20 16:49:29 2018)Welcome to Olex2

We are grateful to our users for testing and supporting Olex2  
Please find the link to credits in the About box

Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H.,  
OLEX2: A complete structure solution, refinement and analysis program (2009).

The screenshot shows the Olex2 software interface. At the top, the title bar reads 'Olex2'. Below it is a menu bar with 'File', 'Edit', 'View', 'Structure', 'Mode', 'Tools', 'Model', 'Select', and 'Help'. The main window displays a project titled 'n18060' with the chemical formula  $C_{12}H_{36}Cl_4Co_2O_6S_6$ . The interface is divided into several sections: a top status bar with unit cell parameters (a, b, c,  $\alpha$ ,  $\beta$ ,  $\gamma$ , Z, Z', V) and R/R<sub>2</sub> values; a middle section with tabs for 'Home', 'Work', 'View', 'Tools', and 'Info'; and a bottom section with tabs for 'Solve', 'Refine', 'Draw', and 'Report'. The 'Solve' tab is active, and a dropdown menu is open, showing the following options: 'ShelXT', 'ShelXD', 'ShelXS', 'ShelXT', and 'olex2.solve'. A red arrow points from the text 'Once you have selected your method for solving the structure, hit "Solve"' to the 'Solve' button. Another red arrow points from the text 'Sometimes you may want to look at other possible spacegroups (you won't have Rigaku's Explain as a possibility; Bruker users could also do this in XPREP)' to the 'Space Group' dropdown menu, which currently shows 'P-1'.

Parameter	Value
a	8.9018(2)
b	9.4275(2)
c	18.2484(4)
$\alpha$	81.945(2)°
$\beta$	81.395(2)°
$\gamma$	89.4290(10)°
Z	2
Z'	1
V	1499.16(6)

Parameter	Value
d min (Cu)	0.84
Shift	0.001
Max Peak	0.4
Min Peak	-0.4
R <sub>1</sub>	4.32 %
wR <sub>2</sub>	8.90 %
complete	96 %
Goof	1.144

Once you have selected your method for solving the structure, hit "Solve"

## ShelXT results



```

File Edit View Structure Model Tools Model Select Help

C
an [E^2-1] 0kl 0.929 h01 0.942 hk0 0.984 Rest 0.879
547 Reflections with E < 0.240 employed for R(weak)

40 unique Patterson peaks with heights greater than 77 selected as
position vectors

Setup: 0.657 secs

Try N(iter) CC R(weak) CHEM CFOM best Sig(min) N(P1) Vol/N
1 100 94.84 0.0597 0.7500 0.8886 2.492 59 25.41
2 100 94.58 0.0727 0.4545 0.8731 0.8886 2.340 60 24.99
3 100 94.55 0.0707 0.8333 0.8748 0.8886 2.441 58 25.85
4 100 94.26 0.0635 1.0000 0.8791 0.8886 2.016 57 26.30

4 attempts, solution 1 selected with best CFOM = 0.8886, Alpha0 = 0.057

Structure solution: 1.547 secs

1 Centrosymmetric and 1 non-centrosymmetric space groups evaluated

Space group determination: 0.013 secs

R1 Rweak Alpha Orientation Space group Flack_x File Formula
0.103 0.016 0.056 as input P-1 n18060_a C13 C19 Co2 O6 S
0.091 0.015 0.000 as input P1 0.49 n18060_b C21 C113 Co4 O13 S8

Assign elements and isotropic refinement 2.962 secs

+++++
+ SHELXT finished at 11:33:28 Total time: 5.178 secs +
+++++

```

**n18060** P1  
 C:\Users\Lou\Desktop\CCCW18\Intro\n18060.res

**C<sub>12</sub>H<sub>36</sub>Cl<sub>4</sub>Co<sub>2</sub>O<sub>6</sub>S<sub>6</sub>**

a = 8.9018(2) α = 81.945(2)° Z = 2  
 b = 9.4275(2) β = 81.395(2)° Z' = 1  
 c = 18.2484(4) γ = 89.4290(10)° V = 1499.16(6)

**Solution**

d min (Cu)	0.84	I/σ	32.9	Rint	7.24%	complete	96%
Shift	0.001	Max Peak	0.4	Min Peak	-0.4	Goof	1.144

Home Work View Tools Info

Solve Refine Draw Report

**ShelXT**

R1	Rweak	Alpha	Orientation	Space group	Flack_x
0.103	0.016	0.056	as input	P-1	---
0.091	0.015	0.000	as input	P1	0.49

Program: ShelXT Method: Intrinsic Phasing

Reflections: n18060.hkl Mon May 21 11:33:28 2018

Composition: C12 H36 Cl4 Co2 O6 S6 25.0 Å³ Z = 2 Z' = 1

Space Group: [Rigaku XPlain] [Olex2] P-1

Run Rigaku XPlain GUI

Solution Settings Extra

Toolbox Work

Labels: Labels OFF/ON

C H Cl Co O S ... Add H

But where is my solution?

Mouse: Hold down the right click button and drag your mouse forward (up); do not scroll

Does this solution make sense? Go back to your reaction.

A few things to try:

Fn F4

Ctrl T

Type “grow”

Type “fuse”

Go to “Refine” arrow  
(Don’t hit “Refine” yet!)

The screenshot shows the n18060 software interface. The title bar indicates the file path: C:\Users\Lou\Desktop\CCCW18\Intro\n18060.res. The main window displays the chemical formula C12H36Cl4Co2O6S6 and various crystallographic parameters. A blue arrow points to the 'Refine' button in the 'Work' tab, which is highlighted in orange. The 'Refine' button is part of a row of buttons including 'Solve', 'Draw', and 'Report'. Below the buttons, the 'Program' is set to 'ShelXT', the 'Method' is 'Intrinsic Phasing', and the 'Space Group' is 'P-1'. The 'Toolbox Work' section at the bottom shows 'Labels OFF/ON' and a list of elements: C, H, Cl, Co, O, S, and a button to 'Add H'.

a		b		c		α		β		γ		Z		Z'		V	
8.9018(2)	81.945(2)°	9.4275(2)	81.395(2)°	18.2484(4)	89.4290(10)°	2	1	1499.16(6)									

d min (Cu)	0.84	I/σ	32.9	Rint	7.24%	complete	96%
Shift	0.001	Max Peak	0.4	Min Peak	-0.4	Goof	1.144

Disagree with the preliminary atom assignments?

Click on atoms you would like to reassign, and click on the identity that you think they should be.

(The “...” option will allow you to select atom types that were not included in your original formula.)

Do you see atoms that shouldn't be there?

Click on them and hit “Delete” on your keyboard.

Note: Sometimes it can be tricky to select atoms (like in this case.) You can also select bonds and delete those (this makes selecting the atom easier.) You can also hold down Shift and your left mouse button to draw a box around the atoms you want to select.

**n18060**  
C:\Users\Lou\Desktop\CCCW18\Intro\n18060.res

C12H36Cl4Co2O6S6

$a = 8.9018(2)$   $\alpha = 81.945(2)^\circ$   $Z = 2$   
 $b = 9.4275(2)$   $\beta = 81.395(2)^\circ$   $Z' = 1$   
 $c = 18.2484(4)$   $\gamma = 89.4290(10)^\circ$   $V = 1499.16(6)$

**Solution**

d min (Cu)	0.84	I/ $\sigma$	32.9	Rint	7.24%	complete	96%
Shift	0.001	Max Peak	0.4	Min Peak	-0.4	Goof	1.144

Home Work View Tools Info

Solve Refine Draw Report

Program: ShelXL Least Squares Cycles: 10 Peaks: 20

hkl file: n18060.hkl hkl: Mon May 21 11:33:28 2018

Weight: ☐ EXT: ☐ NOACTA

☐ Use Solvent Mask This is the Olex2 implementation of BYPASS (a.k.a. SQUEEZE)

Refinement Settings Extra

Toolbox Work

Labels: Labels OFF/ON

C H Cl Co O S ... Add H

q to q to H x C F O

Select atom(s) and then mFit mSplit Split SAME Split

MAP Show Map Map Settings

Peak & Uiso Sliders

Growing

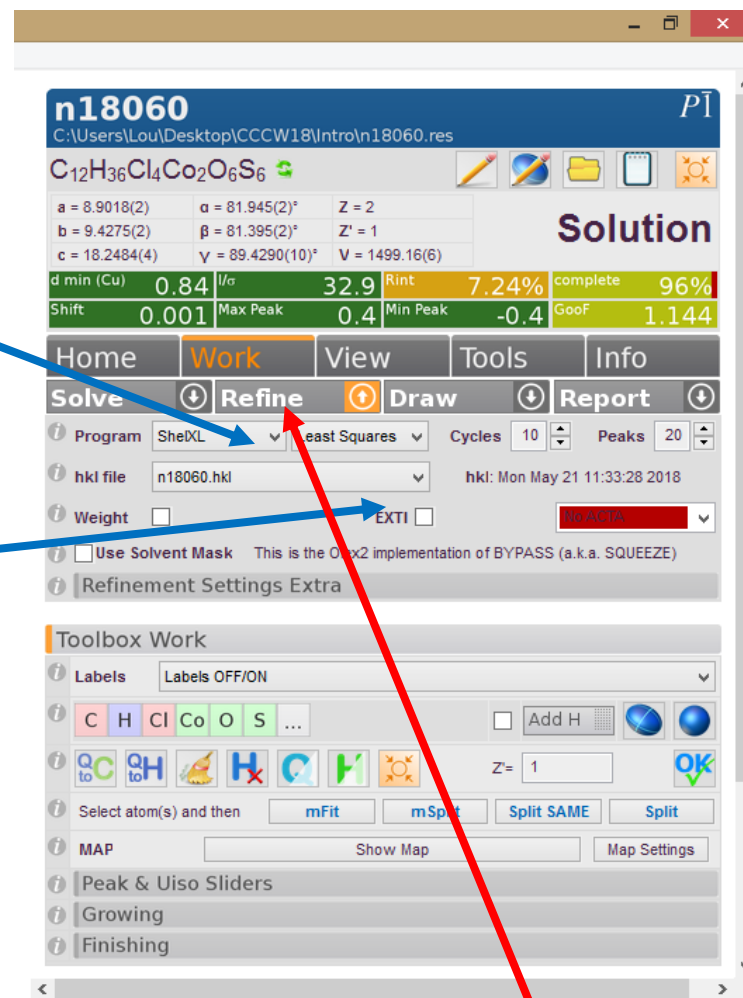
Finishing

**IN OLEX2 THERE ARE MANY, MANY WAYS TO ACHIEVE THE SAME GOAL! SHARE YOUR TIPS! :-D**



Select your refinement program (ShelXL)

Note that it has selected the reflection file with the same file name; if you have other reflection files in this folder, you can select the one you want to use.



If you feel like you are in a good place to get started with your refinement, hit "Refine"

How are things looking?

Ctrl T

Ctrl Q

Hover over a Q peak.

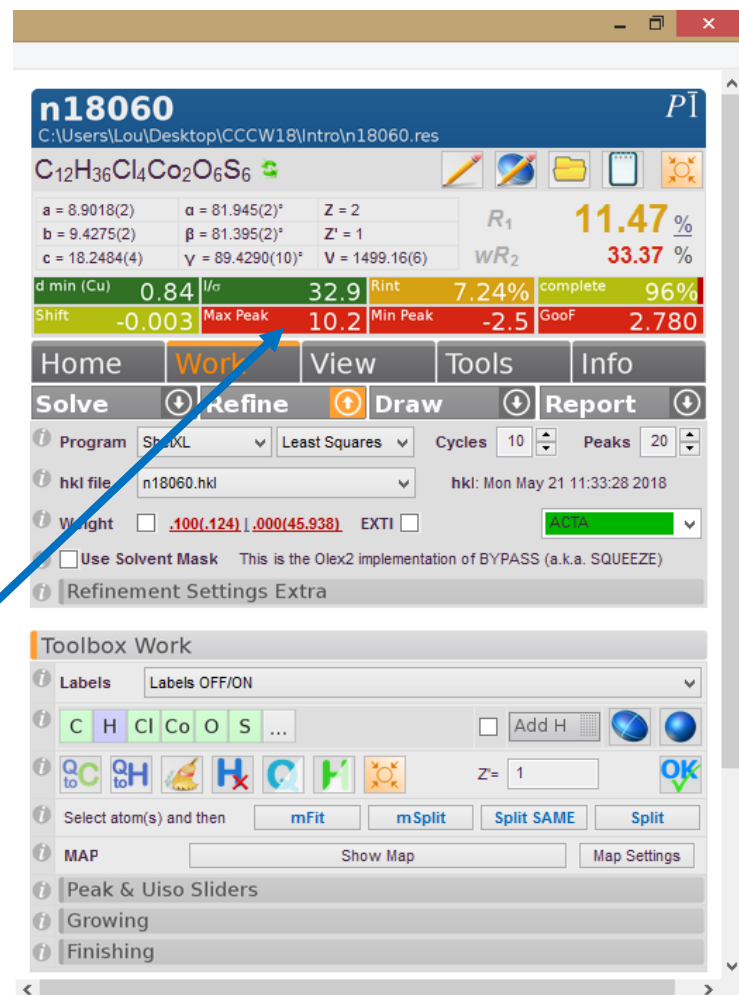
Scroll down on your mouse and the smallest peaks will start to be hidden.

If you would like to find your largest Q peak without hiding the others:

1. Intensity of the Q peak colour corresponds to size
2. Hitting this will highlight the maximum peak.
3. “Fn F3” will label everything

What’s up with that largest peak?

Also, those labels are terrible.



When you feel confident that all non-hydrogen atoms are in your model, but before you attempt to treat any disorder, you should give your atoms sensible names.

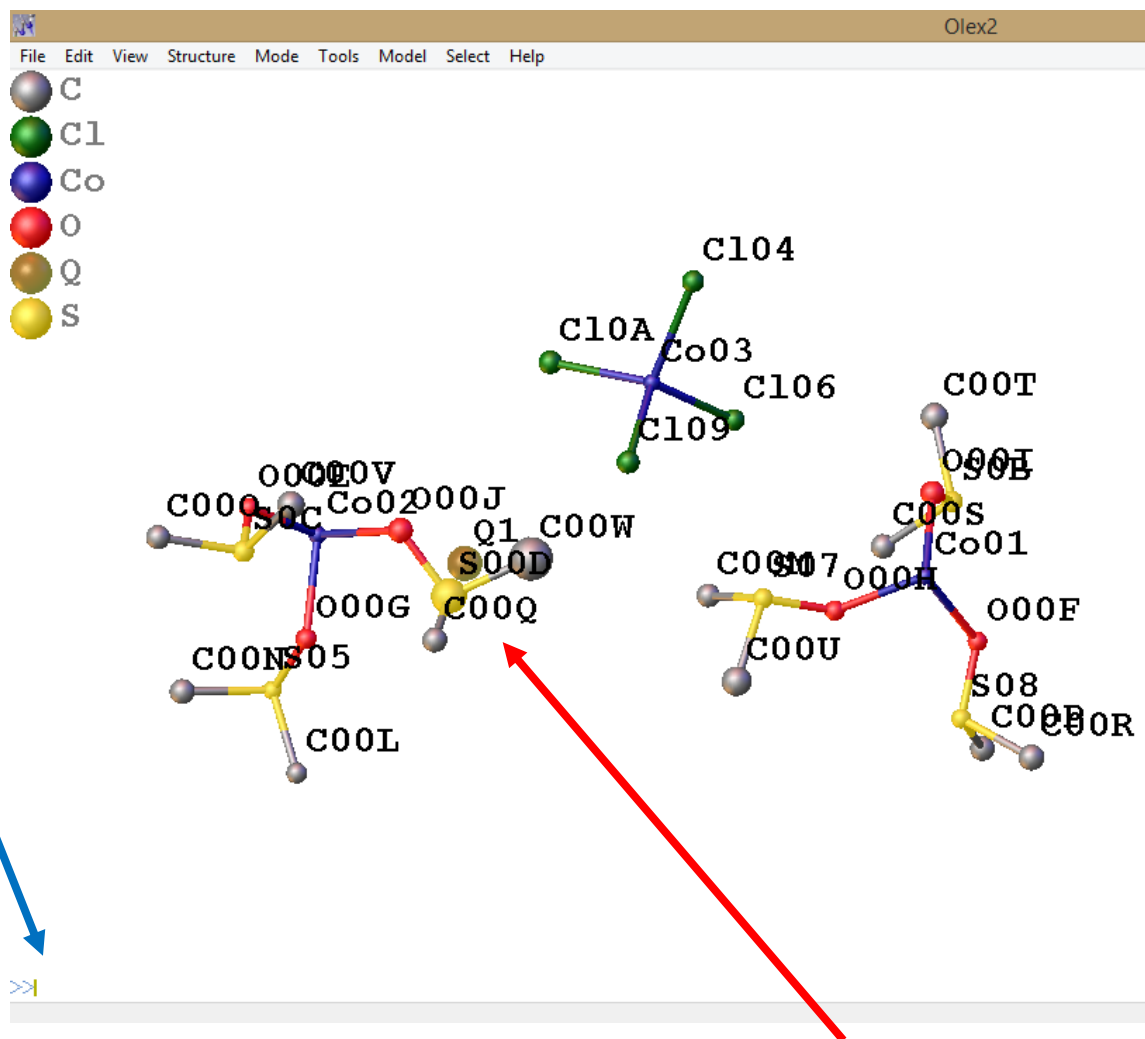
“Ctrl Q” to hide your Q peaks

Click on your cobalt atoms in the order that you would like to number them

At the prompt, type “Name 1”

Repeat for all atom types

Note: You don't have to start with 1 as your first number. For example, if you want to switch just part of a numbering sequence, select the atoms that are involved, and the number you want to start with.



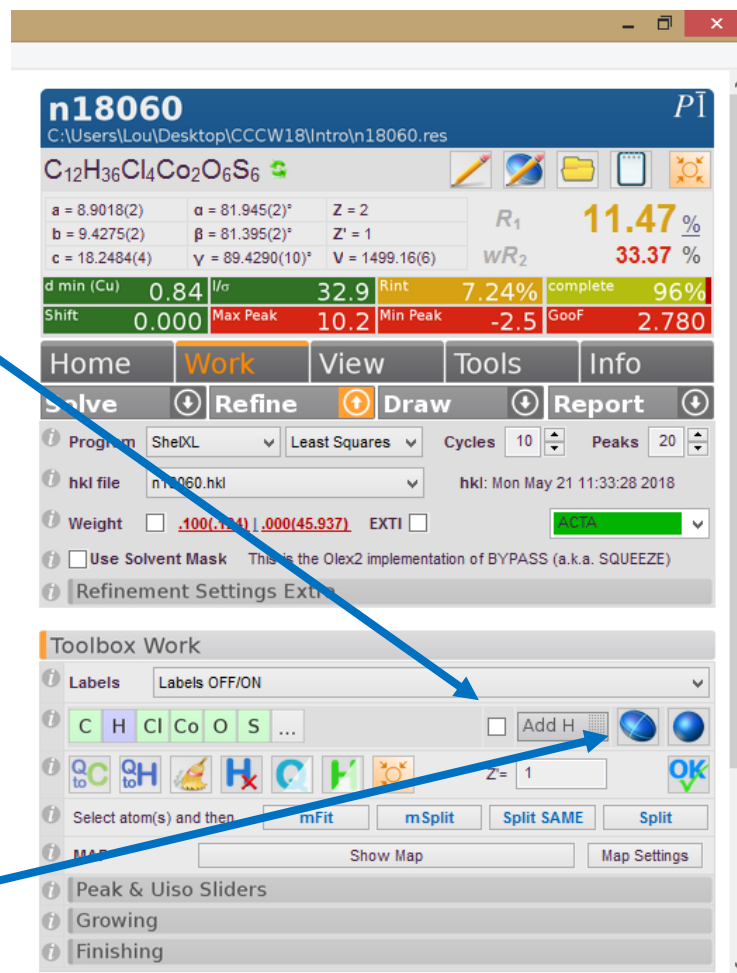
Names okay? “Refine”

So far we’ve been refining everything isotropically, but we’ll want to switch to anisotropic refinement.

But first! Make sure this button is NOT selected.

Then, hit the anisotropic ellipsoid to change how each atom is refined.

Everything okay so far? Hit “Refine”



Now how do things look?

How are your refinement values?

Where is that max peak?

Look at your Q peaks; do they make you think about hydrogen?

You can add hydrogen atoms into calculated positions by hitting the “Add H” button.

If your data is good enough, and especially if you have hydrogen atoms involved in H-bonds, you will likely want to introduce them in their difference map positions and refine them (possibly with the use of restraints.)

“Refine”

The screenshot displays the Olex2 software interface for project n18060. The top section shows the chemical formula C12H36Cl4Co2O6S6 and unit cell parameters:  $a = 8.9018(2)$ ,  $b = 9.4275(2)$ ,  $c = 18.2484(4)$ ,  $\alpha = 81.945(2)^\circ$ ,  $\beta = 81.888(2)^\circ$ ,  $\gamma = 89.4290(10)^\circ$ ,  $Z = 2$ ,  $V = 1499.16(6)$ . Refinement statistics are shown as  $R_1 = 9.97\%$  and  $wR_2 = 30.09\%$ . A table of peaks is visible, with the maximum peak at  $d \min (\text{Cu}) = 0.84$  and  $\text{Shift} = 2.297$ . The interface includes tabs for Home, Work, View, Tools, and Info, and buttons for Solve, Refine, Draw, and Report. The 'Add H' button is highlighted in the 'Toolbox Work' section. The 'Refine' button is also visible in the top navigation bar.

I see problems

1. Should all those hydrogens be there? Go back to your reaction conditions and think about charge balance.
2. Hydrogen atoms can “mask” disorder by mopping up electron density.

Go back and delete any hydrogen atoms that are relevant to these two points (click on them and hit “Delete” on your keyboard.)

“Refine”

My refinement values improved. Did yours? This wasn't a sure thing since points 1 and 2 were likely to off-set each other (ie. Point 1 meant that there were H-atoms introduced where none existed but point 2 meant that some H-atoms might have been used to account for electron density associated with disorder.)

Before we start dealing with disorder, let's do a few routine “things”.

At the prompt type “edit lst”

First, the atoms are not in a sensible order.

Close the .lst and sort your atoms

First, click on “Sorting” (if this menu isn’t open)

Next, decide on your sort order (I’ll just use the default) and hit “Sort”

“Refine”

The screenshot displays the Olex2 software interface. At the top, a status bar shows unit cell parameters:  $a = 8.9018(2)$ ,  $b = 9.4275(2)$ ,  $c = 18.2484(4)$ ,  $\alpha = 81.945(2)^\circ$ ,  $\beta = 81.395(2)^\circ$ ,  $\gamma = 89.4290(10)^\circ$ ,  $Z = 2$ ,  $Z' = 1$ , and  $V = 1499.16(6)$ . It also shows  $R_1 = 8.51\%$  and  $wR_2 = 27.69\%$ . Below this, a table of refinement statistics is visible:  $d \min (Cu) = 0.84$ ,  $I/\sigma = 32.9$ ,  $Rint = 7.24\%$ ,  $complete = 96\%$ ,  $Shift = 0.005$ ,  $Max Peak = 4.7$ ,  $Min Peak = -4.9$ , and  $Goof = 2.307$ . The main menu bar includes Home, Work, View, Tools, and Info. Below it, the Solve, Refine, Draw, and Report tabs are active. The Refine tab is selected, showing settings for Program (ShelXL), Least Squares, Cycles (10), and Peaks (20). The hkl file is n18060.hkl. The Weight section shows  $.100(.049) | .000(37.403)$  and EXTI. The Use Solvent Mask checkbox is checked. The Refinement Settings Extra section is also visible. The Toolbox Work section includes History, Select, Naming, and Sorting. The Sorting menu is open, showing Sort order (Part, Mass, Label, None), Moiety (From sort), and Specific order (Atoms, Moieties). The Sort button is highlighted with a blue arrow.

Parameter	Value
$a$	$8.9018(2)$
$b$	$9.4275(2)$
$c$	$18.2484(4)$
$\alpha$	$81.945(2)^\circ$
$\beta$	$81.395(2)^\circ$
$\gamma$	$89.4290(10)^\circ$
$Z$	2
$Z'$	1
$V$	$1499.16(6)$
$R_1$	8.51 %
$wR_2$	27.69 %

Statistic	Value
$d \min (Cu)$	0.84
$I/\sigma$	32.9
$Rint$	7.24 %
complete	96 %
Shift	0.005
Max Peak	4.7
Min Peak	-4.9
Goof	2.307

Is "ACTA" selected? (This will generate a .cif)

Are you up-dating your weighting? (I've been waiting until I deal with my disorder, because I know I don't yet have my best model.

What will "Info" reveal?

The screenshot shows the Olex2 software interface. The 'Work' tab is active, displaying a table of refinement statistics. The 'Info' panel on the right shows the 'ACTA' option selected in the 'Weighting' section. A red arrow points from the 'Info' tab in the top navigation bar to the 'Info' panel. A blue arrow points from the 'ACTA' option in the 'Weighting' section to the 'Info' panel.

a = 8.9018(2)		α = 81.945(2)°		Z = 2		R <sub>1</sub> 8.51 %	
b = 9.4275(2)		β = 81.395(2)°		Z' = 1		wR <sub>2</sub> 27.69 %	
c = 18.2484(4)		γ = 89.4290(10)°		V = 1499.16(6)			
d min (Cu)	0.84	I/σ	32.9	Rint	7.24%	complete	96%
Shift	0.000	Max Peak	4.7	Min Peak	-4.9	Goof	2.307

Home Work View Tools Info

Solve Refine Draw Report

Program: ShelXL, Least Squares, Cycles: 10, Peaks: 20

hkl file: n18060.hkl, hkl: Mon May 21 11:53:28 2018

Weighting: ☒ .100(.040), .000(37.407), EXTI ☐ ACTA

Use Solvent Mask: This is the Olex2 implementation of BYPASS (a.k.a. SQUEEZE)

Refinement Settings Extra

Toolbox Work

History

Select

Naming

Sorting

Sort order: Part, Mass, Label, None

Moiety: From sort, Treat H atoms independently

Specific order: Atoms, Reorder, Move to first, Moieties, Sort

c = 18.2484(4)		V = 89.4290(10)°		V = 1499.16(6)		WR2		27.69		/0	
d min (Cu)	0.84	I/σ	32.9	Rint	7.24%	complete	96%				
Shift	0.000	Max Peak	4.7	Min Peak	-4.9	Goof	2.307				

Home Work View Tools Info

- Recent Files
- Electron Density Peaks
- Refinement Indicators
- Bad Reflections
- Reflection Statistics
- Reflection Statistics Summary

More info in our .lst!



At the prompt type “edit lst”

In the text file, search for

“disagreeable”

“split”

“Highest peak”

There is so much great information in this file!

Okay, at this point, we can either model this disorder, or leave that to someone else...

What time is it?

In your main screen “Ctrl T” until you have a blank background and “Ctrl Q” to show your Q-peaks, but scroll down on your mouse so that only the two highest ones are displayed.

Open Toolbox Work by clicking on it

There are multiple ways to treat disorder in OLEX2; you can click on the “i” to get info about the various settings that you can apply.

I recommend deleting the H-atoms on the C that is involved in the disorder.

Select the C and S that are involved in disorder, and hit “Split”, leaving the other settings as shown.

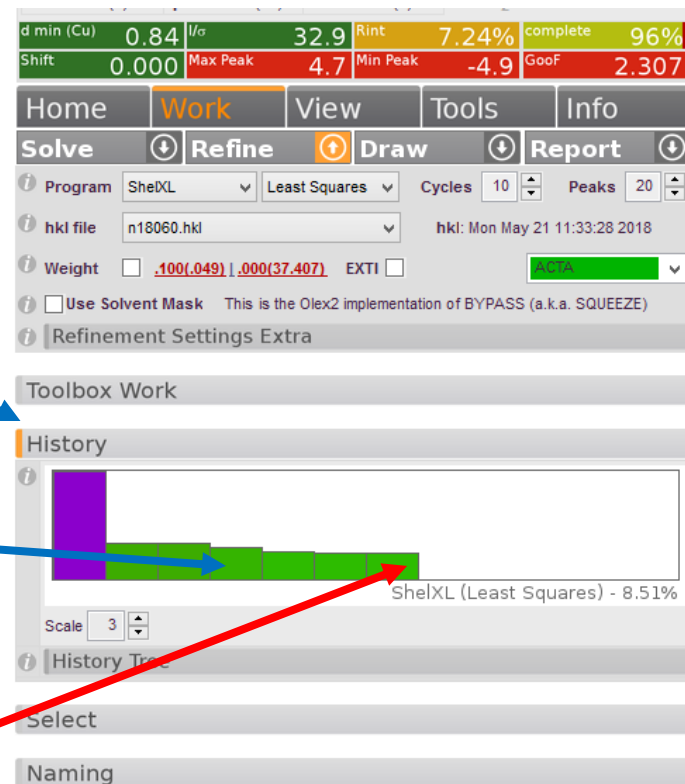
Hover your mouse over the group you want to split, and hold “Ctrl” and your left mouse button while moving your mouse around until the second component fits the two highest Q peaks

The screenshot displays the Olex2 software interface. The top panel shows the title 'n18060' and the file path 'C:\Users\Indaw\OneDrive - Wilfrid Laurier University\Desktop\Quarantine\CCCW22\Guest'. Below this, the chemical formula  $C_{12}H_{36}Cl_4Co_2O_6S_6$  is shown. The unit cell parameters are listed:  $a = 8.9018(2)$ ,  $b = 9.4275(2)$ ,  $c = 18.2484(4)$ ,  $\alpha = 81.945(2)^\circ$ ,  $\beta = 81.395(2)^\circ$ ,  $\gamma = 89.429(1)^\circ$ ,  $Z = 2$ ,  $Z' = 1$ , and  $V = 1499.16(6)$ . The  $R_1$  value is 8.47% and the  $wR_2$  value is 27.61%. The  $d_{min}(Cu/a)$  is 0.84,  $2\theta_{min}$  is 132.7°,  $I/\sigma(I)$  is 32.9,  $R_{int}$  is 7.24%,  $Full\ 132.7^\circ$  is 96.4%,  $Shift$  is -1.280,  $Max\ Peak$  is 4.8,  $Min\ Peak$  is -4.9, and  $GooF$  is 2.300. A warning message states: 'Warning: 2 atoms may be split and 0 atoms NPD'. The main panel has tabs for Home, Work, View, Tools, and Info. The Work tab is active, showing the Solve, Refine, Draw, and Report buttons. The Program is set to ShelXL, L.S. is selected, Cycles is 5, and Peaks is 25. The hkl file is n18060.hkl, and the hkl date is Wed Jun 8 21:00:27 2022. The Weight is set to .100 | .047 | .000 | 36.786, and the Use Solvent Mask checkbox is checked. The Refinement Settings Extra section is visible. The Toolbox Work panel is also shown, with the Labels section set to Labels OFF/ON. The C, H, Cl, Co, O, S atoms are listed, and the Z' value is 1. The MAP section has Show Map and Map Settings buttons. The Disorder Tools section has Fit and Split buttons, with SAME selected for PART, auto for FVAR, and auto for FVAR. The Show/Hide the list of restraints checkbox is checked, and No Restraints are shown. The Peak & Uiso Sliders section is visible, and the Growing section is at the bottom.

If you aren't happy about how these two atoms were split, you can always go back in history to an earlier model.

Just click on any of the Histogram bars, and the model that corresponded to that round of least squares will pop back up on your screen!

If you just don't like how you split your atoms, and want a second try at it, hit the last bar.



You should notice that after you split your atoms they now look like they are being refined isotropically (because they are; anisotropic refinement can also “mop up” electron density, so we refine disorder isotropically first.) Notice the nice work OLEX2 did naming the second disorder component?

You should also notice that the H-atoms on the “not disordered” carbon disappeared. Why do you think that is?

Peter Mueller would tell you that you will never refine disorder without restraints. Let's open our .ins to see what was added.

```
File Edit Format View Help
TITL n18060_a.res in P-1
REM Old TITL n18060_a.res in P-1
REM SHELXT solution in P-1: R1 0.103, Rweak 0.016, Alpha 0.063
REM <I/s> 0.000 for 0 systematic absences, Orientation as input
REM Formula found by SHELXT: C13 C19 Co2 O6 S
CELL 1.54184 8.9018 9.4275 18.2484 81.945 81.395 89.429
ZERR 2 0.0002 0.0002 0.0004 0.002 0.002 0.001
LATT 1
SFAC C H Cl Co O S
UNIT 24 84 8 4 12 12
SADI S6 O6 S6A O6
SADI S6 C11 S6A C11
SADI S6 C12 S6A C12A
SADI 0.04 O6 C12 O6 C12A
SADI 0.04 C11 C12 C11 C12A

L.S. 5
PLAN 25
CONF
BOND
list 4
MORE -1
BOND $H
fmap 2 53
acta
WGHT 0.1
FVAR 0.46789 0.6441
REM <olex2.extras>
REM <HklSrc "%.\n18060.hkl">
REM </olex2.extras>
```

Peter says it here:

<https://www.tandfonline.com/doi/pdf/10.1080/08893110802547240?needAccess=true>

We can add additional restraints and constraints.

First, open up the “Tools” menu

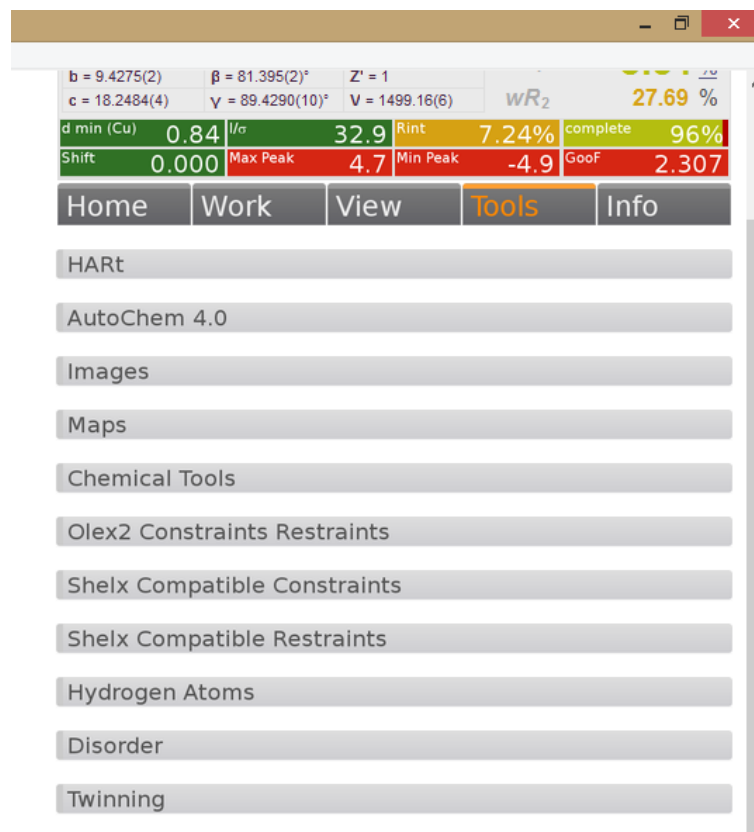
Then, open up “Shelx Compatible Restraints” or “Constraints”

You should not simultaneously refine occupancy and displacements (at least on your first round of least squares).

We can apply an EADP constraint to force selected displacements to be identical.

Later, we can edit these constraints to replace with RIGU restraints.

Shelx instructions! <http://shelx.uni-goettingen.de/shelxl.html.php>



The screenshot shows the Shelx software interface. At the top, a status bar displays various parameters:  $b = 9.4275(2)$ ,  $\beta = 81.395(2)^\circ$ ,  $Z' = 1$ ,  $c = 18.2484(4)$ ,  $\gamma = 89.4290(10)^\circ$ ,  $V = 1499.16(6)$ ,  $wR_2 = 27.69\%$ ,  $d \min (Cu) = 0.84$ ,  $I/\sigma = 32.9$ ,  $R_{int} = 7.24\%$ ,  $complete = 96\%$ ,  $Shift = 0.000$ ,  $Max Peak = 4.7$ ,  $Min Peak = -4.9$ , and  $Goof = 2.307$ . Below the status bar is a menu bar with 'Home', 'Work', 'View', 'Tools' (highlighted in orange), and 'Info'. The 'Tools' menu is open, showing a list of options: 'HART', 'AutoChem 4.0', 'Images', 'Maps', 'Chemical Tools', 'Olex2 Constraints Restraints', 'Shelx Compatible Constraints', 'Shelx Compatible Restraints' (highlighted in grey), 'Hydrogen Atoms', 'Disorder', and 'Twinning'.

Parameter	Value
$b$	$9.4275(2)$
$\beta$	$81.395(2)^\circ$
$Z'$	1
$c$	$18.2484(4)$
$\gamma$	$89.4290(10)^\circ$
$V$	$1499.16(6)$
$wR_2$	27.69 %
$d \min (Cu)$	0.84
$I/\sigma$	32.9
$R_{int}$	7.24 %
complete	96 %
Shift	0.000
Max Peak	4.7
Min Peak	-4.9
Goof	2.307

RIGU!

[http://journals.iucr.org/a/issue\\_s/2012/04/00/pc5011/pc5011.pdf](http://journals.iucr.org/a/issue_s/2012/04/00/pc5011/pc5011.pdf)

If everything looked stable, hit your anisotropic button again, and “Refine”. If things still look good, you will want to add H-atoms.

This time, just select the carbon atoms in the disordered group (if you hit “Add H” before doing this, it will add back in the O-H hydrogen atoms that you already deleted.)

Notice how the carbon atom that was not split now has six hydrogens on it? Let’s see how this makes sense by selecting to view only one “part” at a time.

You can also select to update your weights now.

The screenshot displays the Olex2 software interface for a refinement project named 'n18060'. The top panel shows the chemical formula C12H36Cl4Co2O6S6 and various refinement statistics. The 'Work' tab is active, showing the 'Refine' button highlighted. The 'Toolbox Work' panel at the bottom shows the 'Labels' dropdown set to 'Labels OFF/ON'. The 'Add H' button is visible, and the '0 1' and '0 2' buttons are selected. A red arrow points from the text 'You can also select to update your weights now.' to the 'Weights' field in the 'Refine' panel, which shows the current weights as  $.100(.045) | .000(3.462)$ . A blue arrow points from the text 'Let’s see how this makes sense by selecting to view only one “part” at a time.' to the '0 1' and '0 2' buttons in the 'Toolbox Work' panel.

a = 8.9018(2)		α = 81.945(2)°		Z = 2	
b = 9.4275(2)		β = 81.395(2)°		Z' = 1	
c = 18.2484(4)		γ = 89.4290(10)°		V = 1499.16(6)	

d min (Cu)	0.84	I/σ	32.9	Rint	7.24%	complete	96%
Shift	0.000	Max Peak	0.5	Min Peak	-1.1	Goof	1.154

Home Work View Tools Info

Solve Refine Draw Report

Program: ShelXL, Least Squares, Cycles: 10, Peaks: 20

hkl file: n18060.hkl, hkl: Mon May 21 11:33:28 2018

Weight:  $.100(.045) | .000(3.462)$ , EXTI: ☐

☐ Use solvent Mask, This is the Olex2 implementation of BYPASS (a.k.a. SQUEEZE)

Refinement Settings Extra

Toolbox Work

Labels: Labels OFF/ON

C H Cl Co O S ... Add H

0 1 0 2 All Sel ☒ Unique ☒ Labels

Select atom(s) and then: mFit mSplit Split SAME Split

MAP: Show Map Map Settings

Peak & Uiso Sliders

Growing

In this tutorial I have deliberately not included screenshots of my model because I didn't want to give away the answer!

I've posted this as a file that you can edit. Why don't you go back, work through it again, and add your own notes and screenshots? You can start from scratch by going to Model → Reset

The screenshot displays the Olex2 software interface. On the left, a legend identifies atoms by color: Carbon (grey), Hydrogen (white), Chlorine (green), Cobalt (blue), Oxygen (red), and Sulfur (yellow). The main menu bar includes File, Edit, View, Structure, Mode, Tool, Model, Select, and Help. The 'Model' menu is open, showing options: Refine (Ctrl+R), Move, Q-->C, Tidy, ANIS All, ISOT All, ANIS Selected, ISOT Selected, Reset, and Solve. A red arrow points from the text 'Model → Reset' to the 'Reset' option in the menu. The main workspace shows a 3D ball-and-stick model of a complex molecule. On the right, a panel displays the file name 'n18060' and its path. Below this, the chemical formula C12H36Cl4Co2O6S6 is shown. A table of unit cell parameters and quality indicators is provided:

Unit Cell Parameters		Quality Indicators	
$a = 8.9018(2)$	$\alpha = 81.945(2)^\circ$	$Z = 2$	$R_1 = 4.36\%$
$b = 9.4275(2)$	$\beta = 81.395(2)^\circ$	$Z' = 1$	$wR_2 = 9.01\%$
$c = 18.2484(4)$	$\gamma = 89.4290(10)^\circ$	$V = 1499.16(6)$	

Below the table, a summary of refinement statistics is shown:

Parameter	Value	Parameter	Value
$d \text{ min (Cu)}$	0.84	$I/\sigma$	32.9
Shift	0.000	$R_{\text{int}}$	7.24%
		Max Peak	0.5
		Min Peak	-0.4
		complete	96%
		Goof	1.155

The right panel also features a 'Toolbox Work' section with various options for labels, atom selection, and refinement settings.