

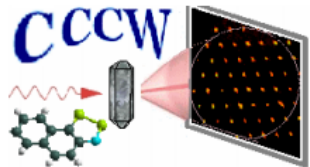
Canadian National Committee  
for Crystallography  
<http://xtallography.ca/>

## OLEX2 – Modeling disorder: Fixed positions

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

## Data from Charlotte Stern

Check out the OLEX2 youtube channel for an alternate way to tackle  
this:

<https://www.youtube.com/watch?v=LkIP0s-KEFQ>

Another good resource:

<http://web.mit.edu/pmueller/www/ACA2007/WK01/Disorder.pdf>

Navigate to SplittingCF3 folder.  
File → Open “Example5.ins”

The screenshot displays the Olex2 software interface. The top window shows the 'File' menu with 'Open' selected, displaying a list of files:

- C:\Users\Lou\Desktop\CCCW17\Olex2 Workshop\Example 0 - Structure Solution\Example0
- C:\Users\Lou\Desktop\Maly\_June\_2017\Maly\_June\_2017\16053 Report Stuff\16053\_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

The main window shows the Olex2 start screen with navigation buttons for 'Welcome to Olex2!', 'Documentation', 'Tutorials', 'Extension Modules', 'Settings', and 'News'. A large banner at the bottom reads 'Setting up Olex2 ... and everything else you need to get going' with the OlexSys logo.

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

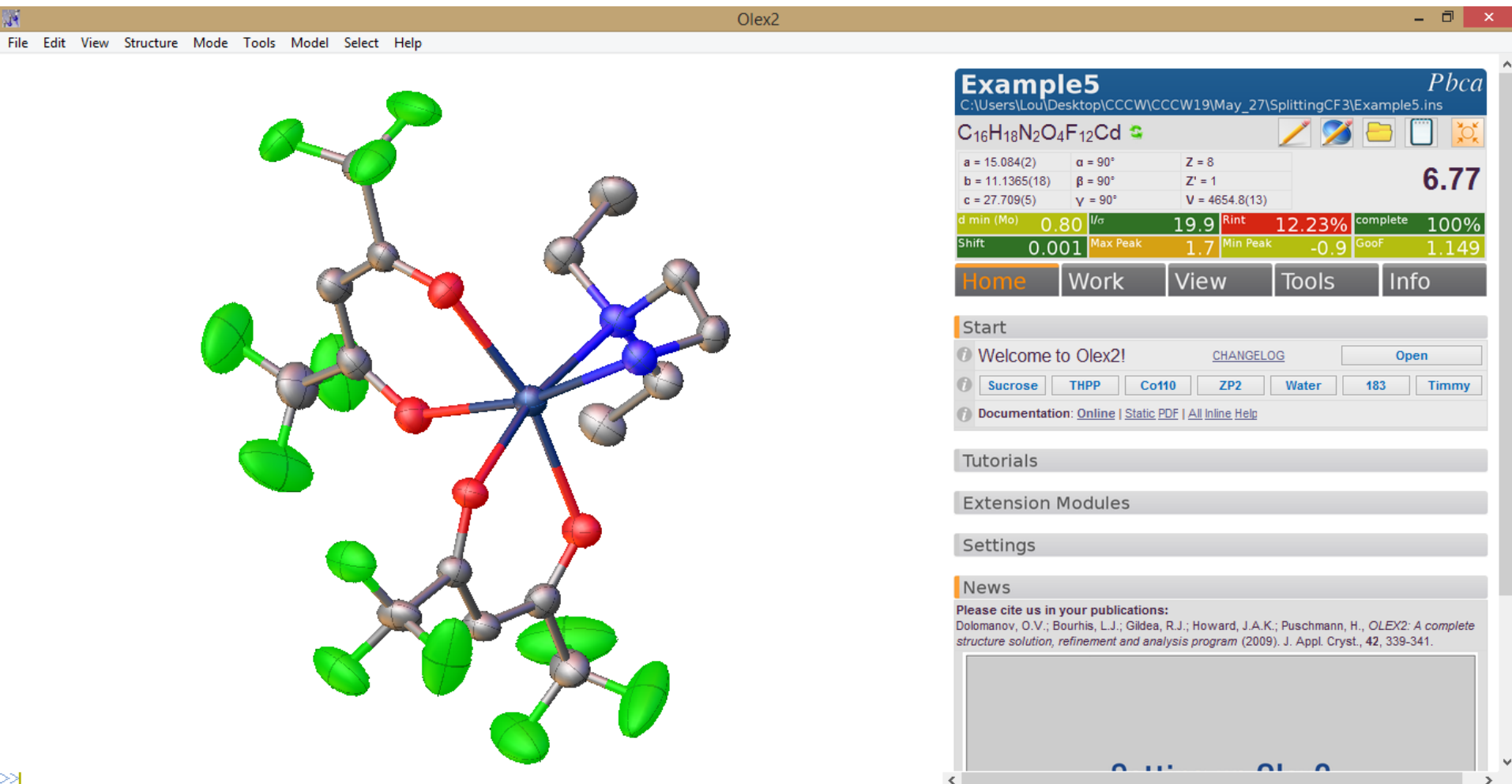
We are grateful to our users for testing and supporting Olex2  
Please find the link to credits in the About box

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  
>>|

Looks like all of our work is done! Coffee?

No hydrogens = No coffee



The screenshot displays the Olex2 software interface. On the left, a 3D ball-and-stick model of a complex molecule is shown, featuring a central blue atom coordinated to several red and grey atoms, with large green thermal ellipsoids representing non-hydrogen atoms. The right-hand side of the window contains a summary panel for 'Example5' with the following data:

**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCW\CCCW19\May\_27\SplittingCF3\Example5.ins  
C16H18N2O4F12Cd

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	<b>6.77</b>
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)	

d min (Mo)	0.80	I/ $\sigma$	19.9	Rint	12.23%	complete	100%
Shift	0.001	Max Peak	1.7	Min Peak	-0.9	Goof	1.149

Navigation tabs: Home | Work | View | Tools | Info

**Start**

Welcome to Olex2! [CHANGELOG](#)

Documentation: [Online](#) | [Static PDF](#) | [All In-line Help](#)

Tutorials

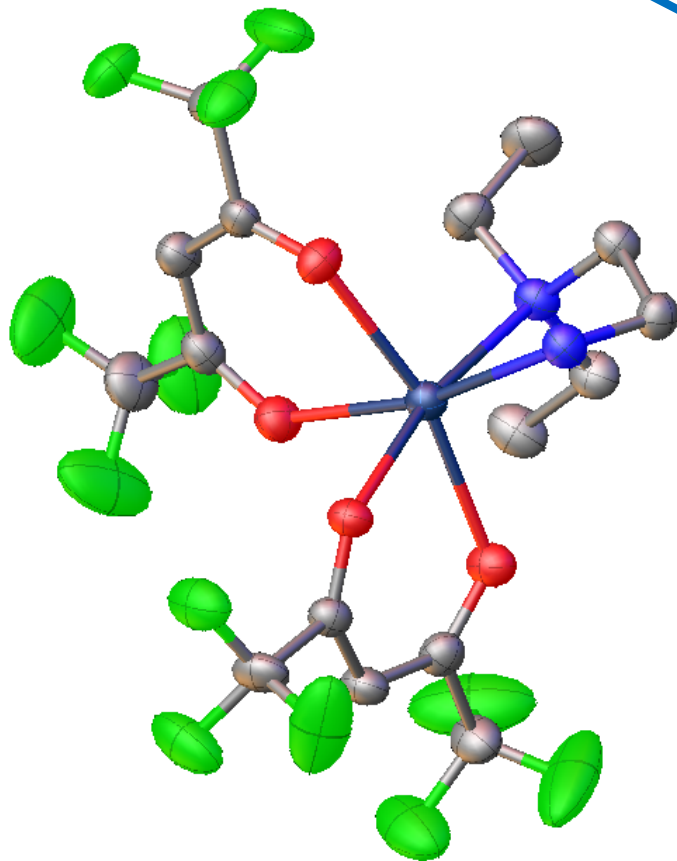
Extension Modules

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.

Go to:  
Work  
Refine dropdown  
Refine with Shelxl



Olex2

File Edit View Structure Mode Tools Model Select Help

### Example5

C:\Users\Lou\Desktop\CCCW\CCCW19\May\_27\SplittingCF3\Example5.ins *Pbca*

**C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub>F<sub>12</sub>Cd**

a = 15.084(2)	α = 90°	Z = 8	<b>6.77</b>
b = 11.1365(18)	β = 90°	Z' = 1	
c = 27.709(5)	γ = 90°	V = 4654.8(13)	

μ (Mo)	0.80	I/σ	19.9	Rint	12.23%	complete	100%
Shift	0.001	Max Peak	1.7	Min Peak	-0.9	Goof	1.149

Home work View Tools Info

Start

Welcome to Olex2! [CHANGELOG](#)

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.

Notice two things here

1. Yes, we missed a bunch of hydrogens

2. There are three residual peaks directly between the F-atoms of this -CF<sub>3</sub> group

The image shows a screenshot of the Olex2 software interface. On the left, a 3D ball-and-stick model of a molecule is displayed. The molecule features a central blue atom (likely Cd) coordinated to several red and grey atoms. A blue circle highlights a region of the structure, and a red circle highlights a specific -CF<sub>3</sub> group. On the right, the software's main panel displays the following information:

**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCW\CCCW19\May\_27\SplittingCF3\Example5.res

**C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>O<sub>4</sub>F<sub>12</sub>Cd**

a = 15.084(2)	α = 90°	Z = 8	R <sub>1</sub>	<b>6.77 %</b>
b = 11.1365(18)	β = 90°	Z' = 1	wR <sub>2</sub>	<b>16.92 %</b>
c = 27.709(5)	γ = 90°	V = 4654.8(13)		

d min (Mo) 0.80 I/σ 19.9 Rint 12.23% complete 100%  
Shift 0.001 Max Peak 1.7 Min Peak -0.9 Goof 1.135

Home Work View Tools Info  
Solve Refine Draw Report

Program: ShelXL, Least Squares, Cycles: 10, Peaks: 20  
hkl file: Example5.hkl, hkl: Wed Sep 24 06:25:14 2014  
Weight:  .061(.061) | 31.2(31.3) EXT1     
 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 N O F Cd ... Add H  
toC toH Hx Hx Hx Hx Z= 1 OK  
Select atom(s) and then mFit mSplit Split SAME Split  
MAP Show Map Map Settings  
Peak & Uiso Sliders  
Growing  
Finishing

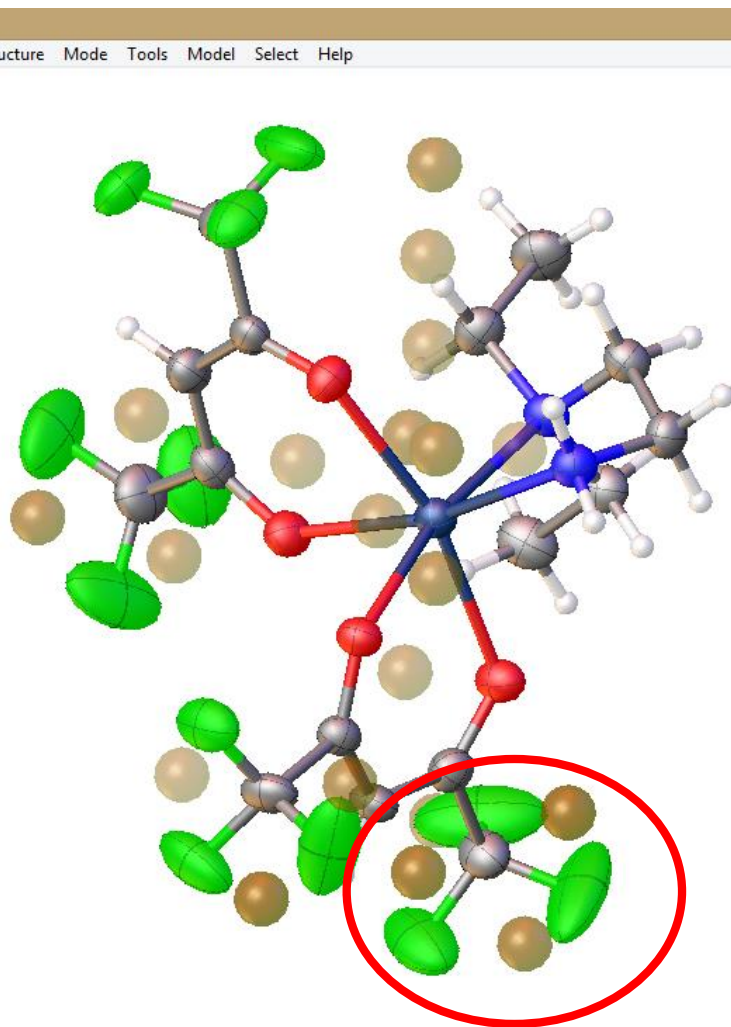




1. Looks good, but

2. Now there are residual peaks between F-atoms in multiple -CF<sub>3</sub> groups!

Let's deal with one of these groups; the highest residual peaks (Q1, Q2, Q4) are associated with the group comprised of C10, F10, F11, F12.



Olex2

C <sub>16</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> F <sub>12</sub> Cd					
a = 15.084(2)	α = 90°	Z = 8	R <sub>1</sub>	6.07 %	
b = 11.1365(18)	β = 90°	Z' = 1	wR <sub>2</sub>	15.27 %	
c = 27.709(5)	γ = 90°	V = 4654.8(13)	d min (Mo)	0.80	I/σ
Shift	-0.001	Max Peak	1.7	Min Peak	-0.9
Rint	12.23%	complete	100%	Goof	1.020

Home Work View Tools Info

Solve Refine Draw Report

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

hkl file: Example5.hkl, hkl: Wed Sep 24 06:25:14 2014

Weight:  0.61(0.38) | 31.3(24.6), EXTI  ACTA 52

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 N O F Cd ... Add H

Z = 1

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

MAP: Show Map Map Settings

Peak & Uiso Sliders

Peaks: Select Delete

Uiso Select Atoms: Delete



I'm going to tidy my display to work on this (mouse scroll down, or "Peak & Uiso Sliders" workbar, to show only the top four peaks and reorient my molecule to carefully inspect this group.)

Also, "Ctrl A", then select C8, C10, F10, F11, F12, then "View" and "Quick Drawing Styles" to convert everything else to a line

(You don't have to do this, but it makes my slide look better!)

The screenshot shows the Olex2 software interface. On the left is a 3D molecular model of a complex organic molecule with a central metal center (Cd) coordinated by several ligands. The model is rendered with a wireframe style for the main structure and green thermal ellipsoids for the non-hydrogen atoms. On the right is the software's control panel, titled "Example5". The panel displays the chemical formula  $C_{16}H_{18}N_2O_4F_{12}Cd$  and the space group  $Pbca$ . It also shows unit cell parameters (a, b, c) and angles ( $\alpha$ ,  $\beta$ ,  $\gamma$ ), as well as Z, Z', and V values. The R1 and wR2 values are 6.07% and 15.27% respectively. The panel includes a "Quick Drawing Styles" section with various icons for rendering styles, and a "Symmetry Generation" section with buttons for "Geometry", "Rotate", and "Stereo View".

Olex2

File Edit View Structure Mode Tools Model Select Help

**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCC\CCCCW19\May\_27\SplittingCF3\Example5.res

$C_{16}H_{18}N_2O_4F_{12}Cd$

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	R <sub>1</sub>	<b>6.07 %</b>
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	wR <sub>2</sub>	<b>15.27 %</b>
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)		

d min (Mo)	0.80	I/ $\sigma$	19.9	Rint	12.23%	complete	100%
Shift	-0.001	Max Peak	1.7	Min Peak	-0.9	Goof	1.020

Home Work View Tools Info

Quick Drawing Styles

Atom r: Set 100 Style: matt.glds

Bond r: Set 80 Colour: elements

Graphical objects:

Align View Plane Lock all Zoom Rotation Translation

Add Fog Clear Fog

Front 0.9

Back 3.6

Symmetry Generation

Geometry

Rotate

Stereo View

Next, select C10, F10, F11, F12 in this order (or at least, C10 first, and then the three F atoms.) Then type:

mode fit -s=1

Note that the C8-C10 bond is now selected as well (even though we didn't select C8.)

The screenshot displays the Olex2 software interface. On the left, a 3D ball-and-stick model of a molecule is shown, with a blue arrow pointing to a specific bond. The right side of the interface features a data panel for 'Example5' with the following information:

**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCC\CCCCW19\May\_27\SplittingCF3\Example5.res  
C16H18N2O4F12Cd

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	$R_1$	<b>6.07 %</b>
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	$wR_2$	<b>15.27 %</b>
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)	d min (Mo)	0.80
			l/ $\sigma$	19.9
			Rint	12.23%
			complete	100%
			Shift	-0.001
			Max Peak	1.7
			Min Peak	-0.9
			Goof	1.020

Below the data panel are tabs for Home, Work, View, Tools, and Info. The 'View' tab is active, showing 'Quick Drawing Styles' with options for Atom r (100), Bond r (80), and Graphical objects. At the bottom, a status bar indicates: 'You are in **mode FIT** -S=1. Press the **ESC** key to exit.'



Go back to your Work menu and Sort your atoms.

Then type "edit ins" at the prompt

Notes: Global RIGU (I'm deleting it), second free variable of 0.75, and two parts at bottom of the file with occupancies tied to the second free variable

Close your .ins

```
File Edit Format View Help
TITL disorder1 in Pbca
REM Solution 1 R1 0.144 Rweak 0.004, Alpha = 0.0755 in Pbca
CELL 0.71073 15.0844 11.1365 27.7091 90 90 90
ZERR 8 0.0024 0.0018 0.0045 0 0 0
LATT 1
SYMM 0.5-X, -Y, 0.5+Z
SYMM -X, 0.5+Y, 0.5-Z
SYMM 0.5+X, 0.5-Y, -Z
SFAC C H N O F Cd
UNIT 128 144 16 32 96 8
RIGU
L.S. 10
PLAN 20
TEMP -120
BOND
LIST 6
MORE -1
CONF
fmap 2
acta 52
WGHT 0.0613 31.275499
FVAR 0.183 0.75
REM <olex2.extras>
REM <HklSrc "%.\\Example5.hkl">
REM </olex2.extras>
H15A 2 0.63802 0.68612 0.39892 11.00000 -1.20000
H15B 2 0.71711 0.63780 0.36572 11.00000 -1.20000
AFIX 0
C16 1 0.69784 0.53366 0.42679 11.00000 0.02955 0.03732 0.03
0.00103 -0.00146 0.00142
AFIX 23
H16A 2 0.72548 0.46001 0.41360 11.00000 -1.20000
H16B 2 0.74321 0.57731 0.44572 11.00000 -1.20000
AFIX 0
PART 1
F10 5 0.54794 0.10701 0.30649 21.00000 0.13570
F11 5 0.62770 0.02720 0.35801 21.00000 0.13234
F12 5 0.49989 -0.02748 0.34778 21.00000 0.09000
PART 0
PART 2
F10a 5 0.48980 0.04404 0.31955 -21.00000 0.13570
F11a 5 0.62024 0.09781 0.32720 -21.00000 0.13234
F12a 5 0.56930 -0.03864 0.36784 -21.00000 0.09000
HKLf 4
END
```



Note the formatting of the restraint; each sequential pair is restrained to have the same distance

We can select individual atoms in a pairwise manner and apply the same restraint. Let's do this with each of the 1,4 pairs in our disorder group.

If we want to do this with the 1,3 pairs we encounter a problem with using a GUI!

We can add these by editing our .ins (note that shelx doesn't "do" angle restraints, so we get around this with SADI)

```
File Edit Format View Help
TITL disorder1 in Pbca
REM Solution 1 R1 0.144 Rweak 0.004, Alpha = 0.0755 in Pbca
CELL 0.71073 15.0844 11.1365 27.7091 90 90 90
ZERR 8 0.0024 0.0018 0.0045 0 0 0
LATT 1
SYMM 0.5-X, -Y,0.5+Z
SYMM -X,0.5+Y,0.5-Z
SYMM 0.5+X,0.5-Y, -Z
SFAC C H N O F Cd
UNIT 128 144 16 32 96 8
SADI F12a C10 F11 C10 F11a C10 F10 C10 F10a C10 F12 C10
SADI F12 F11a F10a F11 F10 F12a
SADI F10 F11 F11 F12 F12 F10 F10a F11a F11a F12a F12a F10a|
L.S. 10
PLAN 20
TEMP -120
BOND
```

CF distance restraints

1,4 distance restraints

1,3 distance restraints

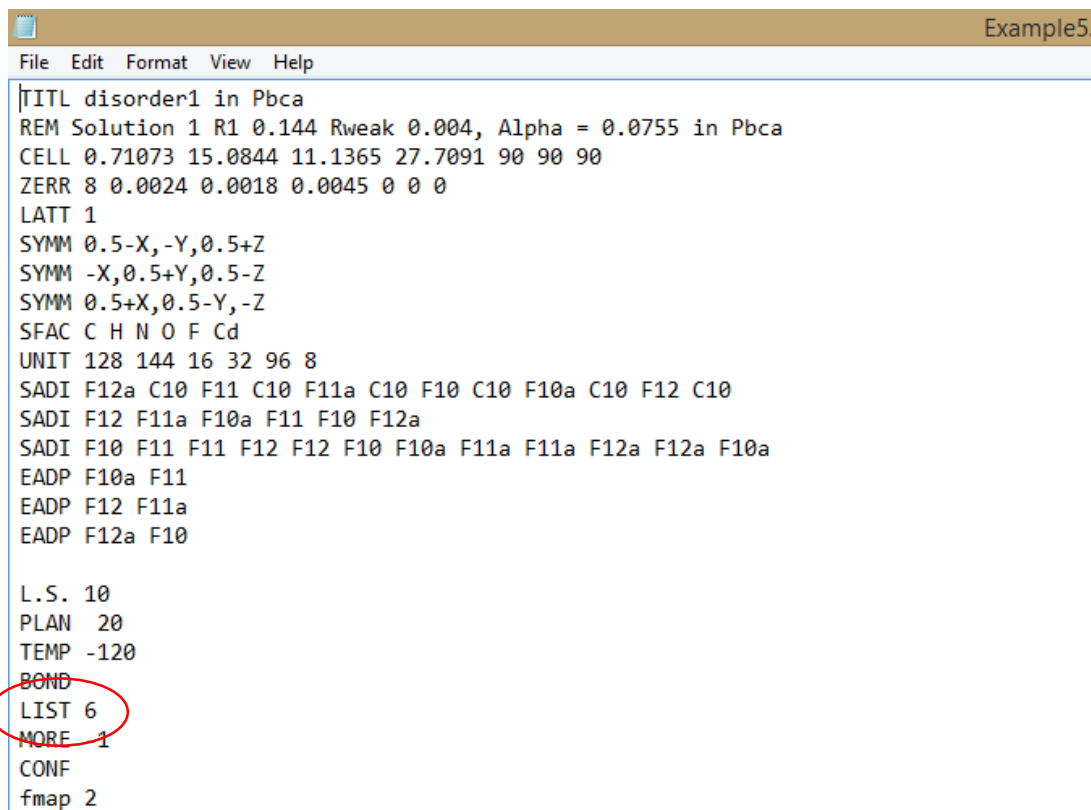
I'm still not ready to hit "Refine"

Let's think about how those 1,4 displacements should "look".

Select the 1,4 F-atoms, one pair at a time, and introduce "Shelx Compatible Constraints" EADP after each selection.

(WHOA! Just noticed that there is a LIST 6 instructions in this ins! This instructs the kind of structure factor output that is generated. LIST 6 is not compatible with many things; let's edit this to LIST 4)

Displacements, distances and effectively angles, are now managed. Let's hit "Refine".



```
Example5.  
File Edit Format View Help  
TITL disorder1 in Pbc  
REM Solution 1 R1 0.144 Rweak 0.004, Alpha = 0.0755 in Pbc  
CELL 0.71073 15.0844 11.1365 27.7091 90 90 90  
ZERR 8 0.0024 0.0018 0.0045 0 0 0  
LATT 1  
SYMM 0.5-X,-Y,0.5+Z  
SYMM -X,0.5+Y,0.5-Z  
SYMM 0.5+X,0.5-Y,-Z  
SFAC C H N O F Cd  
UNIT 128 144 16 32 96 8  
SADI F12a C10 F11 C10 F11a C10 F10 C10 F10a C10 F12 C10  
SADI F12 F11a F10a F11 F10 F12a  
SADI F10 F11 F11 F12 F12 F10 F10a F11a F11a F12a F12a F10a  
EADP F10a F11  
EADP F12 F11a  
EADP F12a F10  
  
L.S. 10  
PLAN 20  
TEMP -120  
BOND  
LIST 6  
MORE 1  
CONF  
fmap 2
```



Sigh...those 1,4 restraints...and those new residual density peaks...

If only Mike Katz was here...

In the meantime, change the isotropic atoms to anisotropic, and refine again.

The screenshot displays the Olex2 software interface. On the left, a 3D ball-and-stick model of a complex molecule is shown, with a central cluster of green and brown spheres. The right side of the window contains a data panel for 'Example5' with the following information:

**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCC\CCCC19\May\_27\SplittingCF3\Example5.res  
C16H18N2O4F12Cd

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	$R_1$	<b>6.51 %</b>
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	$wR_2$	<b>15.07 %</b>
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)		

d min (Mo)	0.80	I/ $\sigma$	19.9	Rint	12.23%	complete	100%
Shift	-3.402	Max Peak	1.9	Min Peak	-0.8	Goof	1.220

Navigation buttons: Home, Work, **View**, Tools, Info

**Quick Drawing Styles**

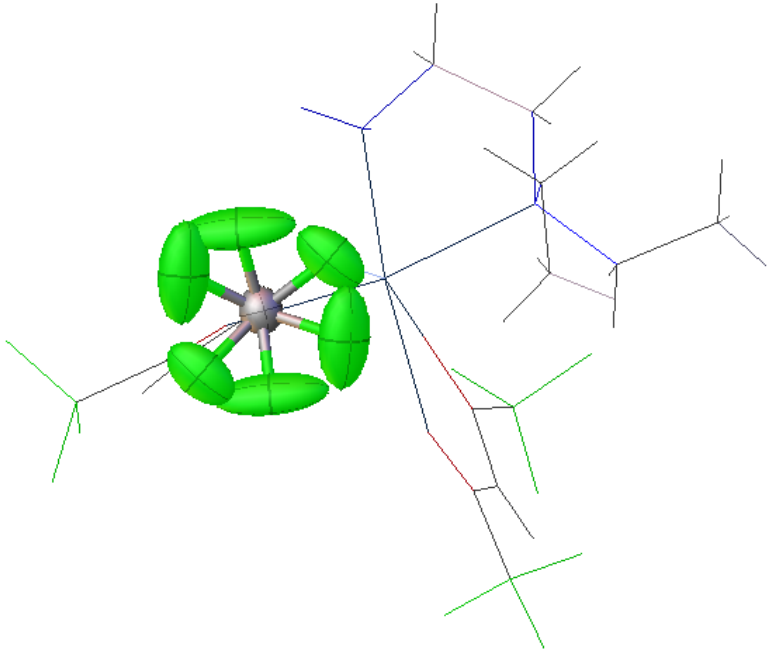
- Atom r: Set [100] Style: matt.glds
- Bond r: Set [80] Colour: elements
- Graphical objects: [Icons]
- Align: View, Plane, Lock all, Zoom, Rotation, Translation
- Add Fog, Clear Fog
- Front: [Slider] 0.9
- Back: [Slider] 3.6

Buttons: Symmetry Generation, Geometry, Rotate

Well, anisotropic refinement dealt with those residual peaks, but is this a good model for what is taking place here?

Olex2

File Edit View Structure Mode Tools Model Select Help



a = 15.084(2)	$\alpha = 90^\circ$	$Z = 8$	$R_1$	5.49 %
b = 11.1365(18)	$\beta = 90^\circ$	$Z' = 1$	$wR_2$	12.91 %
c = 27.709(5)	$\gamma = 90^\circ$	$V = 4654.8(13)$		

d min (Mo)	0.80	$I/\sigma$	19.9	Rint	12.23%	complete	100%
Shift	0.446	Max Peak	1.3	Min Peak	-0.8	Goof	0.966

Home Work View Tools Info

Solve Refine Draw Report

Program SheXL Least Squares Cycles 10 Peaks 20

hkl file Example5.hkl hkl: Wed Sep 24 06:25:14 2014

Weight  .040(013) | 31.6(18.7) EXTI  ACTA 52

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 N O F Cd ...  Add H

C  H  N  O  F  Cd ...  Add H

C  H  N  O  F  Cd ...  Add H

Z = 1

0 | 1 0 | 2 All Sel  Unique  Labels

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

MAP Show Map Map Settings

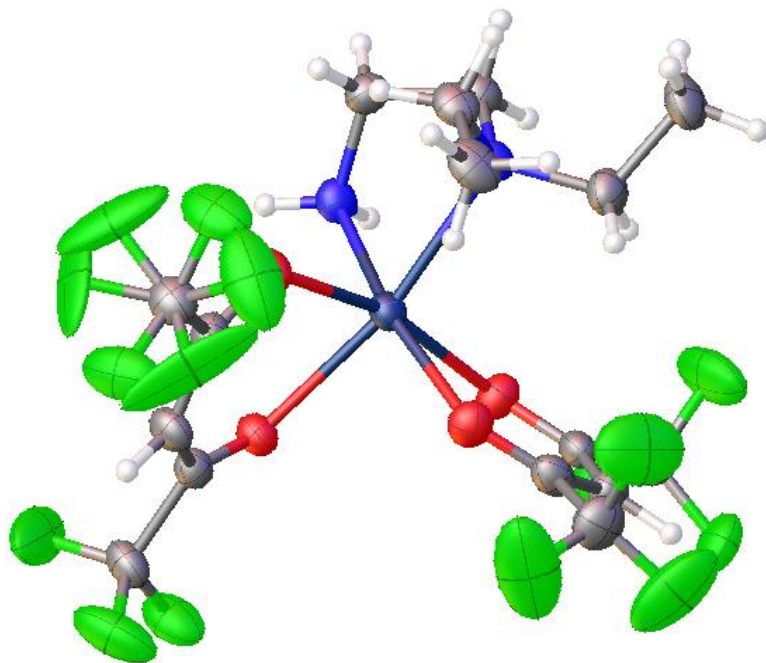
Peak & Uiso Sliders

Peaks Select Delete

25

We are done with this example, but here are a few things to consider:

1. I went in and manually changed the EADP constraints to RIGU restraints but this does not really improve the model.
2. You could go in and further model this with a third (and fourth, and fifth) orientation, tying the occupancies to a SUMP instruction (there is no convenient way to do this in OLEX2; you have to directly edit your ins.)
3. What about the other  $-\text{CF}_3$  groups?
4. Everything would have been tidier if I had rotated my original second component by  $180^\circ$  instead of  $60^\circ$ .



**Example5** *Pbca*  
C:\Users\Lou\Desktop\CCCW\CCCW19\May\_27\SplittingCF3\Example5.res

C16H18N2O4F12Cd

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	$R_1$	5.40 %
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	$wR_2$	11.71 %
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)		

d min (Mo)	0.80	$I/\sigma$	19.9	Rint	12.23%	complete	100%
Shift	0.150	Max Peak	1.3	Min Peak	-0.8	Goof	1.164

Home Work View Tools Info

Quick Drawing Styles

Atom r: Set 100 Style: matt.glds

Bond r: Set 80 Colour: elements

Graphical objects:

Align View Plane Lock all Zoom Rotation Translation

Add Fog Clear Fog

Front 0.9

Back 3.6

Symmetry Generation

Geometry

Rotate

If I was going to split this into three components:

1. Go back to isotropic refinement. Run a round of least squares. Bring back those third component residual electron density peaks.
2. I introduced the next component by directly converting the three peaks to F atoms, and manually named them to F10B, F11B, F12B.
3. "Sort" and then edit ins.
4. Delete the 1,4 SADI restraints. Introduce a new set of C-F and 1,3 F-F SADI restraints for the B component (carry on to the next line using =). Combine all F atoms into a single RIGU line.
5. Find F10B, F11B, F12B, and move them to the bottom of the .cif, under a PART 3.
6. For PART 2, change all of the "-21.000" to "31.000"; for PART 3 change all of the "11.0000" to "41.000"
7. Go back to your FVAR line and change the second FVAR to 0.5, then add in a third with 0.3 and fourth with 0.2
8. Finally, add in the following line with your other instructions (this requires that the sum of the free variables add up to one.)

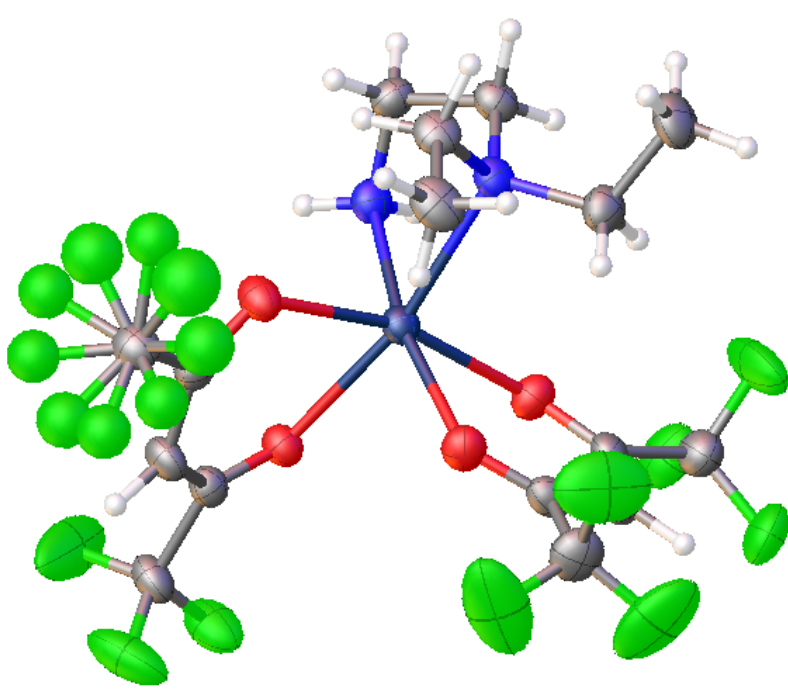
```
SUMP 1.0 0.001 1.0 2 1.0 3 1.0 4
```

9. Hit save. Close the .ins. Hit Refine. Good luck to you. History is still there in case of disaster!

This is before I hit refine. The next slide is what my .ins looks like, just before I hit refine.

Olex2

File Edit View Structure Mode Tools Model Select Help



Crystallographic data:

a = 15.084(2)	$\alpha = 90^\circ$	Z = 8	$R_1$	6.31 %
b = 11.1365(18)	$\beta = 90^\circ$	Z' = 1	$wR_2$	14.50 %
c = 27.709(5)	$\gamma = 90^\circ$	V = 4654.8(13)		

Refinement statistics:

d min (Mo)	0.80	$I/\sigma$	19.9	Rint	12.23%	complete	100%
Shift	-0.070	Max Peak	1.9	Min Peak	-0.8	Goof	1.127

Home Work View Tools Info

Solve Refine Draw Report

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

hkl file: Example5.hkl, hkl: Wed Sep 24 06:25:14 2014

Weight:  0.36(0.037) | 29.6(29.8), EXTI   RATA 32

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

```

SYMM -X,0.5+Y,0.5-Z
SYMM 0.5+X,0.5-Y,-Z
SFAC C H N O F Cd
UNIT 128 144 16 32 96 8
SADI F12A C10 F11 C10 F11A C10 F10 C10 F10A C10 F12 C10 =
F10B C10 F11B C10 F12B C10
SADI F10 F11 F11 F12 F12 F10 F10A F11A F11A F12A F12A F10A =
F10B F11B F11B F12B F12B F10B
RIGU F10 F10A F10B F11 F11A F11B F12 F12A F12B
SUMP 1.0 0.001 1.0 2 1.0 3 1.0 4

```

```

L.S. 10
PLAN 20
TEMP -120
BOND
LIST 4
MORE -1
CONF
fmap 2
acta 52
WGHT 0.0359 29.634201
FVAR 0.18368 0.5 0.3 0.2
REM <olex2.extras>
REM <HklSrc "%.\\Example5.hkl">
REM </olex2.extras>

```

GROUP	GROUP	GROUP	GROUP	GROUP	GROUP
AFIX 23					
H16A 2	0.72559	0.46002	0.41364	11.00000	-1.20000
H16B 2	0.74317	0.57743	0.44573	11.00000	-1.20000
AFIX 0					
PART 1					
F10 5	0.52945	0.10426	0.30518	21.00000	0.06323
F11 5	0.63609	0.05060	0.34907	21.00000	0.06735
F12 5	0.50936	-0.03925	0.35458	21.00000	0.05607
PART 2					
F10A 5	0.48861	-0.01254	0.33930	31.00000	0.05231
F11A 5	0.58560	0.11146	0.30990	31.00000	0.08463
F12A 5	0.61935	0.00110	0.36795	31.00000	0.05323
PART 3					
F10B 5	0.49590	0.04800	0.32080	41.00000	0.05000
F11B 5	0.63050	0.10340	0.32540	41.00000	0.05000
F12B 5	0.56990	-0.03840	0.36780	41.00000	0.05000
PART 0					
HKLF 4					
END					

Post refine. Still some peaks.  
Hover over the atoms to see their occupancies.  
Let's go anisotropic.

The screenshot displays the Olex2 software interface. On the left, a 3D ball-and-stick model of a molecular structure is shown, with atoms colored by element (carbon in grey, oxygen in red, nitrogen in blue, hydrogen in white, and fluorine in green). The structure is complex, featuring a central core with various substituents. On the right, the software's control panel is visible, showing the 'Work' tab selected. The 'Solve' and 'Refine' buttons are highlighted. The 'Refine' section shows the following settings:

- Program: ShelXL
- Least Squares
- Cycles: 10
- Peaks: 20
- hkl file: Example5.hkl
- hkl: Wed Sep 24 06:25:14 2014
- Weight:  .037(.014) | 29.8(22.0) EXT1  ACTA 52
- Use Solvent Mask This is the Olex2 implementation of BYPASS (a.k.a. SQUEEZE)
- Refinement Settings Extra

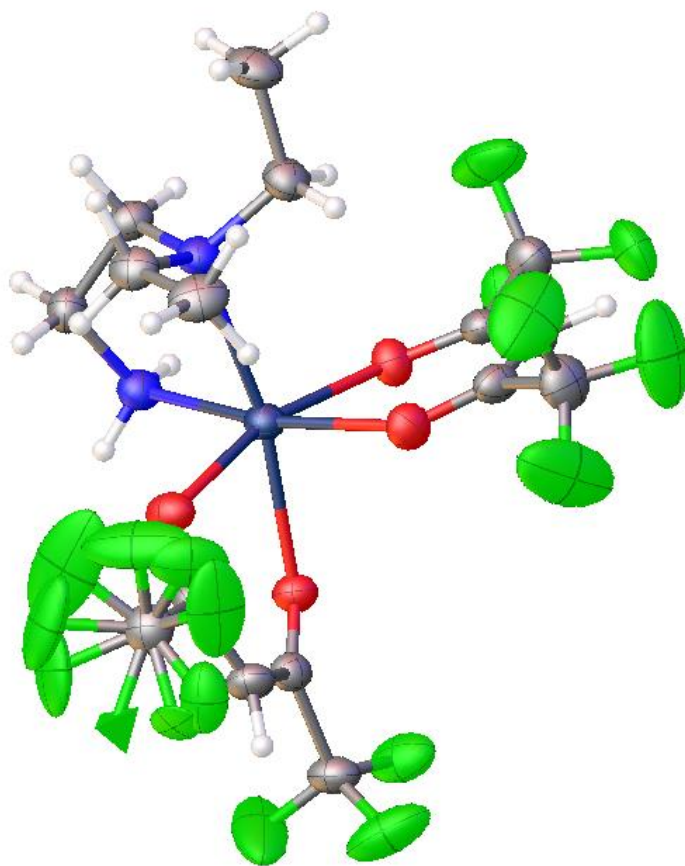
The 'Toolbox Work' section includes:

- Labels: Labels OFF/ON
- Element selection: C H N O F Cd ...
- Buttons: Add H, OK
- Z= 1
- Buttons: 0|1, 0|2, 0|3, All, Sel  Unique  Labels
- Select atom(s) and then: mFit, mSplit, Split SAME, Split
- MAP: Show Map, Map Settings
- Peak & Uiso Sliders: Select, Delete, 25, < 0.025
- Growing
- Finishing



Ugh. Let's go home.

(No, seriously, I could probably "fix" this with some ISOR restraints, or EADP constraints, or changing the default RIGU esd to something much smaller. But that doesn't make it "right".)



0 min (m/s)	0.80	1/a	19.9	Rint	12.23%	complete	100%
Shift	-0.092	Max Peak	1.3	Min Peak	-0.8	Goof	1.095

Home Work View **Tools** Info

HART

AutoChem 3.1

Images

Maps

Chemical Tools

Olex2 Constraints Restraints

Shelx Compatible Constraints

**Shelx Compatible Restraints**

SADI  go

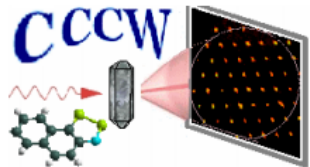
**One Atom Selected** All 'outgoing' bonds will be restrained to be the same, all distances between these bound atoms will also be restrained - with double the e.s.d. This feature allows to 'regularise' entities like spherical counterions.

**Two or more Bonds Selected** The selected bonds will be restrained to be the same.

**Three Atoms in a row** The bonds between the two atoms bound to the central atoms will be restrained to be the same.

**Pairwise atom selection** If an even number of atoms is selected, the distances between pairs of atoms will be restrained to be the same, depending on the order of selection.

Hydrogen Atoms



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

## Solvent or Anion Disorder: Fixed Positions

Check out the OLEX2 youtube channel for a similar example:

[https://www.youtube.com/watch?v=hCl4VdQ8iBg&list=PLJgQksgBlpeAMDW5PVOI0r9EqFZ8p-Y4 &index=7&t=0s](https://www.youtube.com/watch?v=hCl4VdQ8iBg&list=PLJgQksgBlpeAMDW5PVOI0r9EqFZ8p-Y4&index=7&t=0s)

Navigate to Home.

Under Start, select "Malbec"

The screenshot displays the Olex2 software interface. On the left, a 3D ball-and-stick model of a complex organic molecule is shown, featuring a central ring system with various substituents, including a red oxygen atom, a blue nitrogen atom, and a green chlorine atom. The main window title is "malbac" and the file path is "C:\Users\Indaw\AppData\Roaming\Olex2\925a794\samples\malbac\malbac.res". The chemical formula is  $C_{29}H_{25}ClN_2OPd$ . The unit cell parameters are listed as follows:

a = 10.551(2)	$\alpha = 99.77(2)^\circ$	Z = 2
b = 14.251(3)	$\beta = 105.10(2)^\circ$	Z' = 1
c = 9.551(2)	$\gamma = 104.77(2)^\circ$	V = 1297.1(5)

Below the unit cell parameters, a table of diffraction statistics is shown:

d min (Mo)	0.71	$I/\sigma$	32.1	R <sub>int</sub>	n/a	complete	100%
Shift	-0.222	Max Peak	1.4	Min Peak	-0.9	GOF	1.016

The interface includes a menu bar (File, Edit, View, Structure, Mode, Tools, Model, Select, Help) and a sidebar with tabs for Home, Work, View, Tools, and Info. The "Start" section contains a "Welcome to Olex2!" message with a "CHANGELOG" link and an "Open" button. Below this, there are buttons for "Sucrose", "THPP", "Co110", "ZP2", "Water", "Malbac", and "Timmy". The "Malbac" button is highlighted, indicating it is the selected option. The sidebar also lists "Tutorials", "Extension Modules", "Settings", and "News".

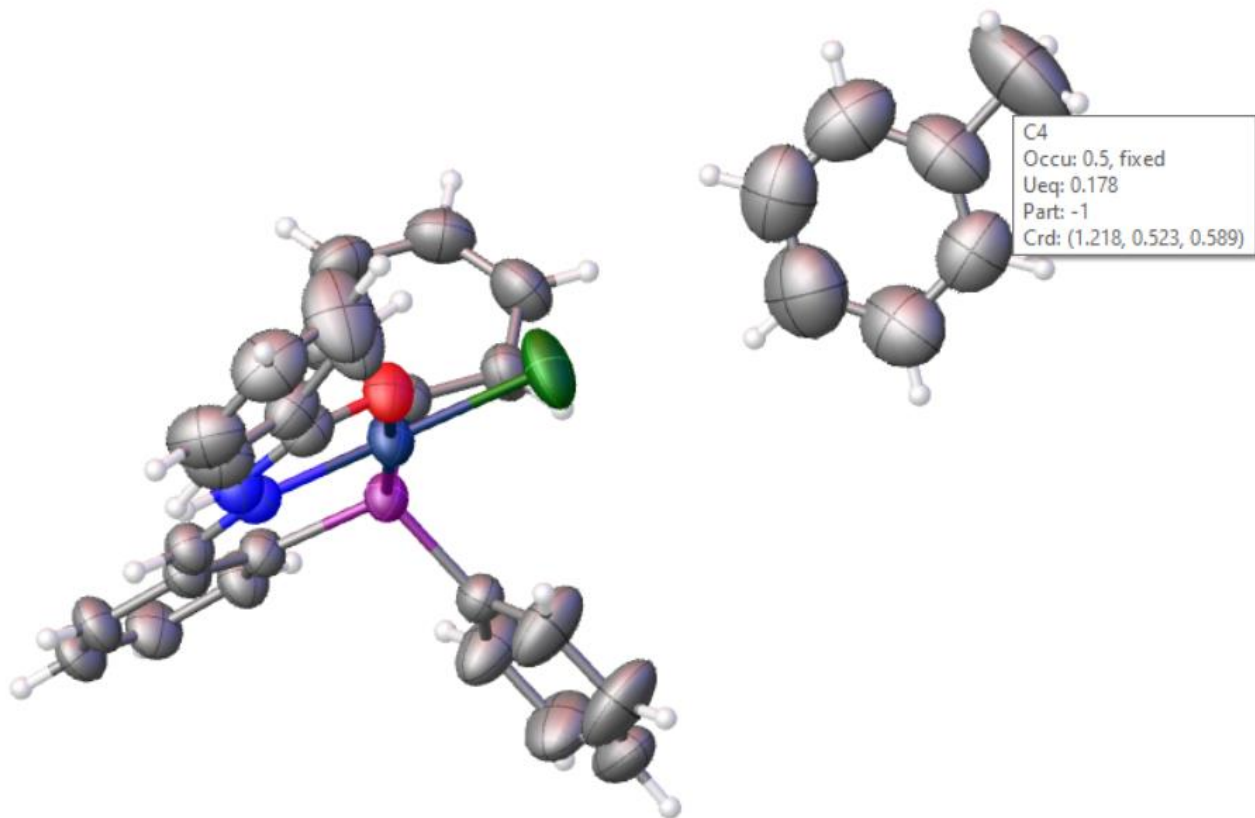
This is the finished structure.

Toluene looks disordered.

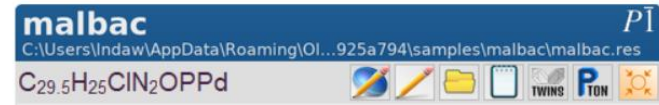
Hover over any atom; occupancy is 0.5, and it is PART -1

Why?

---

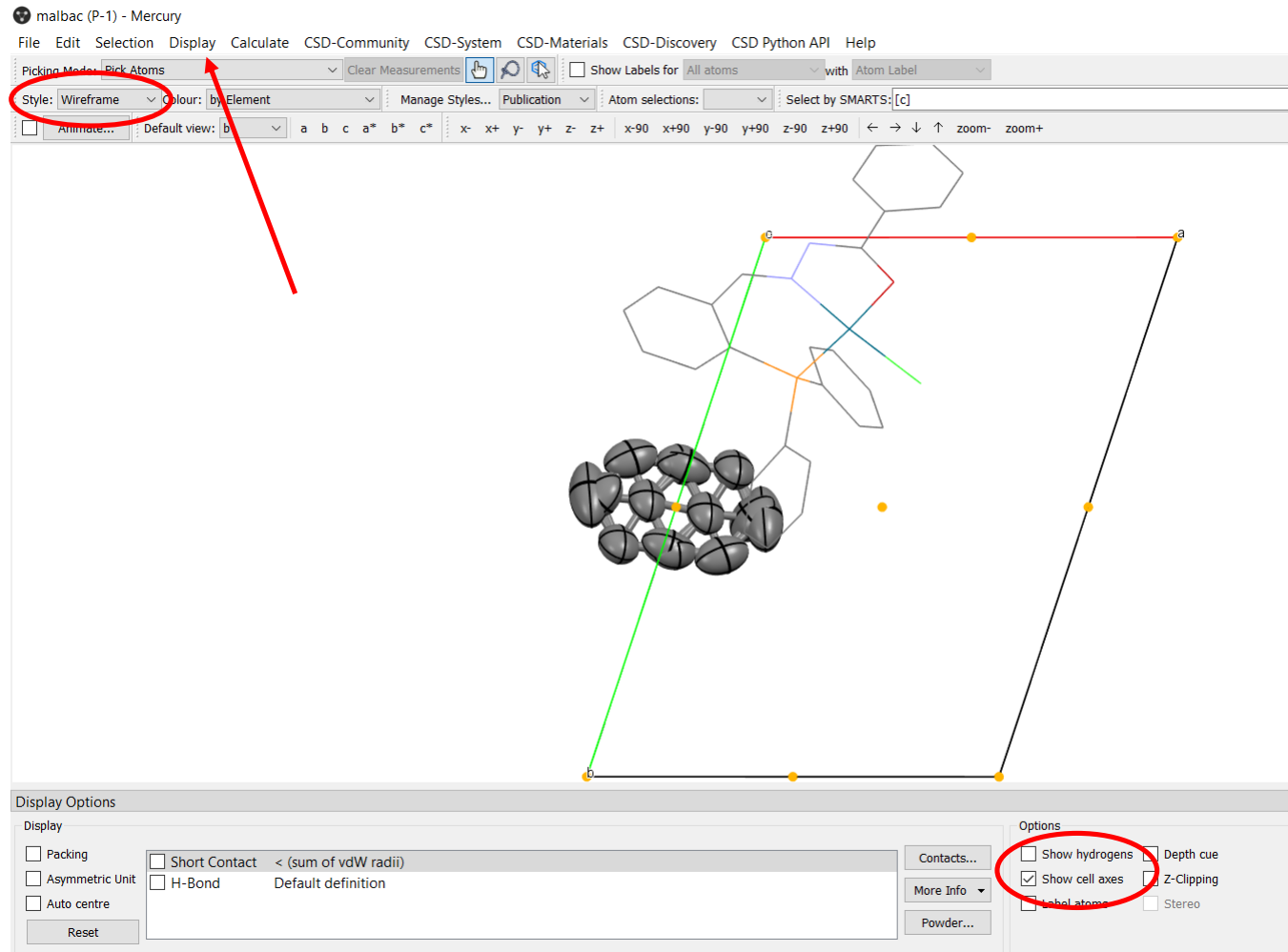


Open the .cif in Mercury; I got my file path by looking here:



Right click on the main molecule (not toluene): Selection → Select Molecule

I then changed my style to “Wireframe”, deselected “Show hydrogen”, selected “Show cell axes”, and the went to: Display → Symmetry Elements... → OK

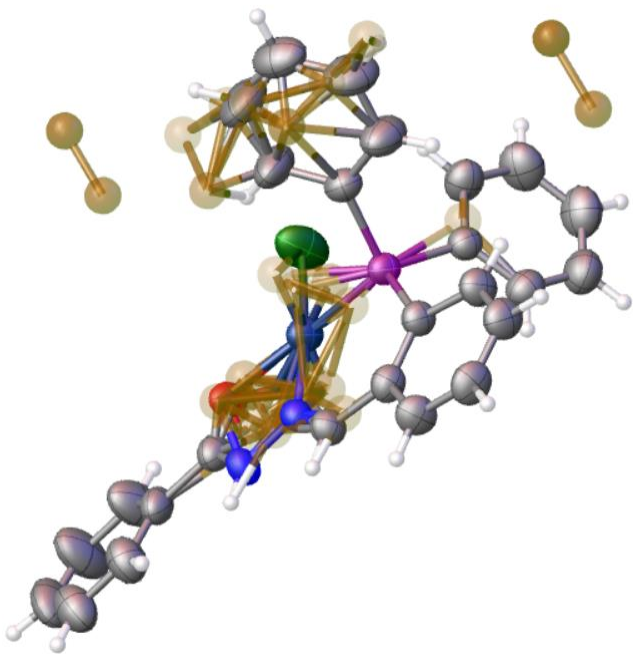




## Back in OLEX2

1. Delete toluene, set your rounds of least squares to five, set your peaks to 25, and refine.
2. Assemble your fragments.
3. Display only your top four peaks, and type "grow".

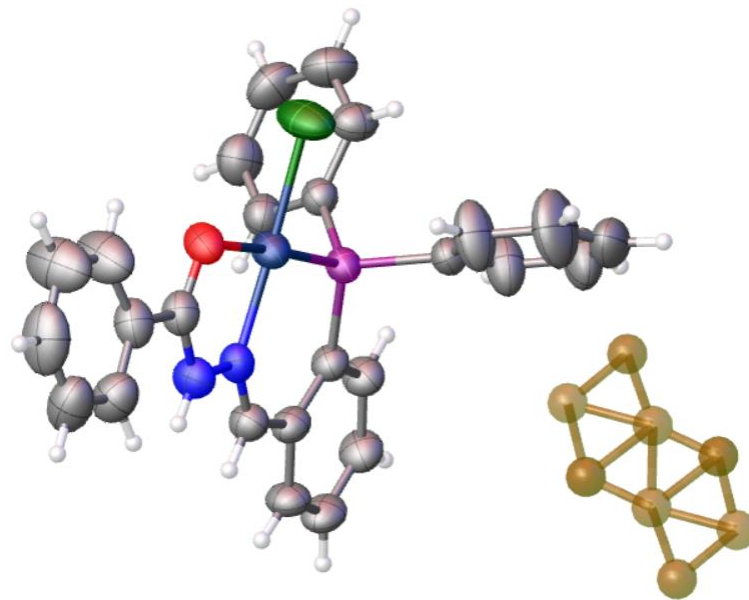
1.



2.



3.

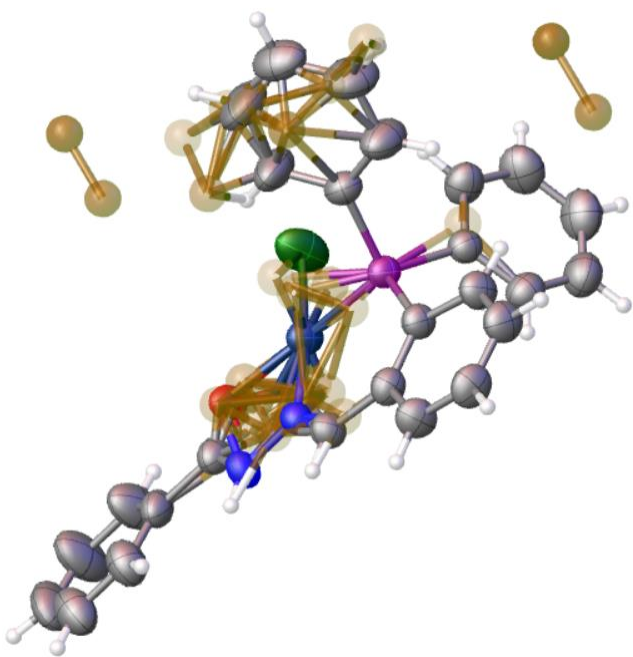




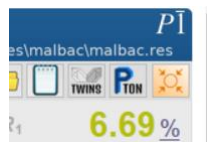
## Back in OLEX2

1. Delete toluene, set your rounds of least squares to five, set your peaks to 25, and refine.
2. Assemble your fragments.
3. Display only your top four peaks, and type "grow".
4. Imagination!

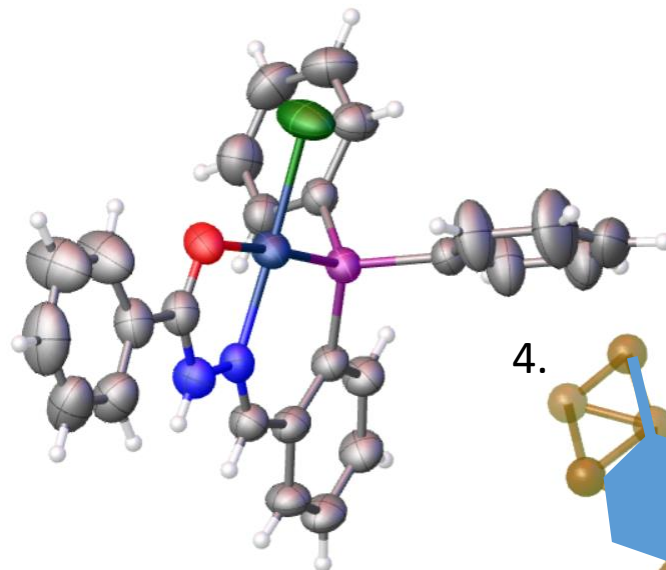
1.



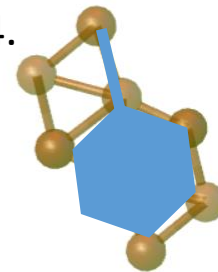
2.



3.



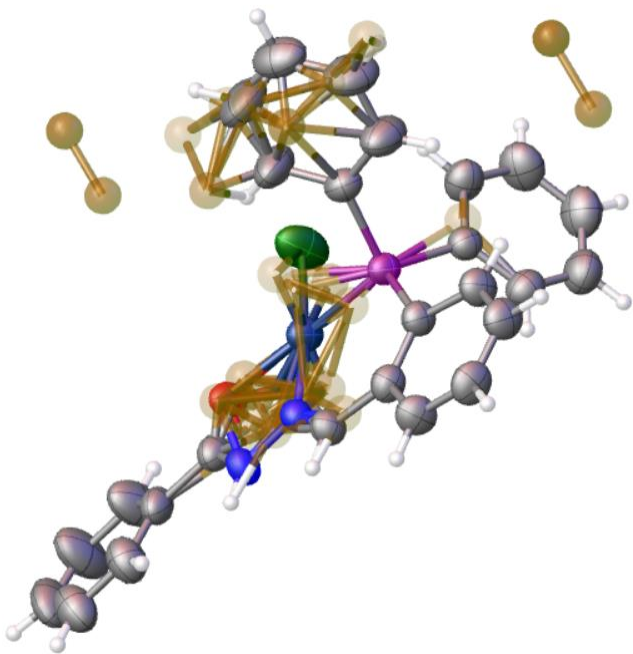
4.



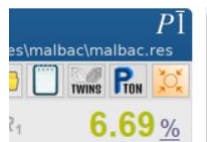
## Back in OLEX2

1. Delete toluene, set your rounds of least squares to five, set your peaks to 25, and refine.
2. Assemble your fragments.
3. Display only your top four peaks, and type “grow”.
4. Imagination! (No seriously now, how do I make it do that?)

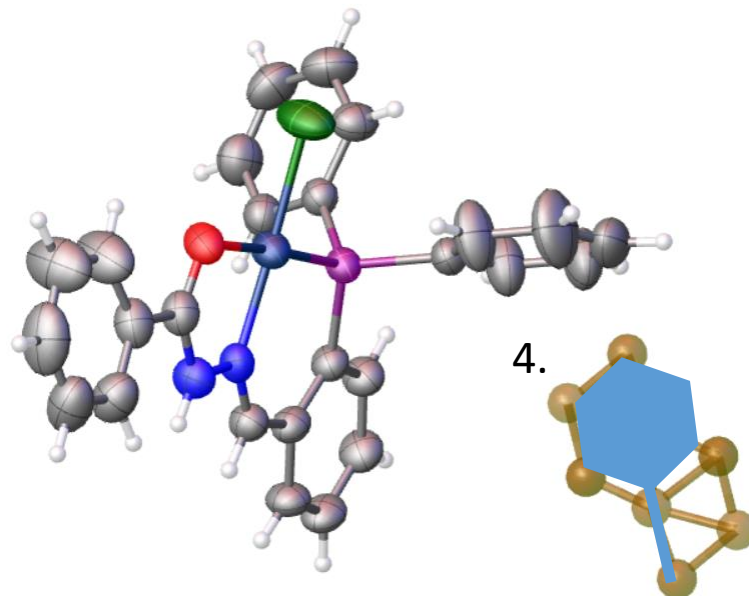
1.



2.



3.



## OLEX2 makes FRAG/FEND “simple”

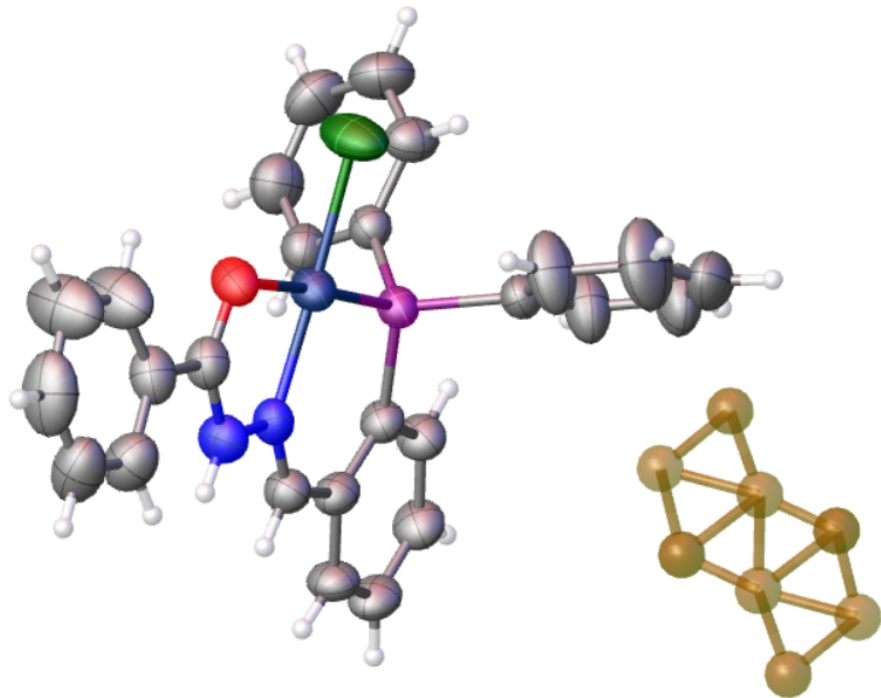
From the shelx manual:

FRAG code[17] a[1] b[1] c[1]  $\alpha$ [90]  $\beta$ [90]  $\gamma$ [90] Enables a fragment to be input using a cell and coordinates taken from the literature. Orthogonal coordinates may also be input in this way. Such a fragment may be fitted to the set of atoms following an AFIX instruction with m=code (code must be greater than 16); there must be the same number of atoms in this set as there are following FRAG, and they must be in the same order. Atoms with zero coordinates are not fitted, but new coordinates are generated for these atoms. The atom names, sfac numbers, sof and Uij of the FRAG fragment are ignored, only the coordinates are used. A FRAG fragment may be given anywhere between UNIT and HKLF or END or in an 'include' file, and must be terminated by a FEND instruction, but must precede any AFIX instruction which refers to it. This rigid fit is often a preliminary to a rigid group refinement (AFIX 6).

Tools → FragmentDB → Find toluene from the list

Next: PART = -1; Occupancy = 0.5 (Why?)

Tools Model Select Help



**malbac**  $P\bar{1}$   
C:\Users\Indaw\AppData\Roaming\Ol...925a794\samples\malbac\malbac.res

C29.5H25ClN2OPPd

a = 10.551(2)	$\alpha = 99.77(2)^\circ$	Z = 2	R <sub>1</sub>	6.69 %
b = 14.251(3)	$\beta = 105.10(2)^\circ$	Z' = 1	wR <sub>2</sub>	21.65 %
c = 9.551(2)	$\gamma = 104.77(2)^\circ$	V = 1297.1(5)		

d min (Mo)	0.71	I/ $\sigma$	32.1	Rint	n/a	complete	100%
Shift	0.224	Max Peak	3.3	Min Peak	-0.8	Goof	1.834

WARNING: Input data appear to be merged: CIF file will be incomplete

Home Work View **Tools** Info

HART

ReportPlus

FragmentDB

Toluene, C7H8

Fit Edit

PART: -1 Free Variable: 1 Occupancy: 0.5 = 10.5

Use a residue:  Residue Class: TOL Invert:  Calculate DFIX:

Replace Mode:  No Restraints:  Rigid Group:  Revert Fit

List of most disagreeable restraints: Short Full

Observed	Target	Error	Sigma	Restraint
--	--	--	--	--

Tools → FragmentDB → Find toluene from the list

Next: PART = -1; Occupancy = 0.5 (Why?)

Directly from Peter Mueller: <http://web.mit.edu/pmueller/www/ACA2007/WK01/Disorder.pdf>

## Disorder Involving Special Positions

Imagine a molecule sitting on or near a special position without fulfilling the geometry of that symmetry element (e.g. toluene on inversion center).

Two possible ways of describing: either use different space group without the symmetry element(s) in question or refine a disorder about the special position.

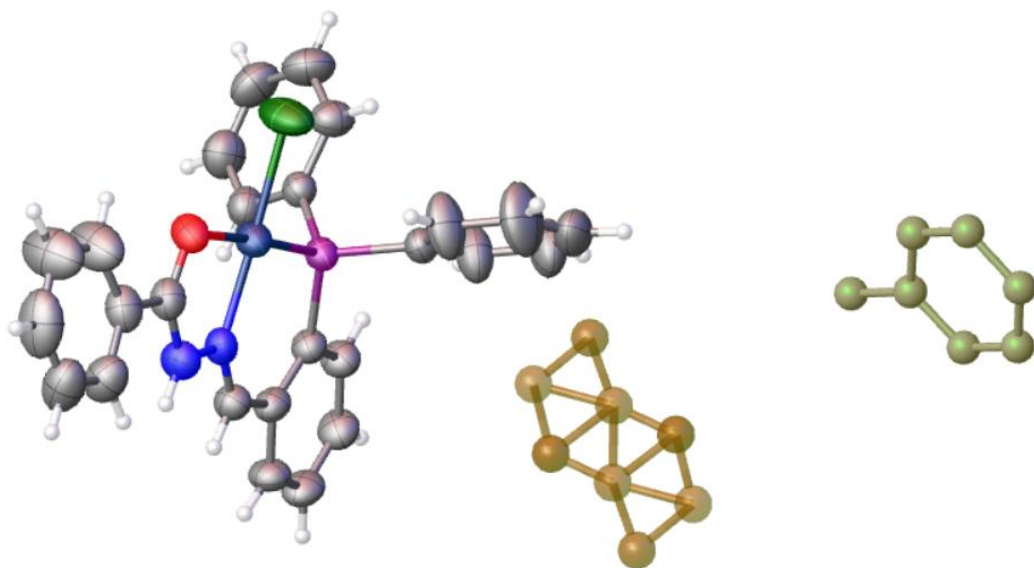
Refinement is easy: you need only one set of coordinates, as the second one can be generated from the first by means of the symmetry operator(s) corresponding to the special position in question. Therefore instead of **PART 1** and **PART 2** you only need one component, which has to be placed in **PART -1**.

The ratio does not need to be refined as it corresponds to the multiplicity of the symmetry operator (0.5 for an inversion center, mirror and twofold axis; 0.3333 for a threefold, 0.25 for a fourfold and 0.1667 for a sixfold). Combinations of symmetry operators are possible, of course.

Once you have selected toluene from the list, hit “Fit”

A “shadow” toluene will appear.

Click on one atom in the shadow, and then click on one of your electron density peaks that you want to line up with the shadow.



b = 14.251(3)	$\beta = 105.10(2)^\circ$	Z' = 1			
c = 9.551(2)	$\gamma = 104.77(2)^\circ$	V = 1297.1(5)	wR <sub>2</sub>	21.65 %	
d min (Mo)	0.71	I/σ	32.1	R <sub>int</sub>	n/a
Shift	0.224	Max Peak	3.3	Min Peak	-0.8
				Goof	1.834

WARNING: Input data appear to be merged: CIF file will be incomplete

Home Work View **Tools** Info

HART

ReportPlus

FragmentDB

Toluene, C7H8

PART: -1 Free Variable: 1 Occupancy: 0.5 => 10.5

Use a residue:  Residue Class: TOL Invert:  Calculate DFIX:

Replace Mode:  No Restraints:  Rigid Group:

List of most disagreeable restraints:

Observed	Target	Error	Sigma	Restraint
--	--	--	--	--

AC4

Twinning

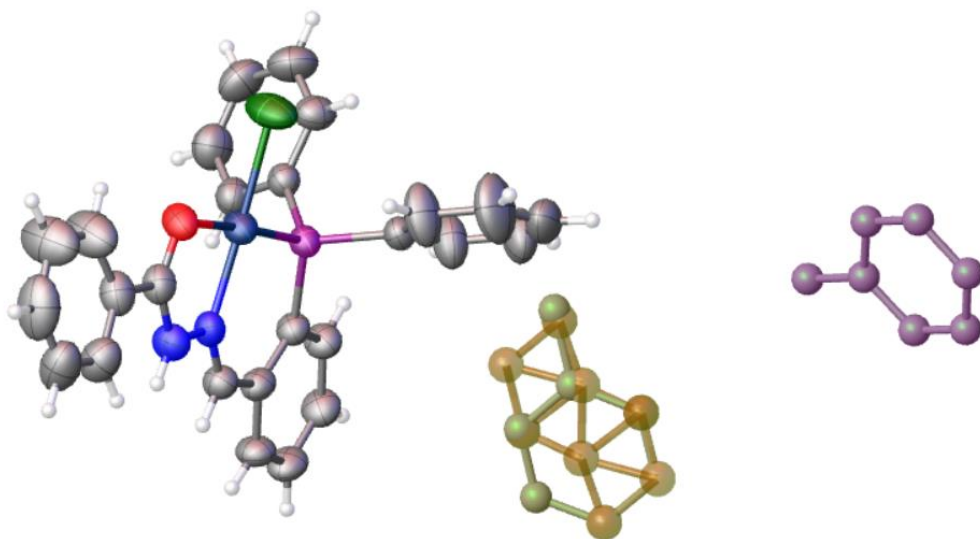
You are in **mode** MODE\_DISP  
Press the **ESC** key to exit.

Once you have selected toluene from the list, hit “Fit”

A “shadow” toluene will appear.

Click on one atom in the shadow, and then click on one of your electron density peaks that you want to line up with the shadow.

Keep selecting one shadow peak (green) and one electron density peak (orange) until you get a reasonable fit. This took me three clicks.



c = 9.551(2)	v = 104.77(2)°	V = 1297.1(5)	WR <sub>2</sub>	21.05	%		
d min (Mo)	0.71	I/σ	32.1	Rint	n/a	complete	100%
Shift	0.224	Max Peak	3.3	Min Peak	-0.8	Goof	1.834

WARNING: Input data appear to be merged: CIF file will be incomplete

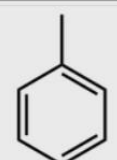
Home Work View Tools Info

HART

ReportPlus

FragmentDB

Toluene, C7H8



PART: -1 Free Variable: 1 Occupancy: 0.5 => 10.5

Use a residue:  Residue Class: TOL Invert:  Calculate DFIX:

Replace Mode:  No Restraints:  Rigid Group:

List of most disagreeable restraints:

Observed	Target	Error	Sigma	Restraint
--	--	--	--	--

AC4

Twining

You are in *mode* MODE\_DISP  
Press the ESC key to exit.



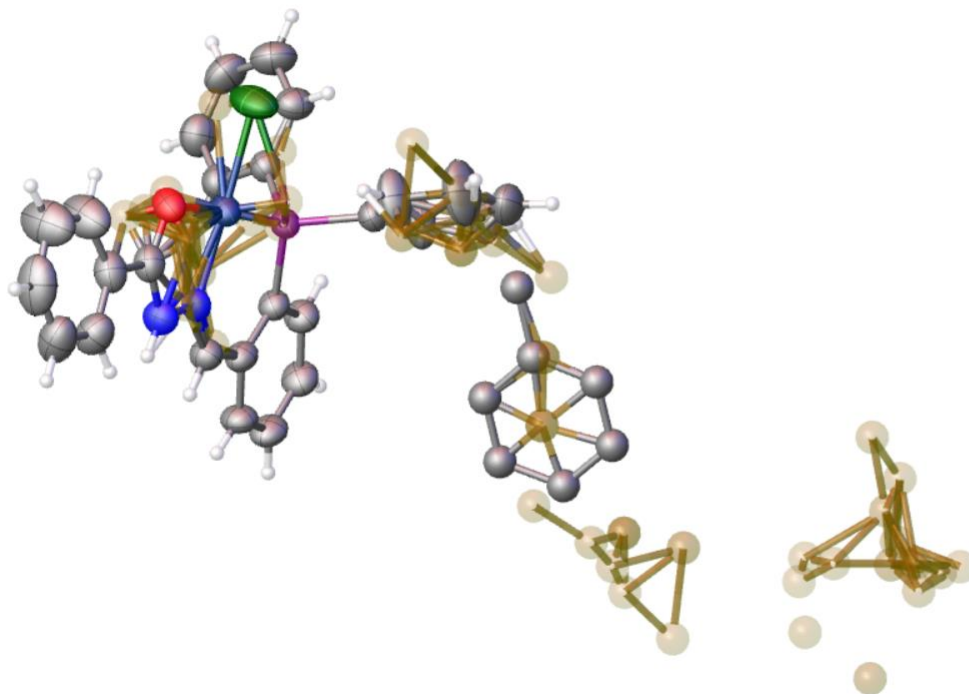
Once you have selected toluene from the list, hit “Fit”

A “shadow” toluene will appear.

Click on one atom in the shadow, and then click on one of your electron density peaks that you want to line up with the shadow.

Keep selecting one shadow peak (green) and one electron density peak (orange) until you get a reasonable fit. This took me three clicks.

Then hit “Esc”



c = 9.551(2)    V = 104.77(2)    V = 1297.1(5)    W/R2    21.00    70

d min (Mo)	0.71	I/a	32.1	Rint	n/a	complete	100%
Shift	0.224	Max Peak	3.3	Min Peak	-0.8	Goof	1.834

WARNING: Input data appear to be merged: CIF file will be incomplete

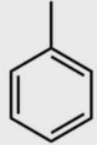
Home    Work    View    **Tools**    Info

HART

ReportPlus

FragmentDB

Toluene, C7H8   

PART:     Free Variable:     Occupancy:  =>

Use a residue:     Residue Class: TOL    Invert:     Calculate DFIX:

Replace Mode:     No Restraints:     Rigid Group:    

List of most disagreeable restraints:       

Observed	Target	Error	Sigma	Restraint
--	--	--	--	--

AC4

Twinning

To keep things ordered, I then named the toluene C50 – C56, and sorted my atoms, so that I could see what all this clicking did in my instruction file.

malbac.ins - Notepad

File Edit Format View Help

```
TITL r26
REM r26
REM 384 parameters refined using 6 restraints
CELL 0.71078 10.551 14.251 9.551 99.77 105.1 104.77
ZERR 2 0.002 0.003 0.002 0.02 0.02 0.02
LATT 1
SFAC C H Cl N O P Pd
UNIT 59 50 2 4 2 2 2
DFIX 1.51 C50 C51
DANG 2.42 C51 C53
FLAT C50 C51 C52 C53 C54 C55 C56
DFIX 1.39 C51 C52 C51 C56 C53 C54
DANG 2.39 C52 C54
DFIX 1.38 C52 C53 C54 C55 C55 C56
RIGU C50 > C56
SADI C51 C52 C52 C53 C53 C54 C54 C55 C55 C56 C56 C51
DANG 2.41 C51 C55
SADI 0.04 C51 C55 C51 C53 C56 C54 C52 C56 C53 C55 C52 C54
DANG 2.4 C53 C55 C54 C56
SIMU C50 > C56
SADI 0.04 C50 C56 C50 C52
DANG 2.52 C50 C52 C50 C56
```

```
PART -1
C50 1 0.22881 0.51794 -0.40920 10.50000 0.05000
C51 1 0.07692 0.50459 -0.47142 10.50000 0.05000
C52 1 0.03148 0.57907 -0.52536 10.50000 0.05000
C53 1 -0.10717 0.56792 -0.58177 10.50000 0.05000
C54 1 -0.20480 0.48040 -0.58520 10.50000 0.05000
C55 1 -0.16180 0.40549 -0.53257 10.50000 0.05000
C56 1 -0.02268 0.41731 -0.47593 10.50000 0.05000
HKLF 4
```

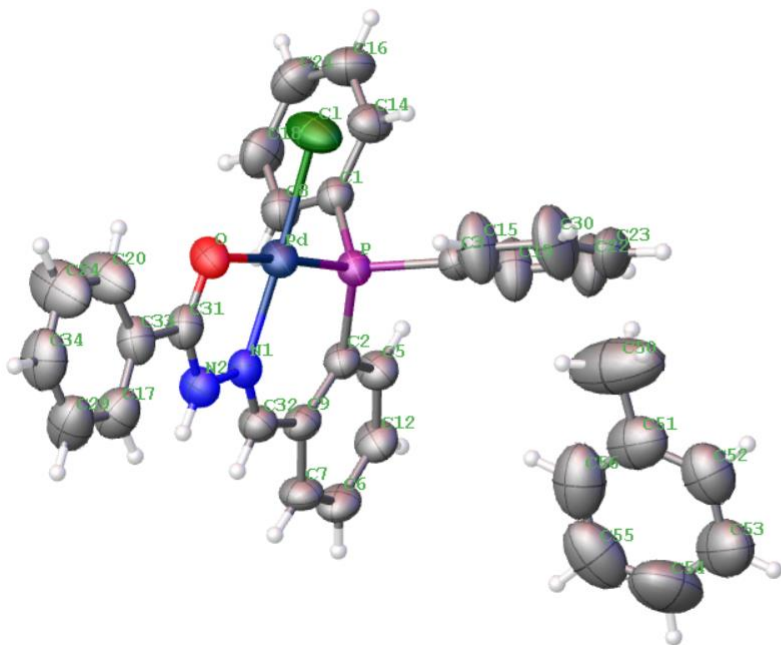
```
END
```

Close your .ins and Refine.

Make your toluene anisotropic, and Refine.

Add your hydrogen atoms, and Refine.

If you “grow”, OLEX2 does not show you the other “half”, but if you open your structure in Mercury, or if you go to “View → Symmetry Generation → Packing → Pack to limits” (for example), your full disorder model will be visible.



A screenshot of the malbac software interface. The main window displays the chemical formula C29.5H25ClN2OPd and various crystallographic parameters. The 'Refine' button is circled in red. The 'Toolbox Work' section at the bottom also has the 'Add H' button circled in red.

Parameter	Value	Unit
a	10.551(2)	Å
b	14.251(3)	Å
c	9.551(2)	Å
$\alpha$	99.77(2)	°
$\beta$	105.10(2)	°
$\gamma$	104.77(2)	°
Z	2	
Z'	1	
V	1297.1(5)	Å <sup>3</sup>

Parameter	Value	Unit
d min (Mo)	0.71	Å
I/I <sub>0</sub>	32.1	
R <sub>1</sub>	4.34	%
wR <sub>2</sub>	12.69	%
Shift	0.101	Å
Max Peak	1.4	Å
Min Peak	-0.9	Å
Goof	1.016	

Warning: 1 atoms may be split and 0 atoms NPD

Program: ShelXL | LS | Cycles: 5 | Peaks: 25

hkl file: malbac.hkl | hkl: Mon May 18 17:35:37 2020

Weight:  0.082 | 0.077 | 0.000 | 0.000 | EXTI  0.0225(18) |  NOVA

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

Refinement Settings Extra

Toolbox Work

Labels: Labels OFF/ON

Add H

Z' = 1

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

Metal-L | PARTs | SHELLs

CTRL+J |  Auto Apply |  M-L |  H-bond H only

thin H bonds |  PARTs |  Fog | 20 | 23 | Reload Style