

Olex Tutorial

the full process for solving a small molecule crystal

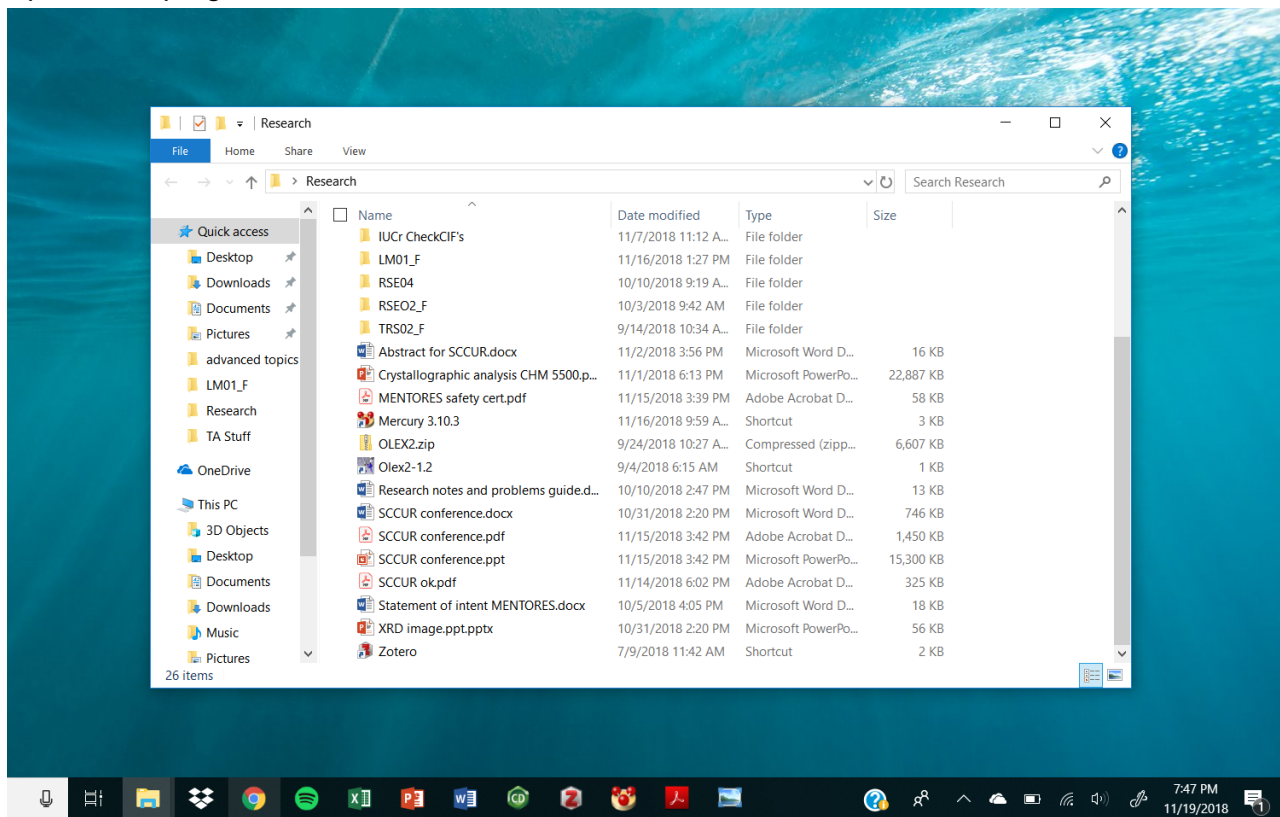
Before starting this tutorial please read the following

- I. This tutorial is to familiarize students with the process for solving a crystal
- II. All of the files for this tutorial have to be located in the same place in order for the tutorial to work.
- III. Students should look at the next step of the tutorial if they are confused on a step before asking for help, this may answer their question.
- IV. If clicking refine ever results in an atom that is large or not supposed to be there, delete the atom and refine the system. Then name the q-peak that appears with the proper name if it is an atom that needs to be there. The full structure is seen in step 15.
- V. This tutorial needs to have color for the purpose of assigning atoms properly. If printed please print in color, or please give access to an electronic copy.

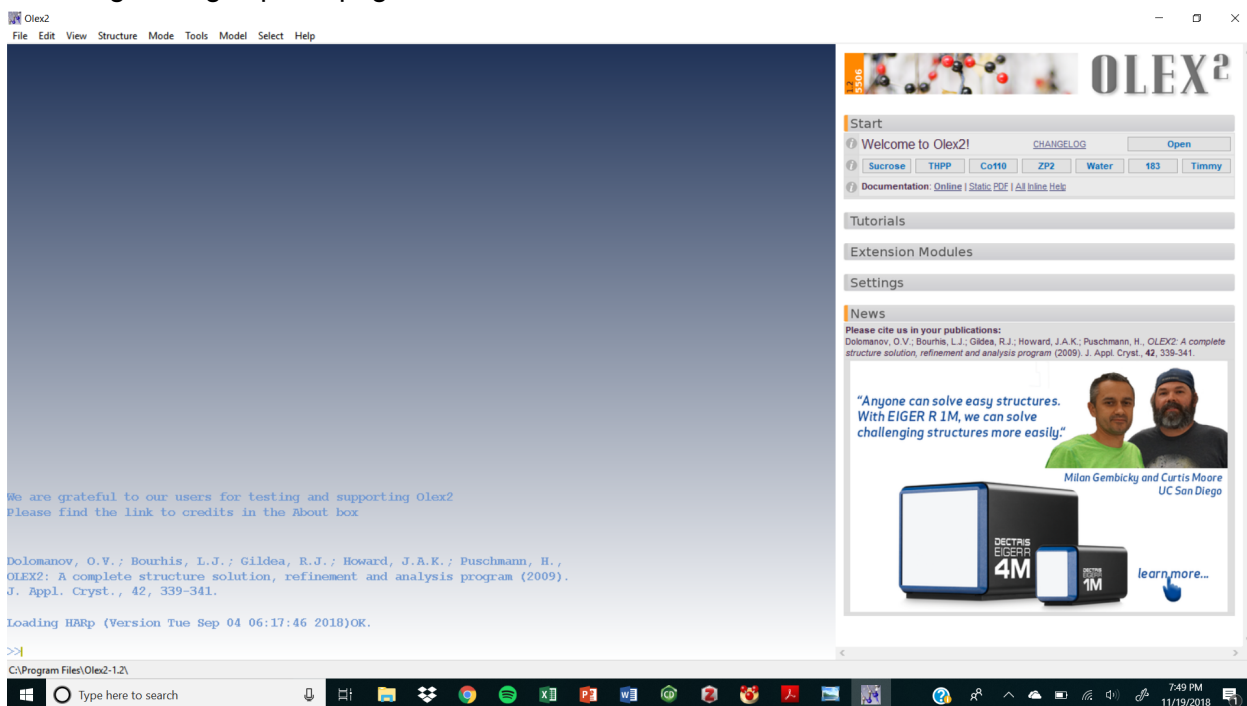
Enjoy and hopefully you will understand how small molecule systems are determined to a publishable quality by the end of this.

Olex²

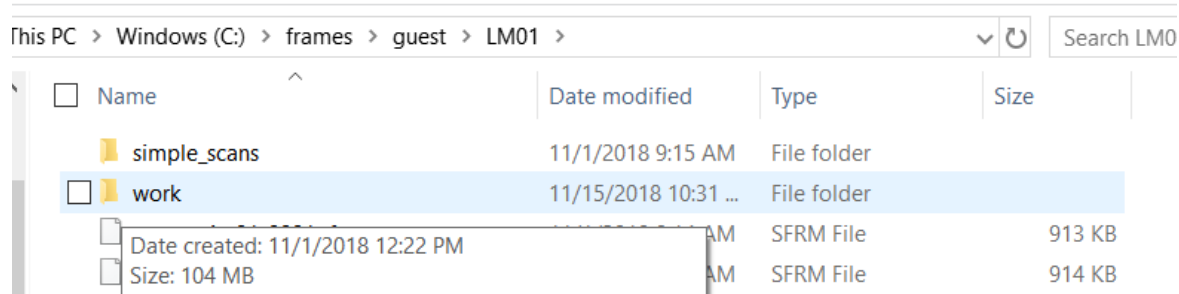
1. Open Olex² program labeled "Olex2-1.2"



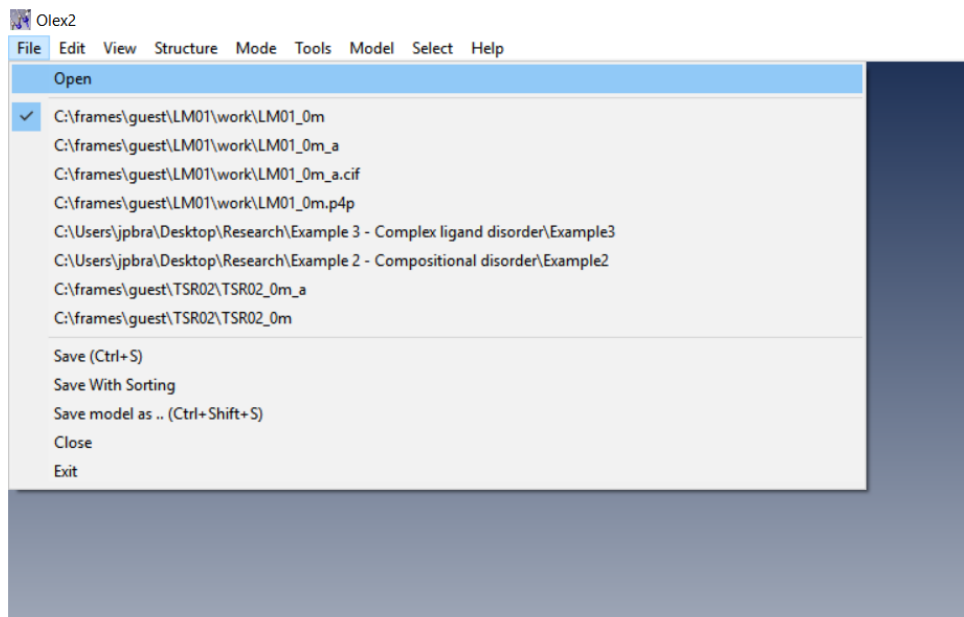
2. Selecting it brings up this page



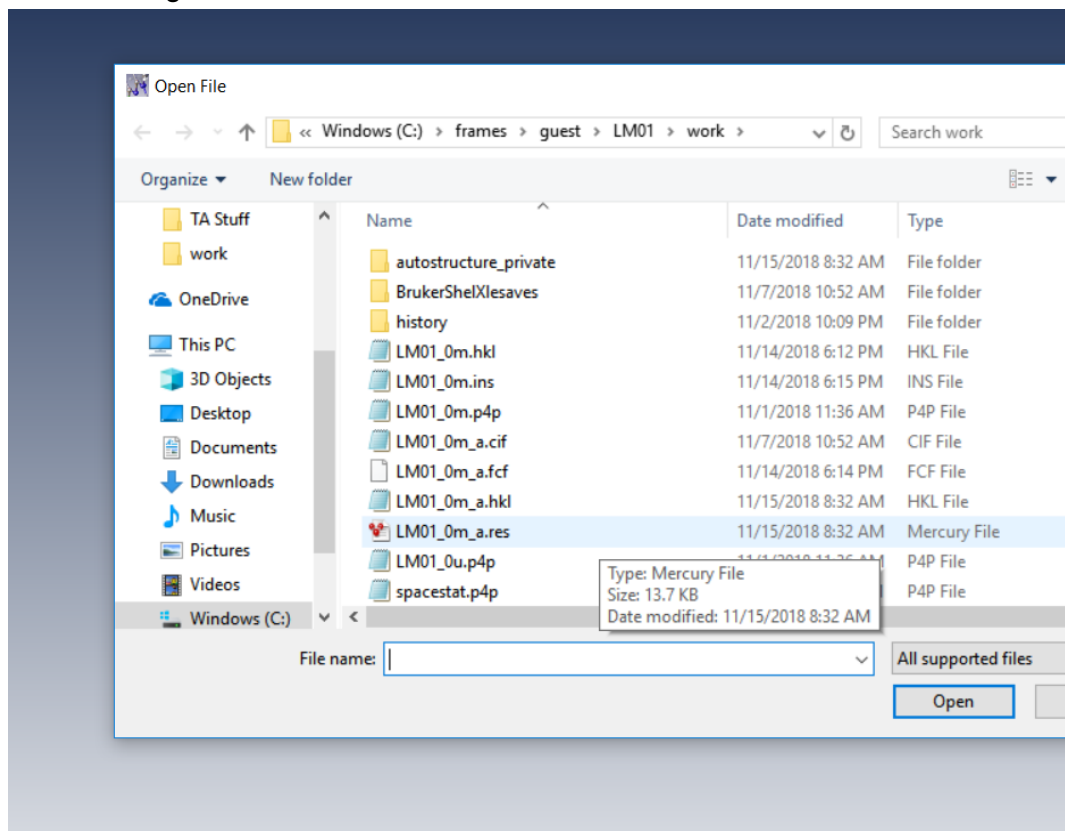
- Now we must make sure that we have the proper files, all of which should be given to you in a series of downloads and can be found in the download folder.



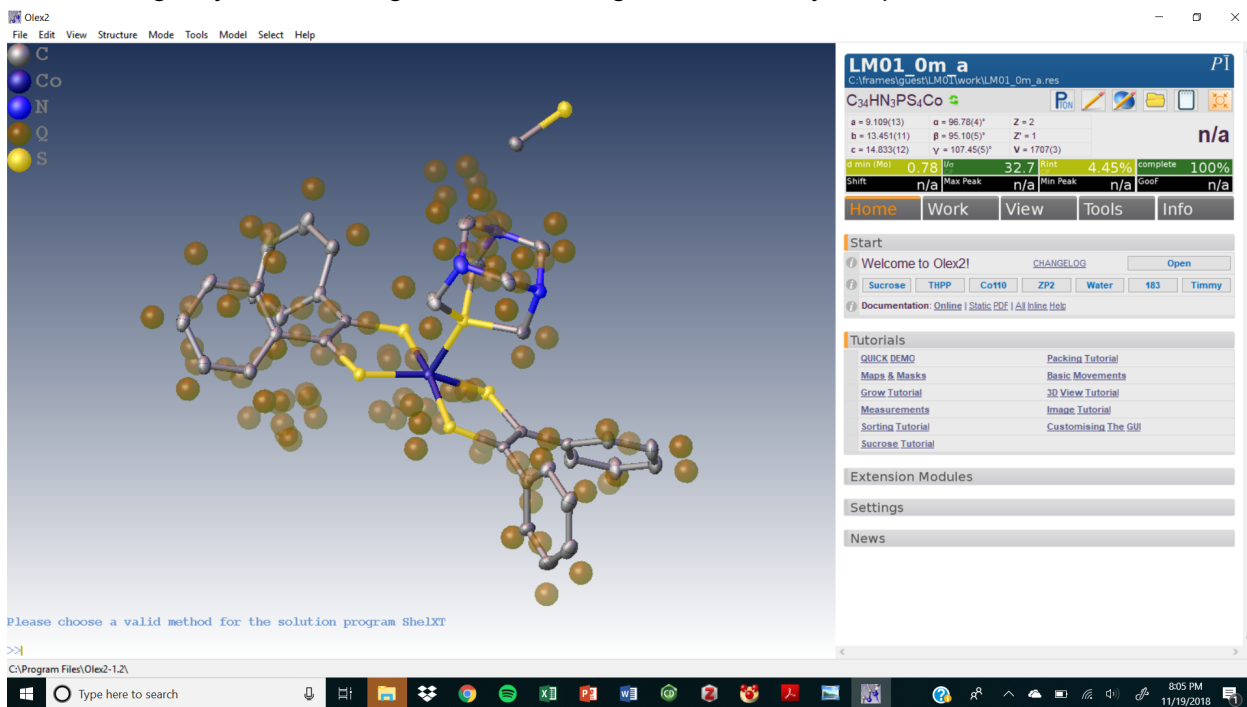
- Inside of the download folder it should contain the following file types
 - LM01_0m.hkl
 - LM01_0m.p4p
 - LM01_0m.lst
 - LM01_0m.fcf
 - Lm01_0m.res
- After making sure that your files are downloaded properly you can open the structure in Olex by going to
 - File
 - Open



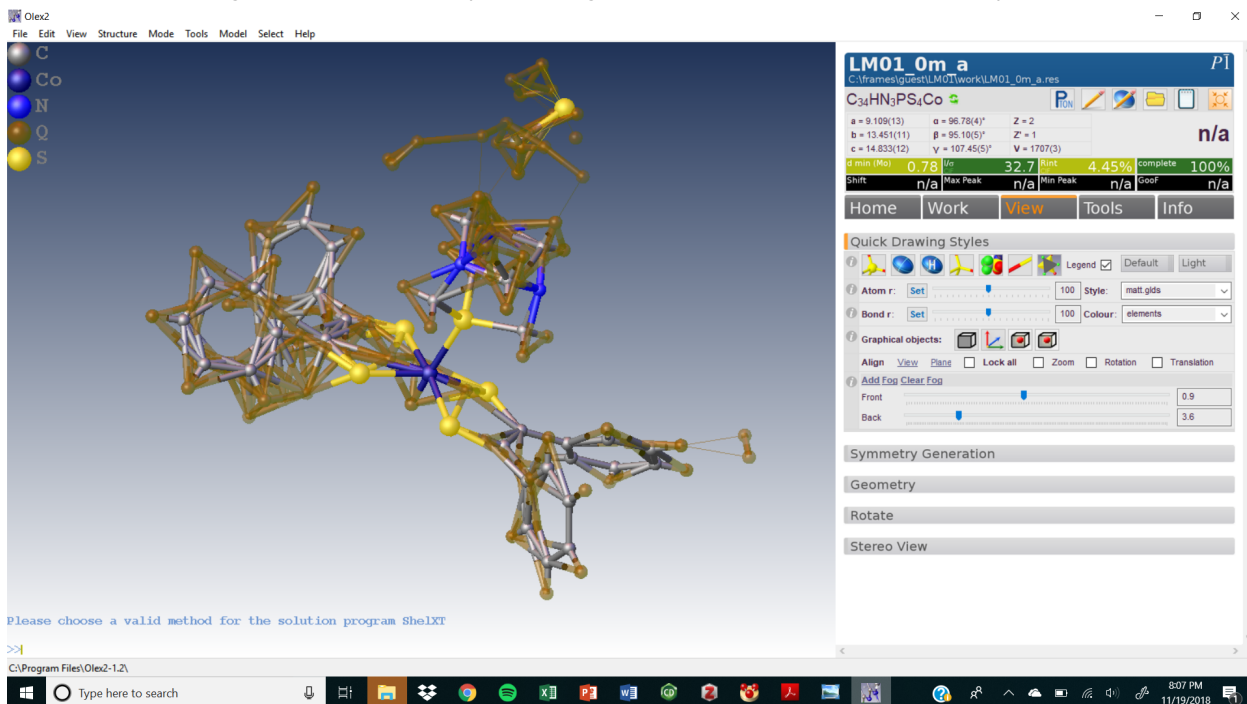
ii. And selecting the RES file in the Work folder



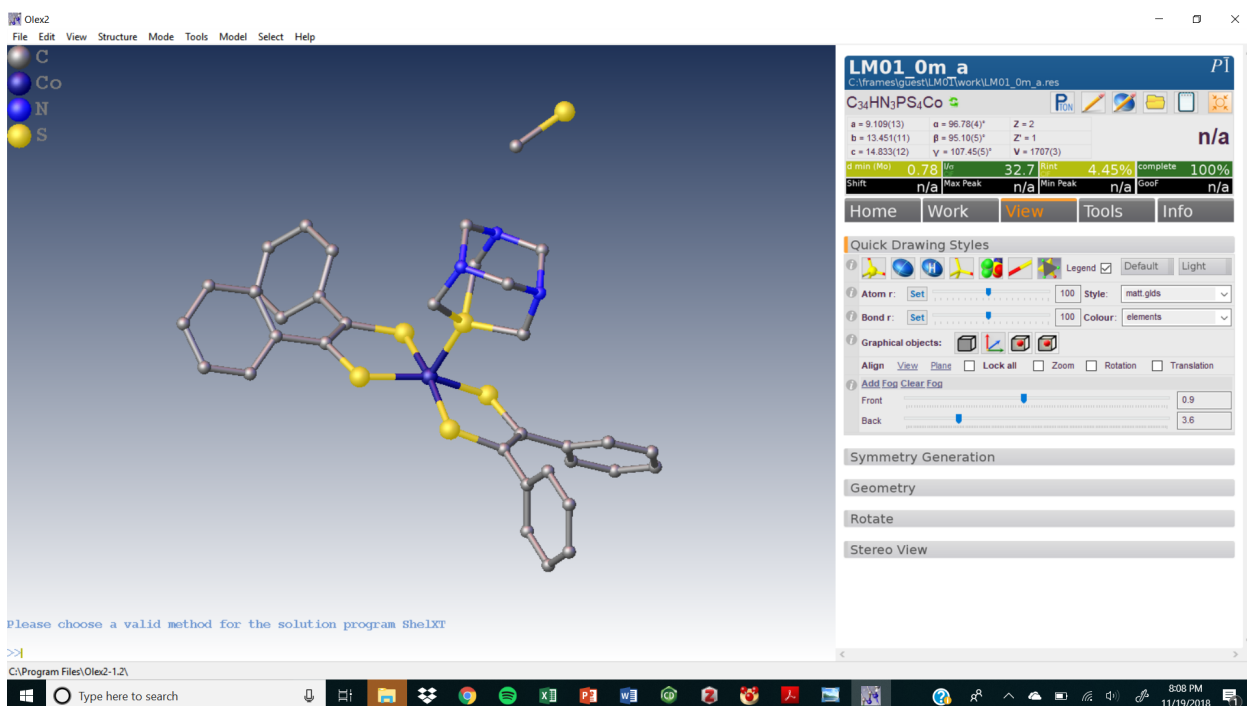
6. Which will give you something like the following screen when you open this file.



7. To clean up the picture we are going to hide the brown spheres known as Q-Peaks (unassigned electron density). To do this press “control Q.” After pressing once it will show the following screen, for Mac (a two-finger scroll should eliminate them).



8. Repeat this command and it will eliminate the Q-peaks, or continue scrolling to remove them.



9. go to the “solve” tab under the “work” insert.

| | | | | |
|----------------|----------------|-------------------------|---------------------|----------|
| c = 14.833(12) | γ = 107.45(5)° | V = 1707(3) | | |
| d min (Mo) | 0.78 | I/σ _{CIF} 32.7 | Rint _{CIF} | |
| Shift | n/a | Max Peak | n/a | Min Peak |

Home **Work** View

Solve Refine Draw

Toolbox Work

Labels Labels OFF/ON

10. Select solve and then select the arrow next to the word solve, you will need to check the assign box and auto box if they are not already selected after you solve the structure. This assigns atoms to the Q-Peaks determined by the solve function.
11. Now that the system is solved and cleaned up it is important to know what the chemical formula is. For this sample it is (C₃₅ H₃₇ Co₁ S₄ N₃ P₁ Cl₁) where the atom type is given followed by the number of atoms of that type. Change the structure to match this formula by changing atoms such as Sulfur, Phosphorus, Nitrogen, and Chlorine by going to the “Work” tab in the sidebar options.

LM01_0m a PI

C:\frames\guest\LM01\work\LM01_0m_a.res

C₃₄H₃N₃P₁S₄Co

| | | |
|----------------|----------------|-------------|
| a = 9.109(13) | α = 96.78(4)° | Z = 2 |
| b = 13.451(11) | β = 95.10(5)° | Z' = 1 |
| c = 14.833(12) | γ = 107.45(5)° | V = 1707(3) |

| | | | | | | | |
|------------|------|-------------------------|---------------------------|---------------|-----|------|-----|
| d min (Mo) | 0.78 | I/σ _{CIF} 32.7 | Rint _{CIF} 4.45% | complete 100% | | | |
| Shift | n/a | Max Peak | n/a | Min Peak | n/a | Goof | n/a |

Home **Work** View Tools Info

Solve Refine Draw Report

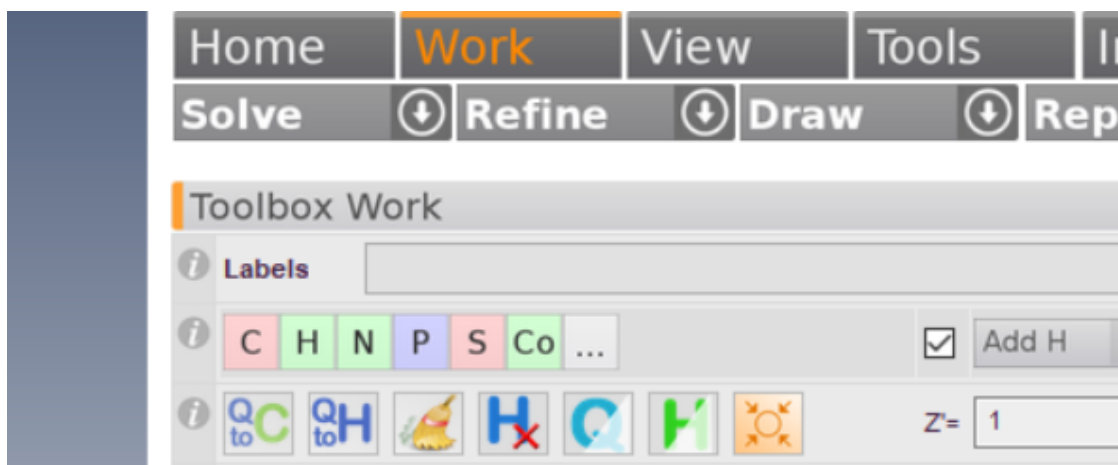
Toolbox Work

Labels Labels OFF/ON

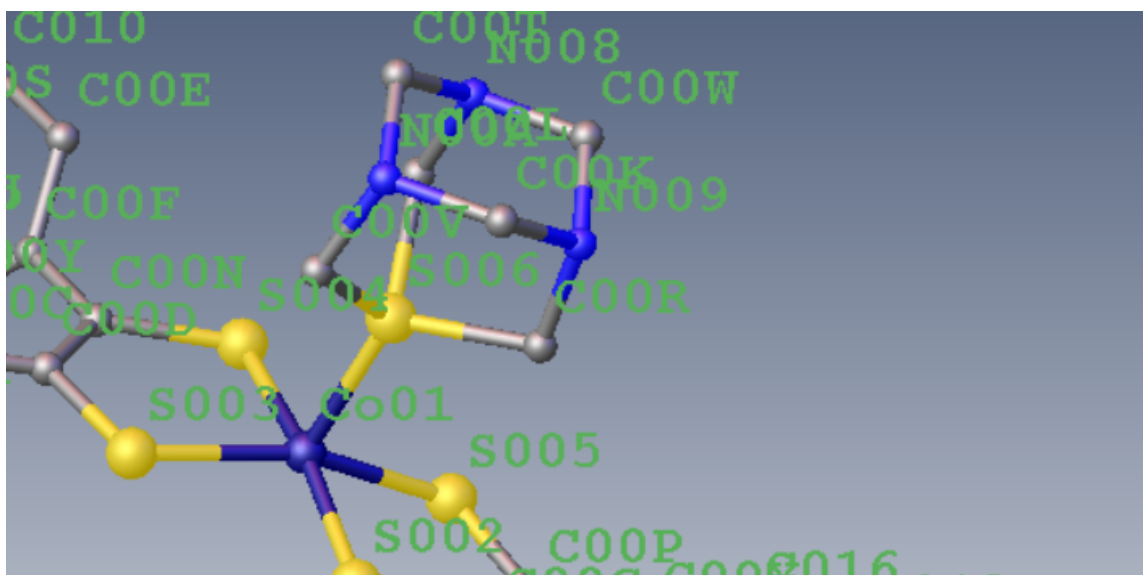
C H N P S Co ... Add H

Z = 1

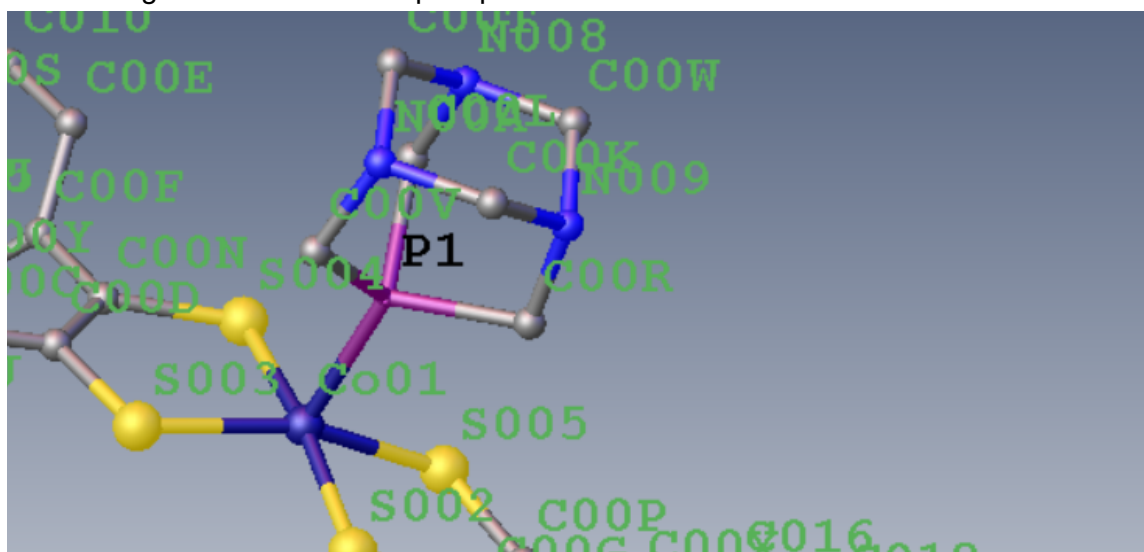
12. Select "P" in the "Toolbox Work" Insert.



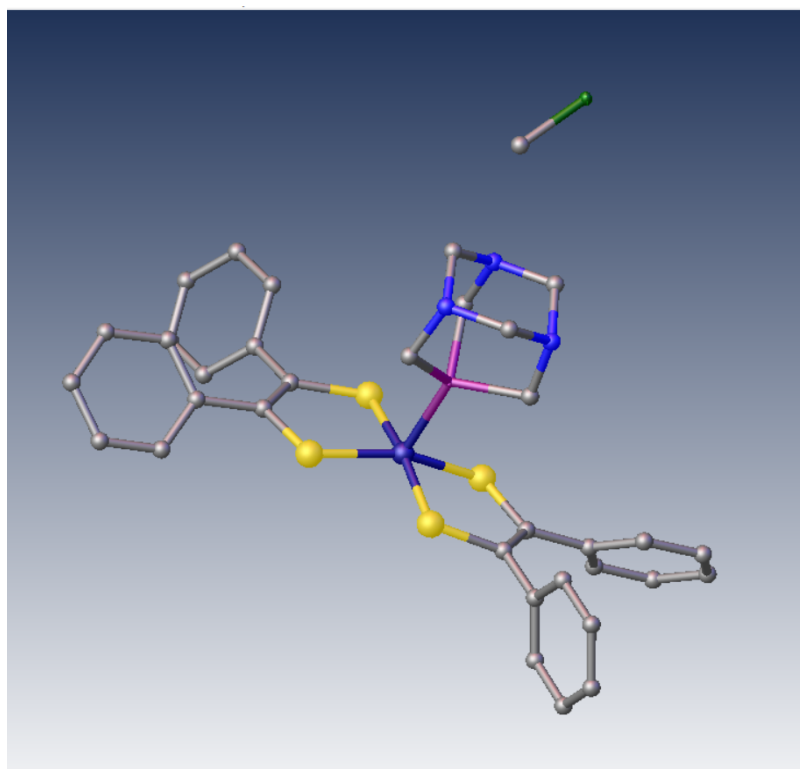
13. Then select the atom in the center above the cobalt core.



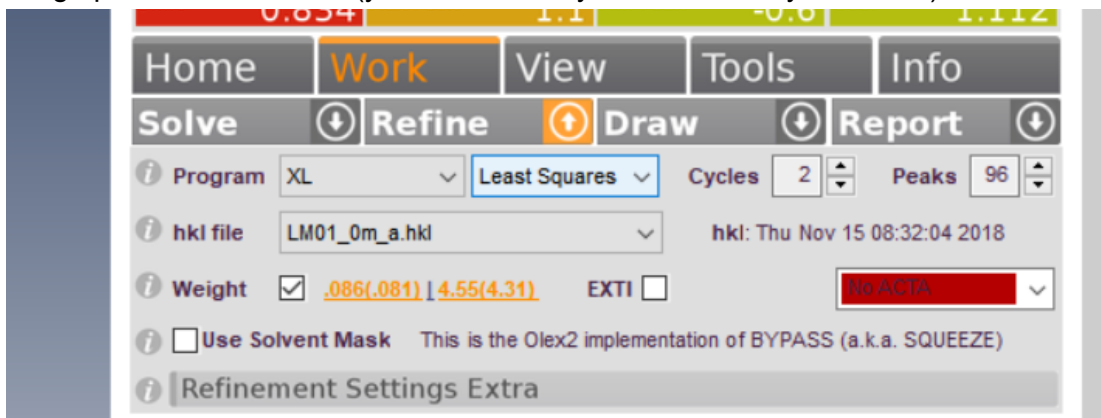
14. It will change sulfur "S006" to a phosphorus "P1" atom as shown.



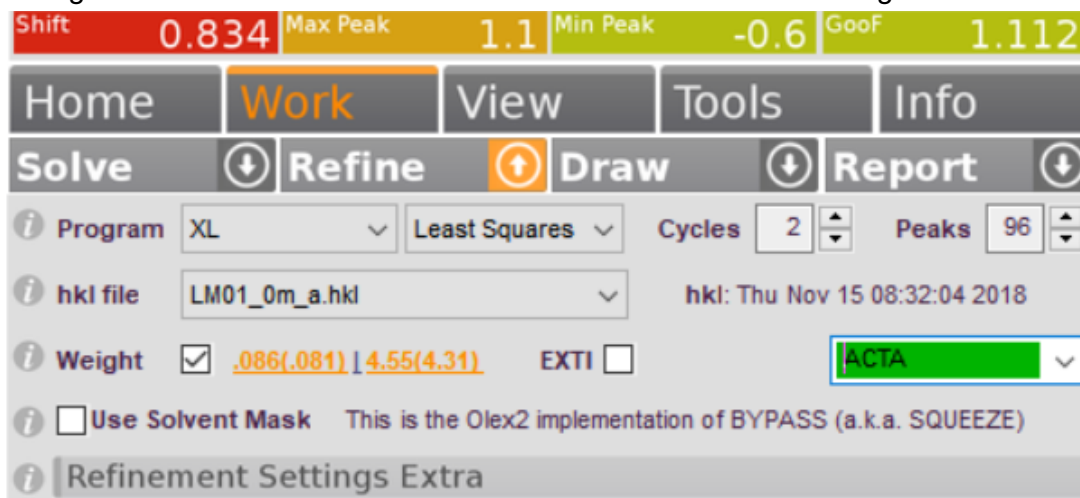
15. Repeat this until the structure has only the number of atoms and the labels that are in the chemical formula (C35 H37 Co1 S4 N3 P1 Cl1) which will result in the following. The solvent may be on the top or bottom of this structure due to the repeating unit cell containing half of the solvent. Depending on the half that is solved it will either be on top or on bottom. Hydrogen atoms are not placed yet so do not worry about them yet. They will be placed later in the tutorial. Make sure not to select a Q peak for the chlorine in the DCE molecule, the solve will automatically place an atom there. If there is a small (almost non-existent) atom that is the chlorine, use shift+click and drag to highlight it then change it to chlorine.



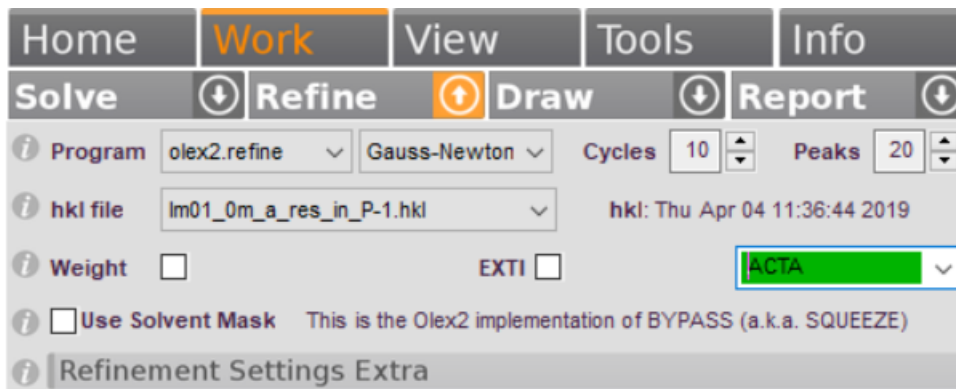
16. After “solving” and “renaming,” go to the “Refine” tab and select the down arrow. To bring up the refine window (your window may not look exactly the same)



17. Change the selection of “No Acta” to read “Acta” and select the “weight” box.



18. Now change the cycles position to 10, this will cause the refinement to happen 10 times. At this time make sure that the OLEX2.refine is in the program box, and that the GaussNewton method of refinement is chosen in the box next to it.



19. Now click on the refine tab and you should see it processing followed by much better R, wR2, GOOF, and Shift values indicated by colors that are closer to green and further from red. If it is not very close to green just click refine again until the weight numbers are green.
20. An anisotropic refinement (where electron densities are not treated as perfect spheres) will need to be done to get the R₁ value below 5% to perform this select the blue ellipsoid in toolbox right of the add H button. The weight should also be converged this will be indicated by green numbers next to the box checked weight. If these numbers are not present select the checkbox and do another refinement. When the value for R₁ is below 5% then you are done with this refining process.

| | | | | | | | |
|-----------------|-----------------|---------------|-----------------|----------|-------|----------|-------|
| a = 9.0954(7) | α = 97.074(3)° | Z = 2 | R ₁ | 4.61 % | | | |
| b = 13.4319(9) | β = 94.680(3)° | Z' = 1 | wR ₂ | 16.68 % | | | |
| c = 14.7905(10) | γ = 107.354(3)° | V = 1698.1(2) | | | | | |
| d min (Mo) | 0.78 | I/σ | 32.7 | Rint | 4.45% | complete | 100% |
| Shift | 0.001 | Max Peak | 1.2 | Min Peak | -0.6 | Goof | 1.050 |

Home **Work** View Tools Info

Solve **Refine** Draw Report

Program: olex2.refine Gauss-Newton Cycles: 10 Peaks: 99

hkl file: lm01_0m_a_res_in_P-1.hkl hkl: Thu Apr 04 11:36:44 2019

Weight: .100(.101) | 4.81(4.81) EXTI ACTA

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

Refinement Settings Extra

21. Now we need to generate a crystallographic information file (CIF) and see if there are any issues with the CIF. Check the folder where you have all of the files stored for a .cif file, if there is not one generate the CIF by selecting the Report tab, this will cause a computation for a moment then a window will pop up, select ok. You will then get another pop-up window that you can exit out of and then it will open a page in your web browser. Which you can close out of. If you have the .cif file (which you should have) you can skip this step.

LM01_0m_a

Table 1 Crystal data and structure refinement for LM01_0m_a.

| | |
|---|---|
| Identification code | LM01_0m_a |
| Empirical formula | C ₃₄ HN ₃ PS ₄ CoCl _{0.5} |
| Formula weight | 686.23 |
| Temperature/K | 113.0 |
| Crystal system | triclinic |
| Space group | P-1 |
| a/Å | 9.109(13) |
| b/Å | 13.451(11) |
| c/Å | 14.833(12) |
| α/° | 96.78(4) |
| β/° | 95.10(5) |
| γ/° | 107.45(5) |
| Volume/Å ³ | 1707(3) |
| Z | 2 |
| ρ _{calc} /g/cm ³ | 1.335 |
| μ/mm ⁻¹ | 0.860 |
| F(000) | 679.0 |
| Crystal size/mm ³ | 0.571 × 0.317 × 0.138 |
| Radiation | MoKα (λ = 0.71073) |
| 2θ range for data collection/° | 3.92 to 54.326 |
| Index ranges | -11 ≤ h ≤ 11, -17 ≤ k ≤ 17, -18 ≤ l ≤ 18 |
| Reflections collected | 46926 |
| Independent reflections | 7494 [R _{int} = 0.0445, R _{sigma} = 0.0306] |
| Data/restraints/parameters | 7494/0/406 |
| Goodness-of-fit on F ² | 1.154 |
| Final R indexes [I ≥ 2σ (I)] | R ₁ = 0.0460, wR ₂ = 0.1427 |
| Final R indexes [all data] | R ₁ = 0.0530, wR ₂ = 0.1590 |
| Largest diff. peak/hole / e Å ⁻³ | 1.05/-0.56 |

22. After making the CIF file you will need to go to "<https://checkcif.iucr.org>" to check the suitability of the CIF for publication, which will look like the following.

checkCIF is sponsored by

IUCr Journals

ELSEVIER

WILEY

IUCrData

ROYAL SOCIETY OF CHEMISTRY

checkCIF

A service of the
International Union of Crystallography

checkCIF reports on the consistency and integrity of crystal structure determinations reported in CIF format.

Please upload your CIF using the form below.

File name:
Choose File No file chosen

Select form of checkCIF report

- HTML
- PDF
- PDF (recommended for CIFs that might take a long time to check)

Select validation type

- Full validation of CIF and structure factors
- Full IUCr publication validation of CIF and structure factors
- Validation of CIF only (no structure factors)

Output Validation Response Form

- Level A alerts only
- Level A and B alerts
- Level A, B and C alerts
- None

Send CIF for checking

Information about this version of checkCIF ...

23. Now select your CIF file in your work folder, make sure that it is the CIF file type as well

| Name | Date modified | Type | Size |
|---|----------------------|------------------------|----------|
| LM01_05_ Js | 11/1/2018 11:35 A... | _LS File | 220 KB |
| LM01_06_ Js | 11/1/2018 11:35 A... | _LS File | 223 KB |
| LM01_07_ Js | 11/1/2018 11:36 A... | _LS File | 220 KB |
| LM01_01_ ma | 11/1/2018 11:36 A... | _MA File | 2,432 KB |
| LM01_0m_a.2fcf | 11/7/2018 10:16 A... | 2FCF File | 417 KB |
| LM01.abs | 11/2/2018 4:46 PM | ABS File | 13 KB |
| LM01_0m_a_tables.html | 11/20/2018 11:47 ... | Chrome HTML Do... | 47 KB |
| LM01.cht | 11/1/2018 11:36 A... | CHT File | 9,985 KB |
| <input checked="" type="checkbox"/> LM01_0m_a.cif | 11/20/2018 11:47 ... | CIF File | 1,408 KB |
| LM01_0m_a.ckf | 11/2/2018 10:42 PM | CKF File | 36 KB |
| rpitic.ini | 11/1/2018 11:35 A... | Configuration setti... | 4 KB |

24. Then select send CIF for checking at the bottom of the page, it may take a minute to perform, then you will be redirected to another page.

← → ↻ https://checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

Apps car stuff Account Inquiry

checkCIF (basic structural check) running

Checking for embedded fcf data in CIF ...

Found embedded fcf data in CIF. Extracting fcf data from uploaded CIF, please wait . .

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) lm01_0m_a

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found.
Please wait while processing

[CIF dictionary](#)
[Interpreting this report](#)

[Structure factor report](#)

Datablock: lm01_0m_a

| | | |
|------------------------|--|--------------------------|
| Bond precision: | C-C = 0.0057 A | Wavelength=0.71073 |
| Cell: | a=9.109(13) b=13.451(11) c=14.833(12) | |
| | alpha=96.78(4) beta=95.10(5) gamma=107.45(5) | |
| Temperature: | 113 K | |
| | Calculated | Reported |
| Volume | 1707(3) | 1707(3) |
| Space group | P -1 | P -1 |
| Hall group | -P 1 | -P 1 |
| Moiety formula | C34 Co N3 P S4, 0.5(C2 Cl2) | C34 Co N3 P S4, C Cl |
| Sum formula | C35 Cl Co N3 P S4 | C34 H0 Cl0.50 Co N3 P S4 |
| Mr | 715.97 | 686.23 |
| Dx, g cm ⁻³ | 1.393 | 1.335 |
| Z | 2 | 2 |
| Mu (mm ⁻¹) | 0.901 | 0.860 |

25. Scroll down until you can see the A, B, C, and G alerts. (your alerts may look different)

← → ↻ ⓘ https://checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

📱 Apps 📁 car stuff 📄 Account Inquiry

Alert level A

[ABSTY03_ALERT_1_A](#) The `_exptl_absorpt_correction_type` has been given as none.
However a value has been given for `_exptl_absorpt_process_details`.
From the CIF: `_exptl_absorpt_process_details` SADABS-2016/2 (Bruker,201

Alert level B

| | | | |
|-----------------------------------|--|--------|-------|
| PLAT043_ALERT_1_B | Calculated and Reported Mol. Weight Differ by .. | 29.74 | Check |
| PLAT057_ALERT_3_B | Correction for Absorption Required RT(exp) ... | 1.22 | Do ! |
| PLAT919_ALERT_3_B | Reflection # Likely Affected by the Beamstop ... | 4 | Check |
| PLAT934_ALERT_3_B | Number of (Iobs-Icalc)/SigmaW > 10 Outliers ... | 6 | Check |
| PLAT939_ALERT_3_B | Large Value of Not (SHELXL) Weight Optimized S . | 329.48 | Check |

Alert level C

[ABSTY03_ALERT_1_C](#) The `_exptl_absorpt_correction_type` has been given as none.
However values have been given for `Tmin` and `Tmax`. Remove these if an absorption correction has not been applied.
From the CIF: `_exptl_absorpt_correction_T_min` 0.661
From the CIF: `_exptl_absorpt_correction_T_max` 0.746

[SHFSU01_ALERT_2_C](#) The absolute value of parameter shift to su ratio > 0.05
Absolute value of the parameter shift to su ratio given 0.052
Additional refinement cycles may be required.

| | | | |
|-----------------------------------|--|--------|--------|
| PLAT041_ALERT_1_C | Calc. and Reported SumFormula Strings Differ | Please | Check |
| PLAT051_ALERT_1_C | Mu(calc) and Mu(CIF) Ratio Differs from 1.0 by . | 4.73 | % |
| PLAT068_ALERT_1_C | Reported F000 Differs from Calcd (or Missing)... | Please | Check |
| PLAT148_ALERT_3_C | s.u. on the a - Axis is (Too) Large ... | 0.013 | Ang. |
| PLAT601_ALERT_2_C | Structure Contains Solvent Accessible VOIDS of . | 32 | Ang**3 |
| PLAT911_ALERT_3_C | Missing FCF Refl Between Thmin & STh/L= 0.600 | 18 | Report |
| PLAT918_ALERT_3_C | Reflection(s) with I(obs) much Smaller I(calc) . | 5 | Check |

Alert level G

[FORMU01_ALERT_1_G](#) There is a discrepancy between the atom counts in the `_chemical_formula_sum` and `_chemical_formula_moiety`. This is usually due to the moiety formula being in the wrong format.
Atom count from `_chemical_formula_sum`: C34 Cl0.5 Co1 N3 P1 S4
Atom count from `_chemical_formula_moiety`:C35 Cl1 Co1 N3 P1 S4

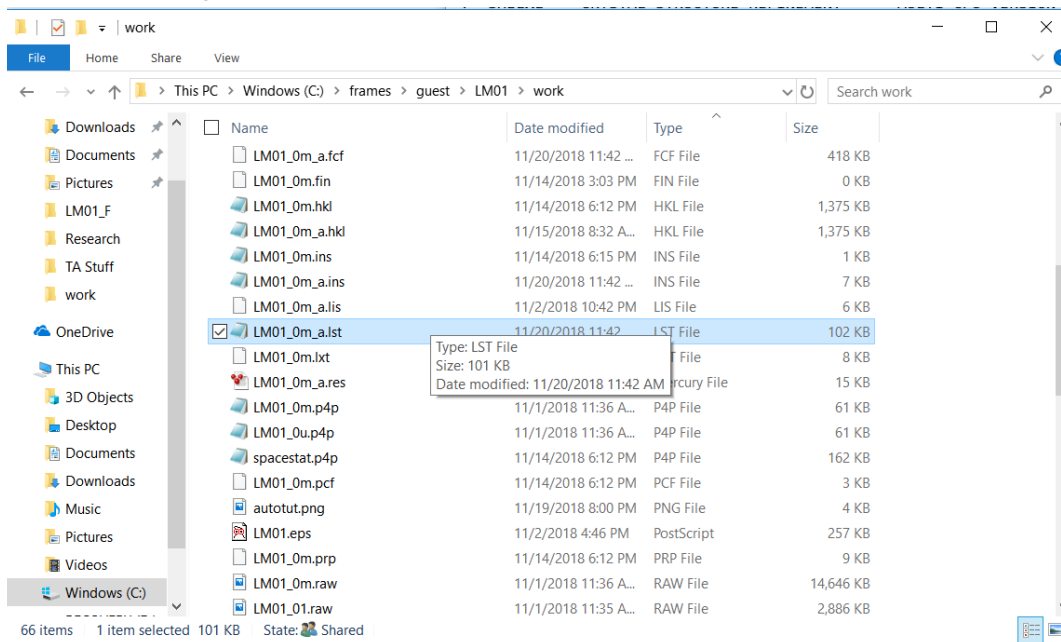
[FORMU01_ALERT_2_G](#) There is a discrepancy between the atom counts in the `_chemical_formula_sum` and the formula from the `_atom_site*` data.
Atom count from `_chemical_formula_sum`:C34 Cl0.5 Co1 N3 P1 S4
Atom count from the `_atom_site` data: C35 Cl1 Co1 N3 P1 S4

[CELLZ01_ALERT_1_G](#) Difference between formula and atom_site contents detected.

[CELLZ01_ALERT_1_G](#) ALERT: Large difference may be due to a symmetry error - see SYMMG tests
From the CIF: `_cell_formula_units_Z` 2
From the CIF: `_chemical_formula_sum` C34 H0 Cl0.50 Co N3 P S4
TEST: Compare cell contents of formula and atom_site data

26. In order to complete the CIF file for publication, we will need to remove as many of the A, B, and C alerts as possible since these signify critical errors in the CIF.

- a. The majority of reflections can be omitted by going to the info tab, selecting the bad reflection box, and selecting omit. It can also be done as presented in the following steps.



27. In programs outside of Olex editing the RES file as shown in the following steps is the best way to do this, and there are extra reflections that will need to be omitted as seen in step 33 and will need to use the following steps to alter. Let's start by removing the bad reflections due to the beam stop. To do this we will need to look in our "LST" file. Open the LST file. Once open do a find function using "control F" or "Command F" for mac,

and search for the word “disagreeable.”

Find what: disagreeable

Direction: Up Down

Most **Disagreeable** Reflections (* if suppressed or used for Rfree).
 Error/esd is calculated as $\sqrt{wD^2/\langle wD^2 \rangle}$ where w is given by the weight formula, $D = F_o^2 - F_c^2$ and $\langle \rangle$ refers to the average over all reflections.

| h | k | l | Fo ² | Fc ² | Error/esd | Fc/Fc(max) |
|----|-----|---|-----------------|-----------------|-----------|------------|
| 0 | -1 | 3 | 0.00 | 7309.86 | 15.57 | 0.505 |
| -1 | 1 | 0 | 0.66 | 5890.67 | 15.34 | 0.454 |
| 1 | 0 | 0 | 0.33 | 5185.70 | 15.19 | 0.426 |
| 0 | -1 | 1 | 0.00 | 954.14 | 11.59 | 0.183 |
| 0 | 1 | 1 | 0.22 | 798.70 | 11.04 | 0.167 |
| -1 | 4 | 3 | 427.41 | 51.50 | 10.26 | 0.042 |
| 2 | 1 | 2 | 273.75 | 44.32 | 8.00 | 0.039 |
| -3 | 0 | 4 | 192.98 | 20.45 | 7.65 | 0.027 |
| 2 | -3 | 4 | 147.15 | 8.29 | 7.48 | 0.017 |
| 1 | -7 | 2 | 125.76 | 2.90 | 7.26 | 0.010 |
| 3 | -3 | 3 | 206.91 | 34.92 | 6.96 | 0.035 |
| -7 | 14 | 2 | 471.73 | 145.85 | 6.41 | 0.071 |
| 0 | 3 | 2 | 711.26 | 303.71 | 6.26 | 0.103 |
| -1 | 3 | 4 | 261.44 | 72.77 | 6.14 | 0.050 |
| -3 | 0 | 7 | 239.10 | 63.59 | 6.08 | 0.047 |
| 1 | -3 | 5 | 230.43 | 60.97 | 6.05 | 0.046 |
| -2 | 1 | 3 | 4781.05 | 2552.12 | 5.95 | 0.299 |
| 1 | 1 | 3 | 287.84 | 98.17 | 5.63 | 0.059 |
| 1 | 2 | 3 | 4804.70 | 2758.50 | 5.58 | 0.310 |
| 0 | -4 | 3 | 152.97 | 33.02 | 5.52 | 0.034 |
| 4 | -4 | 1 | 189.84 | 55.19 | 5.18 | 0.044 |
| -2 | 2 | 1 | 28709.21 | 17026.00 | 5.17 | 0.771 |
| 6 | -13 | 2 | 243.88 | 54.30 | 5.16 | 0.044 |
| -4 | -4 | 1 | 71.30 | 4.66 | 5.12 | 0.013 |

28. We will need find areas where the Fo² is between 1 and 0, while the Fc² is a much higher number. There should only be 6. Write down the HKL numbers, which are the 3 numbers in front of the Fo², on a separate sheet of paper.

29. Now go back into Olex² and select the icon with just a pencil, highlighted below.

| | | |
|-------------|------------------|---------|
| Z = 2 | R ₁ | 4.60 % |
| Z' = 1 | wR ₂ | 15.90 % |
| V = 1707(3) | R _{int} | 4.45 % |
| 32.7 | complete | 100 % |
| 1.1 | Min Peak | -0.6 |
| | GooF | 1.154 |

30. After selecting this you will be able to edit your RES file, scroll down to the bottom and locate the word "acta"

```
LATT 1
SFAC C H N P S Co Cl
UNIT 68 0 6 2 8 2 1

L.S. 2
PLAN -96 0 0
SIZE 0.138 0.317 0.571
TEMP -160.15
BOND $H
fmap 2
acta
WGHT 0.0805 4.3429
FVAR 0.54956
HKLF 4

REM <olex2.extras>
REM <HklSrc "%C:\\frames\\guest\\LM01\\work\\LM01_0m_a.hkl">
REM </olex2.extras>
<
```

OK Cancel

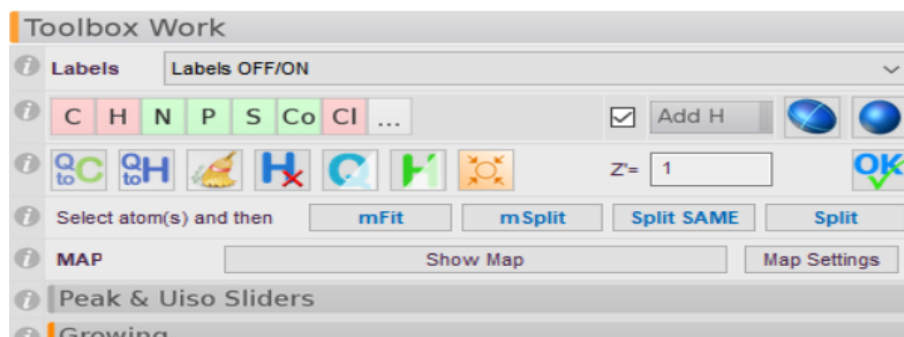
31. Hit enter after acta to go down a row and write "OMIT" then follow it with 1 set of the HKL numbers from the bad reflections. Repeat that until all the reflections have been selected to be omitted. (which can be seen in the next image/step)

```
SIZE 0.138 0.317 0.571
TEMP -160.15
BOND $H
fmap 2
acta
OMIT 0 -1 3
OMIT 1 0 0
OMIT -1 1 0
OMIT 0 -1 1
OMIT 0 1 1
OMIT 3 3 2
WGHT 0.0805 4.3429
FVAR 0.54956
HKLF 4

REM <olex2.extras>
REM <HklSrc "%C:\\frames\\guest\\LM01\\work\\LM01_0m_a.hkl">
REM </olex2.extras>
```

32. After doing this there are 2 other HKL reflections that are more difficult to identify than the five that were initially found, this takes a much more extensive search and because of the time that this can take to find it will be given to you just to move the tutorial along, the two extra sets are "-7 14 2," "-2 2 1," so additionally omit these HKL numbers as well. Select "OK" to close this window
33. After doing this refine the structure.

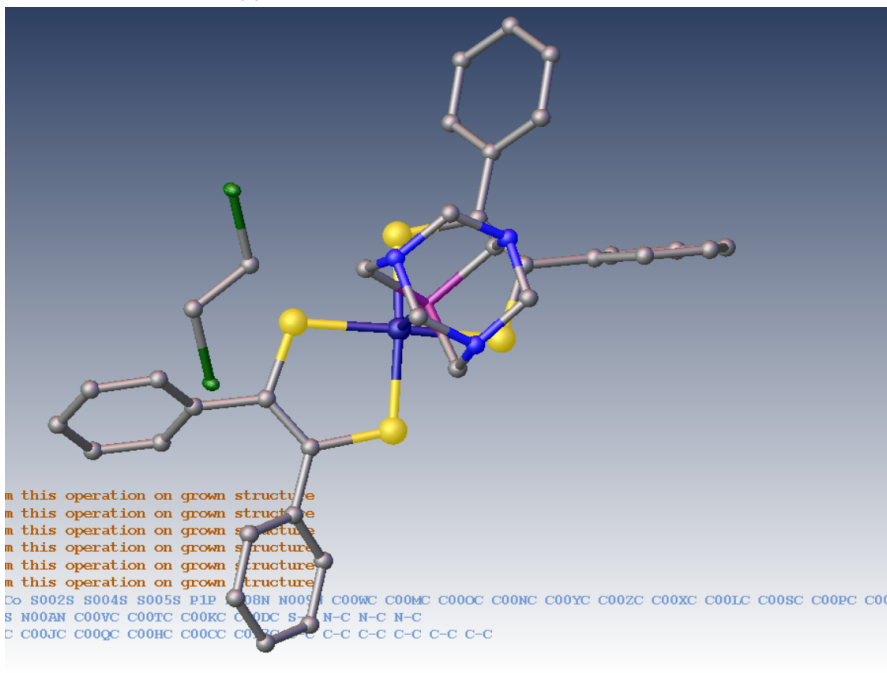
34. Now we need to add the hydrogen atoms to the system by going to the work tab and then the “toolbox work” insert and selecting add H in the box shown below.



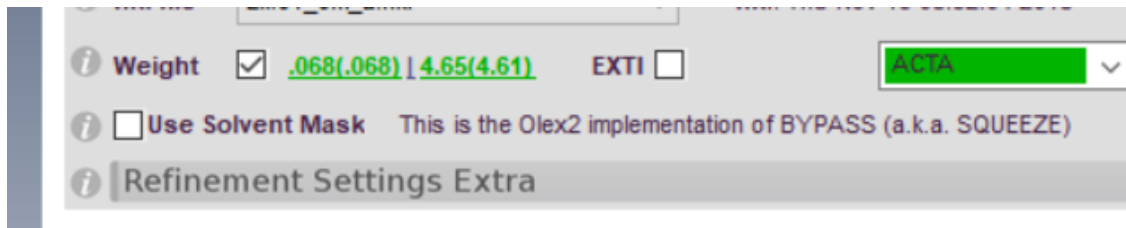
35. Now we need to take the mirrored solvent which is dichloroethane, and make it so that it is a whole molecule. To do this we need to “GROW it, go to the View tab then select the Symmetry Generation insert and in the three windows under the growing field have the three fields as indicated in the following image. You will need to select the grow all option even if it is pre-selected when you open the growing menu.



36. This should make your dichloroethane fully visible, and it might generate another molecule that you will need to delete by selecting the atoms and pressing the delete key. (Hydrogens omitted for clarity)



37. After making your dichloroethane we need to refine the system until the weight converges, make sure you are refining the system 10 cycles each time to make this process faster.



38. Now we will check the B alert about "Molar weight differ" by going into the RES folder like we did for omitting the reflections.

```

Edit
CELL 0.71073 9.1089 13.4509 14.8327 96.779 95.097 107.448
ZERR 2 0.0132 0.0108 0.0119 0.038 0.047 0.054
LATT 1
SFAC C H N P S Co Cl
UNIT 68 0 6 2 8 2 1

L.S. 10
PLAN -96 0 0
SIZE 0.138 0.317 0.571
TEMP -160.15
BOND $H
fmap 2
acta
OMIT 0 -1 3
OMIT 1 0 0
OMIT -1 1 0
OMIT 0 -1 1
OMIT 0 1 1

```

39. Notice where it says "UNIT" all of the hydrogen atoms are missing. This is an easy correction we just need to make sure that the unit reads the same as the Molecular formula (C₃₅ H₃₇ Co₁ S₄ N₃ P₁ Cl₁) that we should have, but due to there being two of these molecules in one unit cell we will need to double all of these values. Which should look like the following picture.

```
Edit
REM </autosolution>
CELL 0.71073 9.1089 13.4509 14.8327 96.779 95.097 107.448
ZERR 2 0.0132 0.0108 0.0119 0.038 0.047 0.054
LATT 1
SFAC C H N P S Co Cl
UNIT 70 74 6 2 8 2 2

L.S. 10
PLAN -96 0 0
SIZE 0.138 0.317 0.571
TEMP -160.15
BOND $H
fmap 2
acta
OMIT 0 -1 3
OMIT 1 0 0
OMIT -1 1 0
OMIT 0 -1 1
<
OK Cancel
```

- 5) "Crystal structure refinement with
40. Once the formula moieties match up, we will run another check CIF through IUCr

41. After running the check CIF, you should get the following errors (or similar)

← → ↻ ⓘ https://checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

Apps car stuff Account Inquiry

Click on the hyperlinks for more details of the test.

Alert level A

[ABSTY03_ALERT_1_A](#) The _exptl_absorpt_correction_type has been given as none.
However a value has been given for _exptl_absorpt_process_details.
From the CIF: _exptl_absorpt_process_details SADABS-2016/2 (Bruker,201

[PLAT415_ALERT_2_A](#) Short Inter D-H..H-X H008 ..H00W . 1.72 Ang.
x,y,z = 1_555 Check

Alert level B

[PLAT057_ALERT_3_B](#) Correction for Absorption Required RT(exp) ... 1.23 Do !

Alert level C

[ABSTY03_ALERT_1_C](#) The _exptl_absorpt_correction_type has been given as none.
However values have been given for Tmin and Tmax. Remove these if an absorption correction has not been applied.
From the CIF: _exptl_absorpt_correction_T_min 0.661
From the CIF: _exptl_absorpt_correction_T_max 0.746

[PLAT148_ALERT_3_C](#) s.u. on the a - Axis is (Too) Large 0.013 Ang.

[PLAT415_ALERT_2_C](#) Short Inter D-H..H-X H009 ..H000 . 2.02 Ang.
1+x,y,z = 1_655 Check

[PLAT415_ALERT_2_C](#) Short Inter D-H..H-X H009 ..H00R . 2.03 Ang.
x,-1+y,z = 1_545 Check

[PLAT420_ALERT_2_C](#) D-H Without Acceptor N008 --H008 . Please Check

And 2 other PLAT420 Alerts

[PLAT420_ALERT_2_C](#) D-H Without Acceptor N009 --H009 . Please Check

[PLAT420_ALERT_2_C](#) D-H Without Acceptor N00A --H00A . Please Check

[PLAT910_ALERT_3_C](#) Missing # of FCF Reflection(s) Below Theta(Min). 6 Note

[PLAT911_ALERT_3_C](#) Missing FCF Refl Between Thmin & STh/L= 0.600 22 Report

[PLAT976_ALERT_2_C](#) Check Calcd Resid. Dens. 1.03A From N009 -0.86 eA-3

And 2 other PLAT976 Alerts

[PLAT976_ALERT_2_C](#) Check Calcd Resid. Dens. 1.09A From N00A -0.70 eA-3

[PLAT976_ALERT_2_C](#) Check Calcd Resid. Dens. 1.08A From N008 -0.69 eA-3

[PLAT977_ALERT_2_C](#) Check Negative Difference Density on H008 -0.60 eA-3

And 2 other PLAT977 Alerts

[PLAT977_ALERT_2_C](#) Check Negative Difference Density on H009 -0.86 eA-3

[PLAT977_ALERT_2_C](#) Check Negative Difference Density on H00A -0.67 eA-3

Alert level G

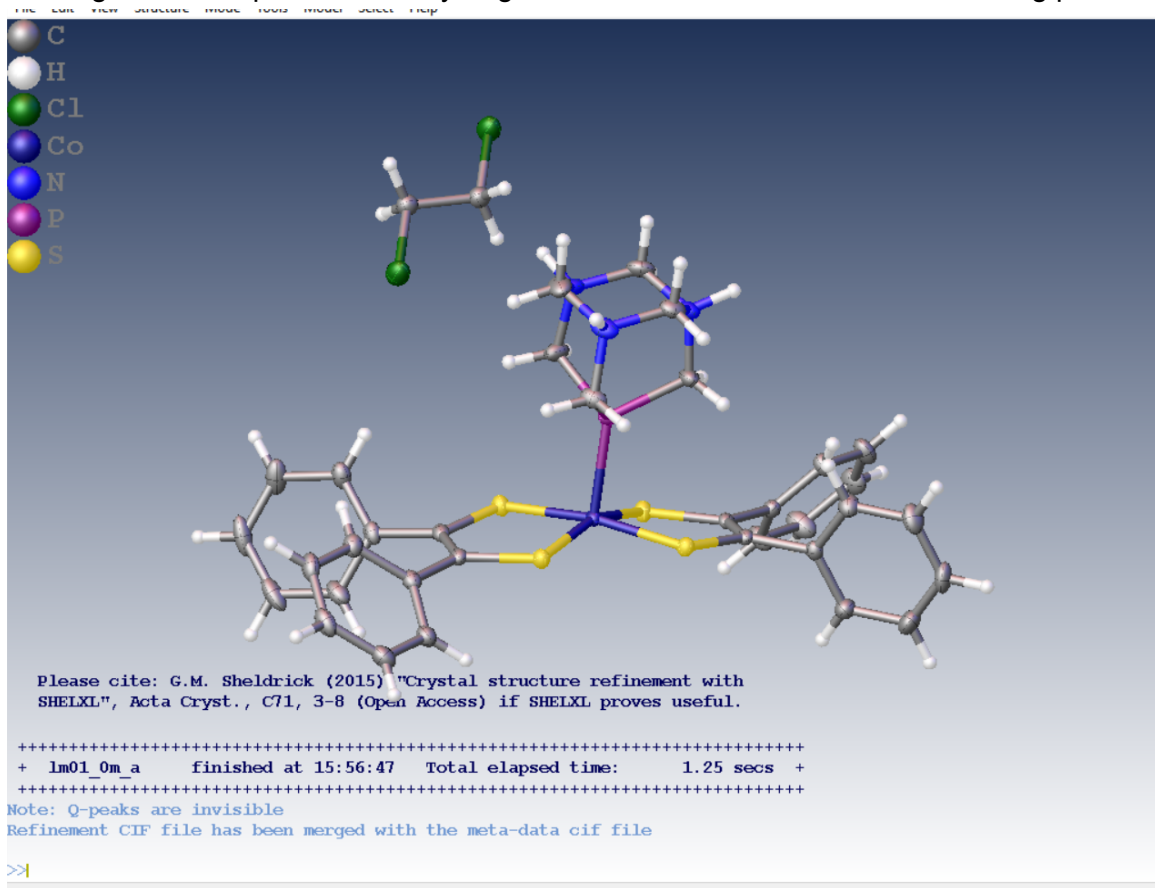
[PLAT007_ALERT_5_G](#) Number of Unrefined Donor-H Atoms 3 Report

[PLAT042_ALERT_1_G](#) Calc. and Reported MoietyFormula Strings Differ Please Check

[PLAT232_ALERT_2_G](#) Hinchfeld Test Diff (M-X) Co01 --S001 6 0 e "

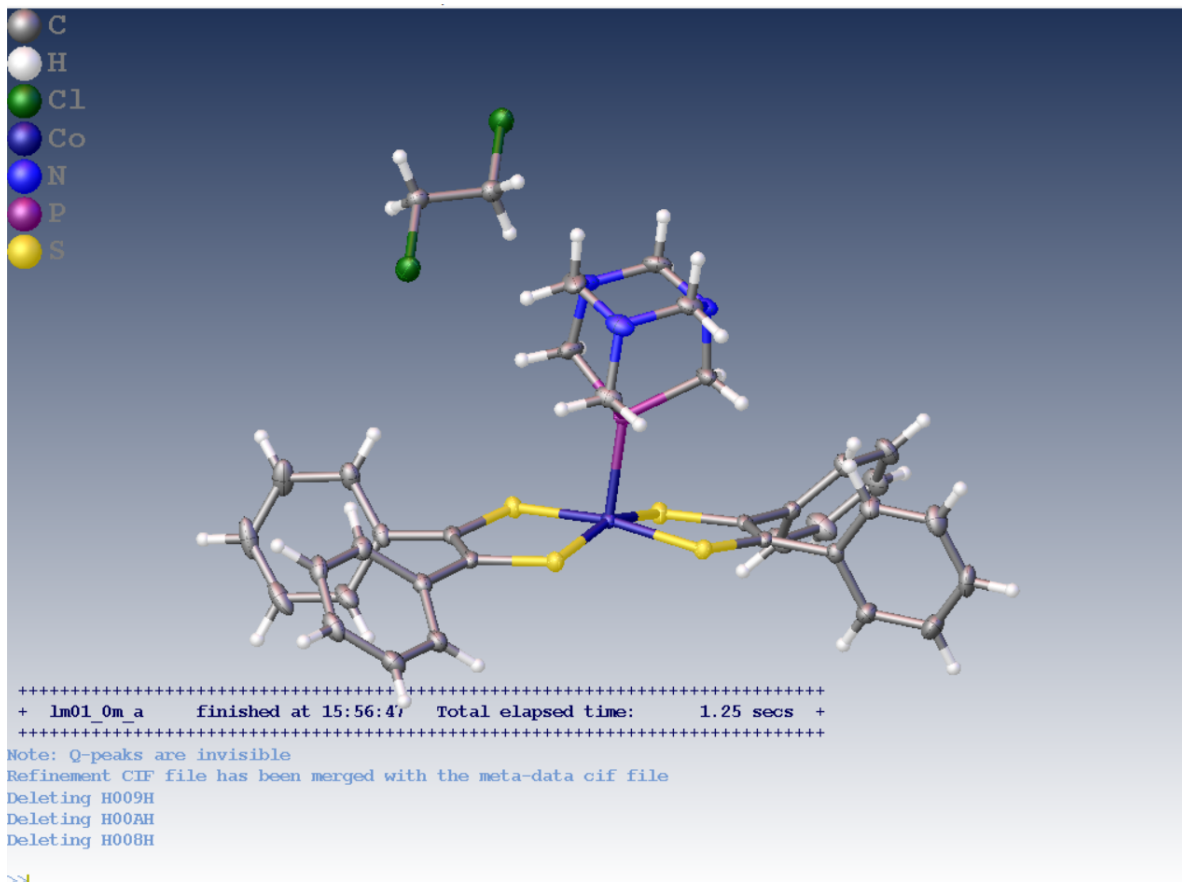
42. Notice how there are a lot of C alerts now we can address those by looking at the hydrogen atoms that are present in the system to make sure that all of them should be there.

43. Looking at the complex with the hydrogens attached we should see the following picture.



44. Make sure that all of the hydrogens in the nitrogen cage-like structure are supposed to be there, everything in this complex is neutrally charged in the adamantane "cage"

45. You should notice that there are 3 extra hydrogen atoms that will need to be deleted, after doing so your molecule should look like the following.






46. Now that we have removed those hydrogen atoms from the structure, we will need to remove them from the formula moiety as well, remember that we have to count for 2 of these complexes in every unit cell.

```
REM 'N00A': 'AtomInfo(hybridisation=3)'  
REM </autosolution>  
CELL 0.71073 9.1089 13.4509 14.8327 96.779 95.097 107.448  
ZERR 2 0.0132 0.0108 0.0119 0.038 0.047 0.054  
LATT 1  
SFAC C H N P S Co Cl  
UNIT 70 68 6 2 8 2 2  
  
L.S. 10  
PLAN -96 0 0  
SIZE 0.138 0.317 0.571  
TEMP -160.15  
BOND $H  
fmap 2  
acta  
OMIT 0 -1 3  
OMIT 1 0 0  
OMIT -1 1 0
```

OK Cancel

47. Once these hydrogen atoms are taken care of, we can run another refinement then another check CIF through IUCr. You should get the following.

← → ↻ https://checkcif.iucr.org/cgi-bin/checkcif_hkl.pl

 Apps
  car stuff
  Account Inquiry

Tmin' 0.591
 Correction method= # Reported T Limits: Tmin=0.661 Tmax=0.746
 AbsCorr = NONE
 Data completeness= 0.988 Theta(max)= 27.163
 R(reflections)= 0.0304(6777) wR2(reflections)= 0.0838(7486)
 S = 1.035 Npar= 406

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
 Click on the hyperlinks for more details of the test.

Alert level A

[ABSTY03_ALERT_1_A](#) The _exptl_absorpt_correction_type has been given as none.
 However a value has been given for _exptl_absorpt_process_details.
 From the CIF: _exptl_absorpt_process_details SADABS-2016/2 (Bruker,201

Alert level B

[PLAT057_ALERT_3_B](#) Correction for Absorption Required RT(exp) ... 1.23 Do !

Alert level C

[ABSTY03_ALERT_1_C](#) The _exptl_absorpt_correction_type has been given as none.
 However values have been given for Tmin and Tmax. Remove these if an absorption correction has not been applied.
 From the CIF: _exptl_absorpt_correction_T_min 0.661
 From the CIF: _exptl_absorpt_correction_T_max 0.746

[PLAT148_ALERT_3_C](#) s.u. on the a - Axis is (Too) Large 0.013 Ang.
[PLAT910_ALERT_3_C](#) Missing # of FCF Reflection(s) Below Theta(Min). 6 Note
[PLAT911_ALERT_3_C](#) Missing FCF Refl Between Thmin & STh/L= 0.600 22 Report

Alert level G

[PLAT042_ALERT_1_G](#) Calc. and Reported MoietyFormula Strings Differ Please Check
[PLAT232_ALERT_2_G](#) Hirshfeld Test Diff (M-X) Co01 --S004 . 6.0 s.u.
[PLAT720_ALERT_4_G](#) Number of Unusual/Non-Standard Labels 77 Note
[PLAT912_ALERT_4_G](#) Missing # of FCF Reflections Above STh/L= 0.600 63 Note
[PLAT913_ALERT_3_G](#) Missing # of Very Strong Reflections in FCF 2 Note
[PLAT933_ALERT_2_G](#) Number of OMIT Records in Embedded .res File ... 8 Note
[PLAT978_ALERT_2_G](#) Number C-C Bonds with Positive Residual Density. 11 Info

1 **ALERT level A** = Most likely a serious problem - resolve or explain
 1 **ALERT level B** = A potentially serious problem, consider carefully
 4 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 7 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 3 ALERT type 2 Indicator that the structure model may be wrong or deficient

48. To fix the remaining A issues (If there are any of the following type) open the CIF, and Search for “_cell_formula_unit_z” and under it put the following table in by typing each component into the correct column. These are experimental parameters collected during the measurement.

- a. If there are many A, B, and C alerts (>5), this could be due to updates made after this tutorial. However, the purpose of this tutorial is to familiarize you with the software and not how to fix complex issues. You may skip these alerts and finish the tutorial using the Finalized_CIF.cif file.

| | |
|-------------------------------|--------|
| _cell_measurement_refl_n_used | 28282 |
| _cell_measurement_temperature | 113(2) |
| _cell_measurement_theta_min | 4.577 |
| _cell_measurement_theta_max | 54.196 |

next search for “_exptl_crystal_density_meas” directly above this type in “_exptl_crystal_description” and “_exptl_crystal_color” then on the right column state ‘prism’ and ‘black’ respectively. The apostrophes are necessary. under “_exptl_crystal_F_000” put in the following table by typing out each component in the appropriate column.

| | |
|---------------------------------|--------------------------------------|
| _exptl_crystal_size_max | 0.571 |
| _exptl_crystal_size_mid | 0.317 |
| _exptl_crystal_size_min | 0.138 |
| _exptl_absorpt_correction_type | ‘multi-scan’ |
| _exptl_absorpt_correction_T_min | 0.6611 |
| _exptl_absorpt_correction_T_max | 0.7455 |
| _exptl_absorpt_process_details | ‘SADABS-2016/2 (Krause et al. 2015)’ |

Then find “_diffrn_reflns_number” and above that insert “_diffrn_measurement_device_type” answered with ‘Bruker D* Venture Kappa’

49. Now validate the last C alerts by writing a validation report into your CIF file. To do this go back to the main check CIF page and make the same selections as in the following image.



A service of the
International Union of Crystallography

checkCIF reports on the consistency and integrity of crystal structure determinations reported in CIF format.

Please upload your CIF using the form below. 

File name:

LM01_0m.cif

Select form of checkCIF report

HTML

PDF

PDF (recommended for CIFs that might take a long time to check)

Select validation type

Full validation of CIF and structure factors

Full IUCr publication validation of CIF and structure factors

Validation of CIF only (no structure factors)

Output Validation Response Form

Level A alerts only

Level A and B alerts

Level A, B and C alerts

None

[Information about this version of checkCIF ...](#)

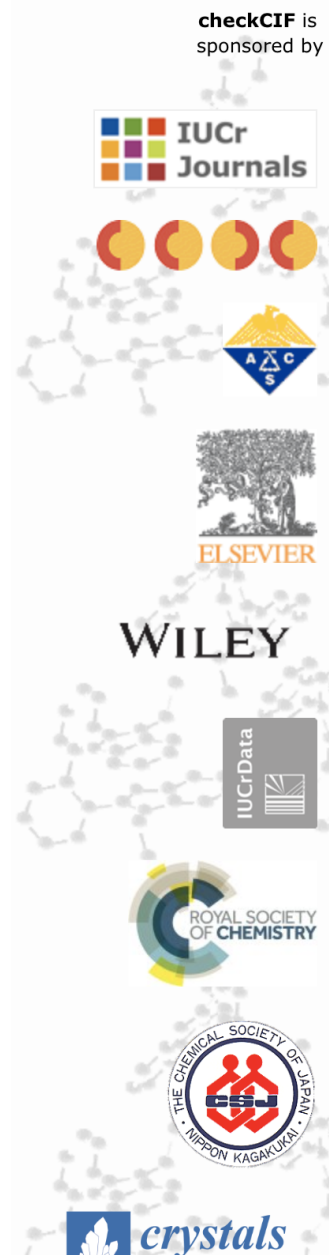
Useful links

Prepublication check for submissions to IUCr journals

[Details of checkCIF/PLATON tests](#)

[CIF dictionary](#)

[Download CIF editor \(pubCIF\) from the IUCr](#)



50. Run the CIF and go to the segment below the alerts and copy the validation report form.

● **Alert level C**

| | | |
|-----------------------------------|--|-----------|
| PLAT910 ALERT 3 C | Missing # of FCF Reflection(s) Below Theta(Min). | 6 Note |
| PLAT911 ALERT 3 C | Missing FCF Refl Between Thmin & STh/L= 0.600 | 21 Report |

● **Alert level G**

| | | |
|-----------------------------------|--|--------------|
| PLAT042 ALERT 1 G | Calc. and Reported MoietyFormula Strings Differ | Please Check |
| PLAT154 ALERT 1 G | The s.u.'s on the Cell Angles are Equal ..(Note) | 0.003 Degree |
| PLAT232 ALERT 2 G | Hirshfeld Test Diff (M-X) Co01 --S004 . | 6.0 s.u. |
| PLAT720 ALERT 4 G | Number of Unusual/Non-Standard Labels | 79 Note |
| PLAT912 ALERT 4 G | Missing # of FCF Reflections Above STh/L= 0.600 | 5 Note |
| PLAT913 ALERT 3 G | Missing # of Very Strong Reflections in FCF | 2 Note |
| PLAT933 ALERT 2 G | Number of OMIT Records in Embedded .res File ... | 7 Note |
| PLAT978 ALERT 2 G | Number C-C Bonds with Positive Residual Density. | 11 Info |

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT910_lm01_0m
;
PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min).          6 Note
RESPONSE: ...
;
_vrf_PLAT911_lm01_0m
;
PROBLEM: Missing FCF Refl Between Thmin & STh/L= 0.600          21 Report
RESPONSE: ...
;
# end Validation Reply Form
```

51. Make sure that all the red text is highlighted and paste it into the CIF file at the very top below “data_lm01_0m”. Make sure there is a blank line before and after the validation form is inserted.

```
data_lm01_0m

# start Validation Reply Form
_vrf_PLAT910_lm01_0m
;
PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min).           6 Note
RESPONSE: ...|
;
_vrf_PLAT911_lm01_0m
;
PROBLEM: Missing FCF Refl. Between Thmin & STh/L=    0.600       21 Report
RESPONSE: ...
;
# end Validation Reply Form

_audit_creation_date          2018-11-17
_audit_creation_method
;
Olex2 1.2
(compiled May 18 2018 14:05:52 for OlexSys, GUI svn.r5506)
;
shelx_SHELXL_version_number  '2018/3'
_audit_contact_author_address ?
_audit_contact_author_email  ?
_audit_contact_author_name   ''
_audit_contact_author_phone  ?
publ_contact_author_id_orcid ?
publ_section_references
;
Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H.
(2009), J. Appl. Cryst. 42, 339-341.
```

52. The response for the missing number of reflections is due to “Omitted reflections from the beam stop.”
53. The response for the missing FCF reflections between Thmin and STh/L is due to “Reflections replaced by fast scans due to overload.”

54. Put the proper quotation in the proper response location and save the CIF.

data_lm01_0m

start Validation Reply Form

vrf_PLAT910_lm01_0m

;

PROBLEM: Missing # of FCF Reflection(s) Below Theta(Min).

6 Note

RESPONSE: Omitted reflections from beamstop.

;

vrf_PLAT911_lm01_0m

;

PROBLEM: Missing FCF Refl Between Thmin & STh/L= 0.600

21 Report

RESPONSE: Reflections replaced by fast scans due to overload.

;

end Validation Reply Form

_audit_creation_date 2018-11-17

_audit_creation_method

;

Olex2 1.2

(compiled May 18 2018 14:05:52 for OlexSys, GUI svn.r5506)

;

shelx_SHELXL_version_number '2018/3'

_audit_contact_author_address ?

_audit_contact_author_email ?

_audit_contact_author_name ''

_audit_contact_author_phone ?

publ_contact_author_id_orcid ?

publ_section_references

;

Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H.
(2009), J. Appl. Cryst. 42, 339-341.

Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.

Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

55. Run the CIF through IUCr and it should look like the following complete CIF.

The following ALERTS were generated. Each ALERT has the format

test-name_ALERT_alert-type_alert-level.

Click on the hyperlinks for more details of the test.

Alert level C

[PLAT910_ALERT_3_C](#) Missing # of FCF Reflection(s) Below Theta(Min). 6 Note

Author Response: Omitted reflections from beamstop.

[PLAT911_ALERT_3_C](#) Missing FCF Refl Between Thmin & STh/L= 0.600 21 Report

Author Response: Reflections replaced by fast scans due to overload.

Alert level G

| | | |
|-----------------------------------|--|--------------|
| PLAT042_ALERT_1_G | Calc. and Reported MoietyFormula Strings Differ | Please Check |
| PLAT154_ALERT_1_G | The s.u.'s on the Cell Angles are Equal ..(Note) | 0.003 Degree |
| PLAT232_ALERT_2_G | Hirshfeld Test Diff (M-X) Co01 --S004 . | 6.0 s.u. |
| PLAT720_ALERT_4_G | Number of Unusual/Non-Standard Labels | 79 Note |
| PLAT912_ALERT_4_G | Missing # of FCF Reflections Above STh/L= 0.600 | 5 Note |
| PLAT913_ALERT_3_G | Missing # of Very Strong Reflections in FCF | 2 Note |
| PLAT933_ALERT_2_G | Number of OMIT Records in Embedded .res File ... | 7 Note |
| PLAT978_ALERT_2_G | Number C-C Bonds with Positive Residual Density. | 11 Info |

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check
