



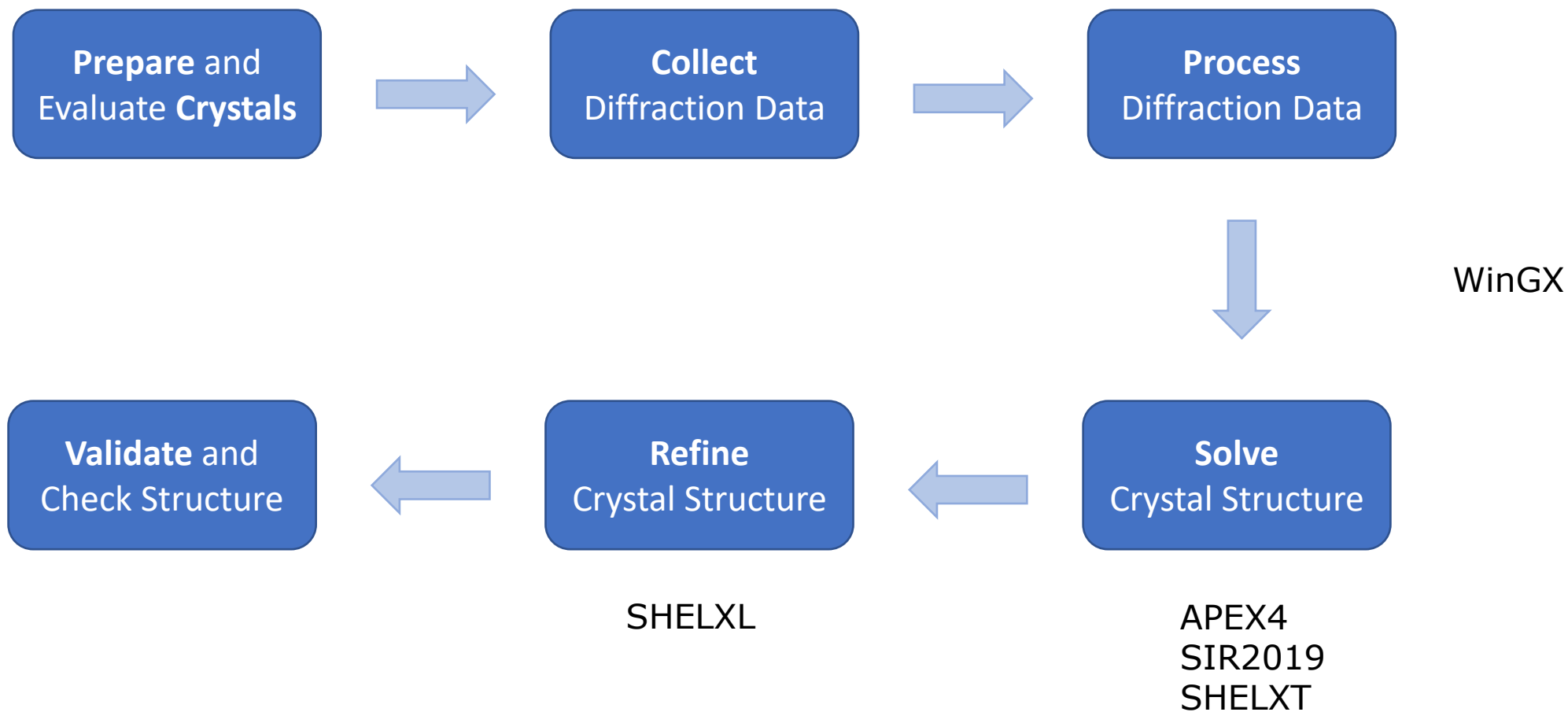
Structure solution and refinement – WinGX

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Crystal Structure Determination and Analysis: Step by Step



Structure solution – APEX4

Find Structure

APEX4 v2021.10-0 - User: (guest) - Sample: Mo_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help Solve Structure

Set Up Evaluate Collect Reduce Data Examine Data Find Structure

Instructions Listing Results Structure View

```

1 TITL mo_Mo_Ylid_0m.in P2(1)2(1)2(1)
2 CBLI 0.71073 5.96610 9.04430 18.39690 90.0000 90.0000 90.0000
3 ZERR 4.00 0.00010 0.00020 0.00030 0.0000 0.0000 0.0000
4 LATT -1
5 SIMM x,-y,-z
6 SIMM -x,y,-z
7 SIMM -x,-y,z
8 SFAC C H O S
9 UNIT 44 40 8 4
10 TEMP 23.000
11 SIZE 0.20 0.20 0.20
12
13 HKLF 4
14 END

```

UTOSTRUCTURE

Solve Structure

Refine Structure

View Structure

Mercury

ORTEP

Report

Reflections 12038 R(int) [%] 5.6 Unique 2442 Observed [%] 72
Rejected 0 R(sigma) [%] 4.65 Observed 1770 Observed [%] 97
in 1.1-1.2Å Shell

Statistics Output Summary

Solve Refine

Input

Reflection File mo_Mo_Ylid_0m.hkl

P4P File mo_Mo_Ylid_0m.p4p

Instruction File mo_Mo_Ylid_0m.ins

Formula C11H10O2S Z 4

Method

Intrinsic Phasing Intrinsic Phasing 1

a= 5.97Å, α=90.00°, V=993Å³
b= 9.04Å, β=90.00°, P2(1)2(1)2(1)
c=18.40Å, γ=90.00°

Patterson Symmetry Original

Chirality Any

Save Patterson Symmetry as Unit Cell

Please integrate after solve if Unit Cell changed.

Direct Direct 1

Dual Space Dual Space 1

Patterson Patterson 1 Expand

Solve Structure

Input files (found in the **/work** folder):

Name.hkl, **Name.p4p** (not mandatory) and **Name.ins**

Available methods:

- 1) **Intrinsic phasing** (XT) – does not require an accurate chemical formula. Heavy atoms are added if missing. Combination
- 2) **Direct methods** (TREF – XS) – the chemical formula should be correct.
- 3) **Dual space** (XM) – requires special settings in the *.ins* file.
- 4) **Patterson** (PATT – XS) – identifies heavy atoms in the asymmetric unit.

Structure solution – APEX4

Find Structure

APEX4 v2021.10-0 - User: (guest) - Sample: Mo_Ylid - Licensed to User at Universidade Nova de Lisboa

Sample Instrument Windows Help

Solve Structure

Set Up Evaluate Collect Reduce Data Examine Data Find Structure

UTOSTRUCTU Solve Structure Refine Structure View Structure Mercury ORTEP Report

Instructions Listing Results Structure View

Statistics Output Summary

[a] P2(1)2(1)2(1) R1 [%] 9.50 Flack X 0.1
alpha 0.002

Solve Refine

Input

Reflection File mo_Mo_Ylid_0m.hkl
P4P File mo_Mo_Ylid_0m.p4p
Instruction File mo_Mo_Ylid_0m.ins
Formula C11H10O2S z 4

Method

Intrinsic Phasing Intrinsic Phasing 1
a= 5.97Å, α=90.00°, V=993Å³
b= 9.04Å, β=90.00°, P2(1)2(1)2(1)
c=18.40Å, γ=90.00°
Patterson Symmetry Original
Chirality Any
Save Patterson Symmetry as Unit Cell
Please integrate after solve if Unit Cell changed.
 Direct Direct 1
 Dual Space Dual Space 1
 Patterson Patterson 1 Expand

Solve Structure

Intrinsic phasing solutions are called **Name_a(/b/c/...).res.**

An individual **Name_a(/b/c/...).hkl** is created for each .res.

Refinement can be easily started on all solutions.

Structure solution – APEX4

Find Structure

Input

Reflection File

P4P File

Instruction File

Formula

Output

Space Group

Formula

Z

R1

wR2

Start

Stop

Input

Reflection File

P4P File

Instruction File

Formula

Output

Space Group

Formula

Z

R1

wR2

Finished!

Start

Stop

Mercury

ORTEP

Report

Legend:

- Q
- S
- O
- H
- C

AUTOSTRUCTURE uses Intrinsic Phasing method for structure solution and performs automatic refinement.

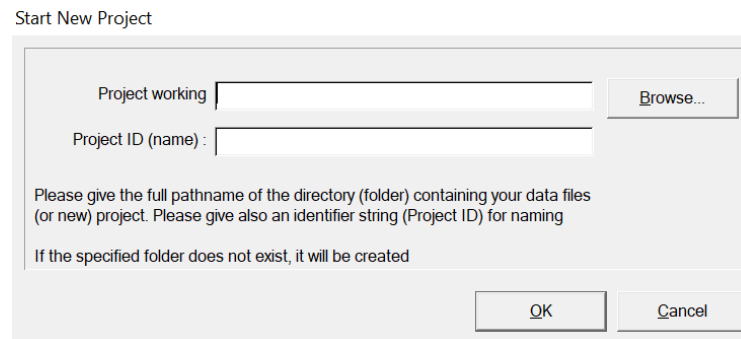
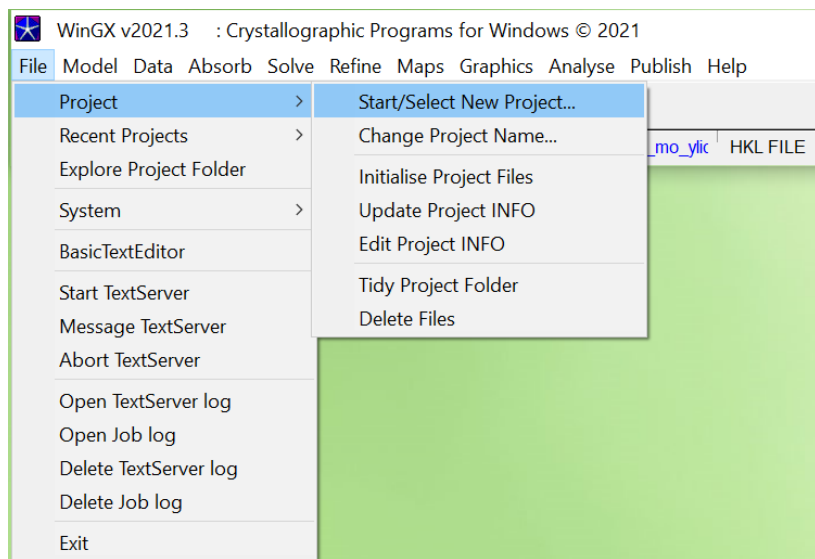
Output area displays the results, including the **space group**, **formula**, **Z**, **R1** and **wR2**.

ALWAYS check the obtained results!!

Structure solution and refinement – WinGX



To start a new structure solution and refinement:



Possible input files:

1) ***name.hkl***, 2) ***name.ins*** and 3) ***name.res***.

1) and 2) generated during data processing in APEX4. 3) is generated after structure solution.

Structure solution and refinement – WinGX

hkl file

h	k	l	I_{hkl}	σ_{hkl}
-1	0	0	0.10	0.30
-1	0	0	0.00	0.10
1	0	0	0.10	0.30
-2	0	0	4074.69	171.98
-2	0	0	3640.14	171.88
2	0	0	3451.65	171.88
-3	0	0	0.80	0.50
-3	0	0	0.80	0.70
-3	0	0	0.30	0.90
3	0	0	0.30	0.70
-4	0	0	441.16	20.70
-4	0	0	437.76	20.50
-4	0	0	440.06	20.90
-5	0	0	0.60	1.40
-5	0	0	2.80	2.10

INS file

```

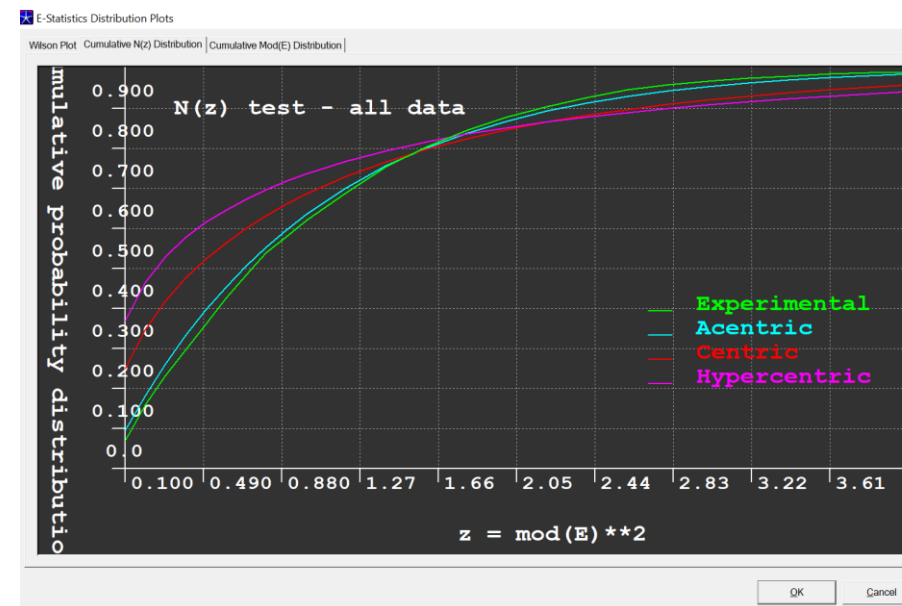
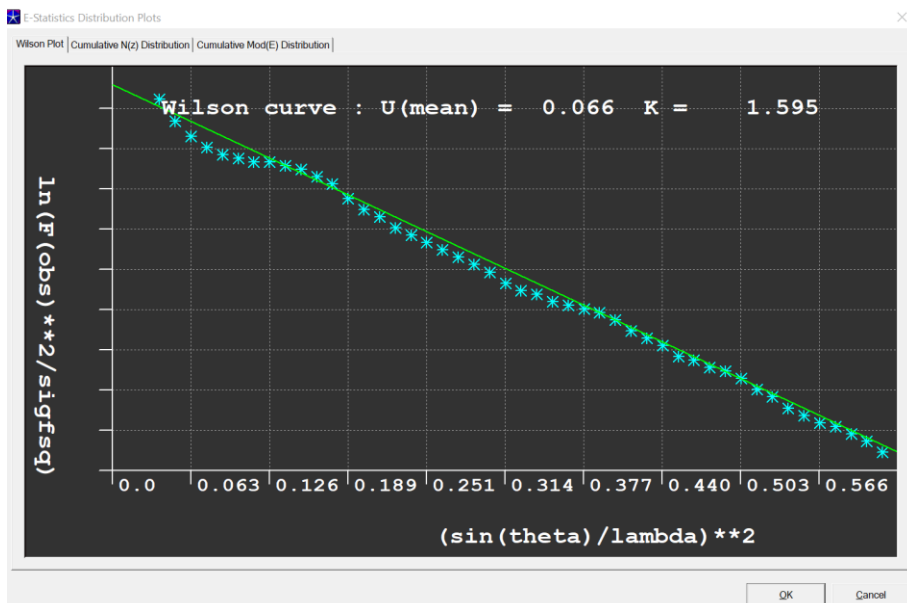
TITL Polymorph1_OrtoR_Pbca in Pbca
CELL 0.71073 9.4493 18.1864 21.2044 90.000 90.000 90.000      Unit cell parameters
ZERR 16.00 0.0014 0.0027 0.0031 0.000 0.000 0.000        Z, standard deviations
LATT 1                                                       Lattice type (P = 1, I = 2, etc.). Negative for non-centrosymmetric
SYMM 0.5-X, -Y, 0.5+Z } space groups
SYMM -X, 0.5+Y, 0.5-Z } symmetry operations for that space group
SYMM 0.5+X, 0.5-Y, -Z }
SFAC C H N           Structure Factors (element symbols) – the first two are C and H, if presente
UNIT 176 160 32      number of atoms of each type in the unit cell
TEMP 0               Temperature
SIZE 0.20 0.30 0.60
TREF                Command for Direct methods using XS – omitted once the structure is solved
HKLF 4
END

```

NOTE: The order of the above seven lines must not be changed, and you can't insert any other command lines in here.

WinGX – statistical analysis

Check E-statistics: Model → PRELIM → E-statistics



		Distribution of E - number of E's .gt. limit																								
E		0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5
No.		4101	4005	3842	3581	3288	2996	2641	2257	1887	1573	1284	981	746	558	409	260	186	127	82	63	45	37	26	15	11
%age		99.0	96.7	92.8	86.5	79.4	72.3	63.8	54.5	45.6	38.0	31.0	23.7	18.0	13.5	9.9	6.3	4.5	3.1	2.0	1.5	1.1	0.9	0.6	0.4	0.3

RMS deviations from theoretical distributions of E's for all data :

Acentric = 0.01117
Centric = 0.09015
Hypercentric = 0.15098

CONCLUSIONS :

The probability that the structure is centrosymmetric is 21.9 %
Sheldrick's |E2 - 1| criterion is 0.668 (Ideal values CENTRIC = 0.968, ACENTRIC = 0.736)

The E-statistics suggest the structure is NON-CENTROSYMMETRIC

IMPORTANT CAVEAT !

The E-statistics are only a GUIDE as to the presence of a centre of symmetry. The calculated probability distributions assume a random distribution of like atoms in the unit cell. Any deviation from this assumption, such as flat ring systems, or the presence of heavy atoms, particularly on special positions, may lead to conclusions which are misleading, or even incorrect !

See R. E. Marsh, Acta Cryst (1995) B51, 897.

WinGX – space group assignment

Space Group: Data → HKL TOOL → SpaceGroup

wgxTextServer - HKL Tool

k+1 odd	10.65	1.21	572
k+1 = 4n	11.88	1.35	285
k+1 / = 4n	10.59	1.20	855

Reflection class	<h h l>	<I/sig(I)>	R(e)	NRef1
No conditions		10.84	1.23	711
h even		9.79	1.11	340
h odd		11.81	1.34	371
l even		11.57	1.31	358
l odd		10.10	1.14	353
2h+1 = 4n		12.30	1.39	182
2h+1 / = 4n		10.34	1.17	529
l = 3n		11.58	1.31	240
l / = 3n		10.46	1.19	471
h+1 even		12.30	1.39	351
h+1 odd		9.42	1.07	360

Reflection class	<h -h l>	<I/sig(I)>	R(e)	NRef1
No conditions		10.38	1.18	591
l even		11.37	1.29	292
l odd		9.40	1.07	299
h+1 = 3n		10.01	1.13	199
h+1 / = 3n		10.56	1.20	392
-h+1 = 3n		10.51	1.19	199
-h+1 / = 3n		10.31	1.17	392

Reflection class	<h h 0>	<I/sig(I)>	R(e)	NRef1
No conditions		17.40	1.97	22
h even		15.60	1.77	8
h odd		18.44	2.09	14

Reflection class	<h h h>	<I/sig(I)>	R(e)	NRef1
No conditions		18.61	2.11	14
h even		16.57	1.88	6
h odd		20.14	2.28	8

AssignSpaceGroup Control Panel

The program has automatically chosen the following settings:
 Lattice type Primitive Laue mmm
 If these are not suitable, please select the correct settings and check the

Unit cell constants		Lattice type	Laue symmetry
a	5.9661	Primitive	-1
b	9.0443	A-Centered	2/m
c	18.396	B-Centered	2/m 1 1
		C-Centered	1 1 2/m
		I-Centered	mmm
		F-Centered	4/m
		R-Centered(ot)	4/mmm

Check/modify absence conditions

OK Cancel

2h+1 / = 4n	10.34	1.17	529
l = 3n	11.58	1.31	240
l / = 3n	10.46	1.19	471
h+1 even	12.30	1.39	351
h+1 odd	9.42	1.07	360

Reflection class	<h -h l>	<I/sig(I)>	R(e)	NRef1
No conditions		10.38	1.18	591
l even		11.37	1.29	292
l odd		9.40	1.07	299
h+1 = 3n		10.01	1.13	199
h+1 / = 3n		10.56	1.20	392
-h+1 = 3n		10.51	1.19	199
-h+1 / = 3n		10.31	1.17	392

Reflection class	<h h 0>	<I/sig(I)>	R(e)	NRef1
No conditions		17.40	1.97	22
h even		15.60	1.77	8
h odd		18.44	2.09	14

Reflection class	<h h h>	<I/sig(I)>	R(e)	NRef1
No conditions		18.61	2.11	14
h even		16.57	1.88	6
h odd		20.14	2.28	8

Extinction symbol : P212121

Candidate space groups :

H-M symbol	#	Centric	Laue class	M	R(int)	N(obs)	CSD	ICSD	CFOM
P 21 21 21	19	chiral	mmm	4	0.057	11909	17453	362	1.262

-->>> Selected space group = P 21 21 21

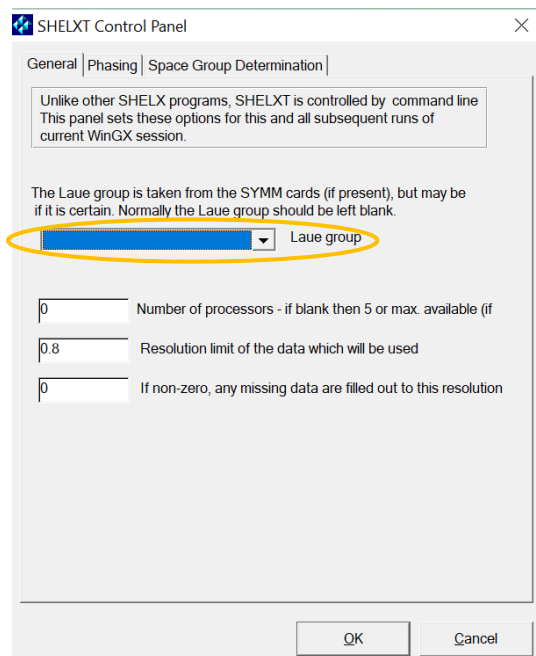
Selected Space Group

The space group P 21 21 21 has been selected as the most
 If this is not correct, please update in the box below.

Space group : P 21 21 21 Browse...

Update Structure Model using this Space Group Close

Structure Solution: Solve → SHELXT → GUI control



```

Mean |E2-1|    0k1 0.863    h01 0.922    hk0 0.926    Rest 0.682
448 Reflections with E < 0.310 employed for R(weak)

40 unique Patterson peaks with heights greater than 30 selected as
superposition vectors

Setup: 0.191 secs

4 threads running in parallel

Try N(iter) CC R(weak) CHEM CFOM best Sig(min) N(P1) Uo1/N
1 100 95.75 0.0794 1.0000 0.8781 0.8781 2.082 57 17.42
2 100 95.70 0.0803 1.0000 0.8767 0.8781 2.032 57 17.42
3 100 95.72 0.0790 0.9811 0.8783 0.8783 2.650 57 17.42
4 100 95.60 0.0798 0.9821 0.8762 0.8783 1.952 58 17.12

4 attempts, solution 3 selected with best CFOM = 0.8783, Alpha0 = 0.553

Structure solution: 0.569 secs

0 Centrosymmetric and 56 non-centrosymmetric space groups evaluated

Space group determination: 2.389 secs

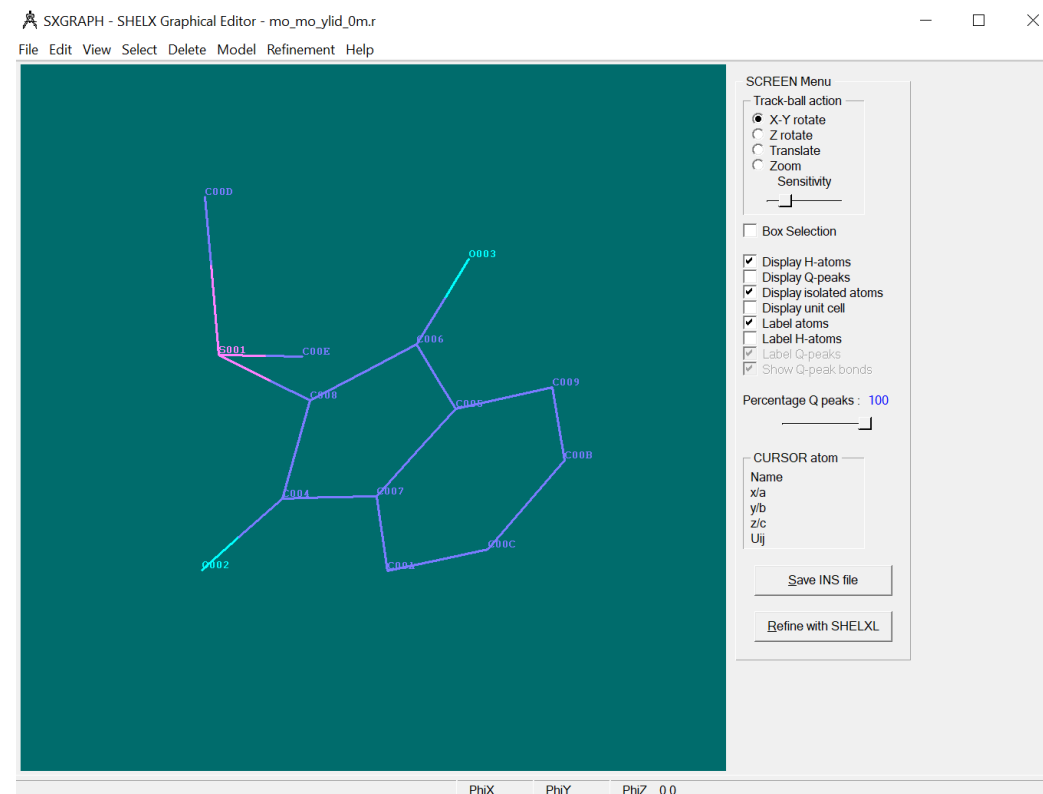
R1 Rweak Alpha SysAbs Orientation Space group Flack_x File Formula
0.094 0.029 0.002 1.06 as input P2(1)2(1)2(1) 0.07 shelxt_a C11 O2 S

Assign elements and isotropic refinement 0.275 secs

Please cite: G.M. Sheldrick (2015) "SHELXT - Integrated space-group
and crystal-structure determination", Acta Cryst., A71, 3-8 (Open
Access) if SHELXT proves useful.

*****
+ SHELXT finished at 00:47:18 Total time: 3.423 secs +
*****

```



Choose Laue group from drop down menu

After space group determination and structure solution, a **name.res** file is created and the GUI SXGRAPH window opens.

Structure solution – WinGX / SIR2019 (alternative to SHELXT)

Structure Solution: Solve → SIR Programs → SIR-2019 → GUI control



automatic *ab initio* and *non ab initio* crystal structure solution

SIR-2019 Control Panel

The WinGX GUI writes a basic control file for SIR-2019 (called SIR2019.sir) using the information given in
If more sophisticated aspects of SIR-2019 are required, then this control file should be manually edited
SIR-2019 can then be run directly using this instruction file from the <Use Command File> menu option for

Replace all fields of ? or zero's with appropriate
NOTE: only one element type and number is

If SIR-2019 fails, examine SIR2019.LST for possible

Unit cell lengths(Å)	Cell angles(deg)
a: 5.9661	alpha: 90
b: 9.0443	beta: 90
c: 18.3969	gamma: 90

Elements present: C H O S

Number in unit cell: 44 40 8 4

Space group symbol: P212121

Use space group information

VLD phasing DM phasing Patterson phasing

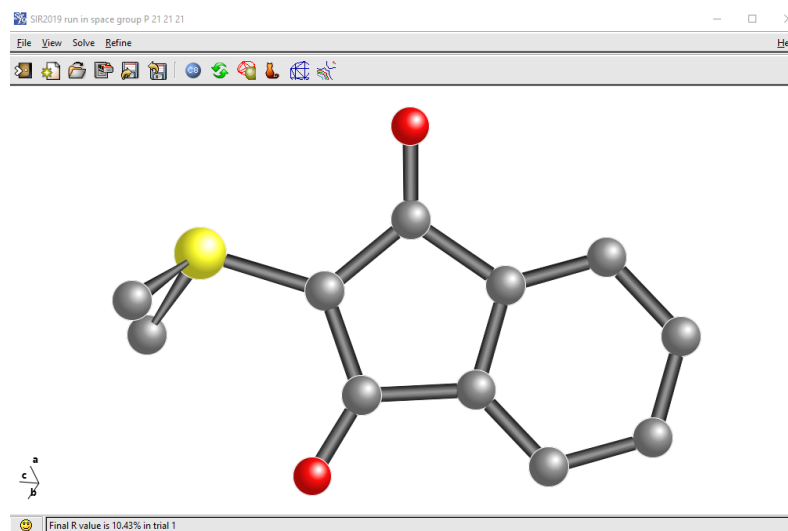
Reflection data

Reflection: mo_Mo_Ylid_0m.hkl F F-sq

Sin(theta)/lambda: 0.5 Use all data No GUI

Resolution (Å): 1.000

OK Cancel



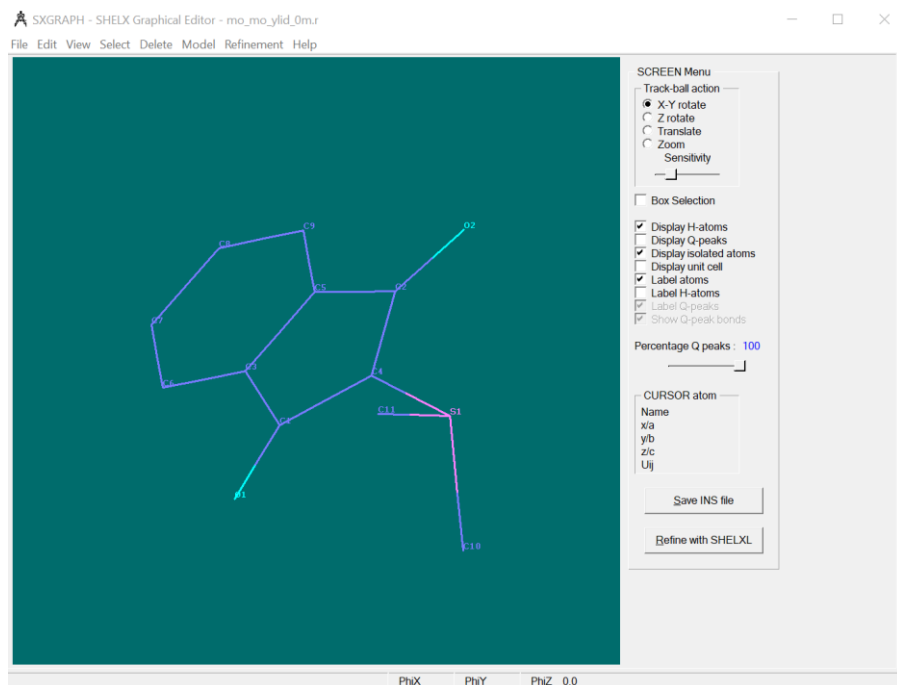
File View Solve Refine

- New
- Load & Go
- Old Project
- Recent Projects
- Load Examples
- Import
- Export
 - Shelx (*.res)
 - Crystals (*.cry)
 - Xtal (*.phs, *.map)
 - CCP4 (*.map)
 - PDB (*.pdb)
 - Moldraw (*.mol)
 - XYZ format (*.xyz)
 - PovRay format (*.pov)
 - C.I.F. format (*.cif)
 - Z-matrix format (*.fhz)
- Save
- Restore
- View output file
- Summary (htm)
- HardCopy
- Exit

WinGX - Structure refinement (SHELXL)

Structure refinement: iterative process

Exiting SIR2019, SXGRAPH  window opens:



↪ Corresponds to ***name.res*** file which resulted from structure solution

RES file

```
TITL SIR2019 run in space group P 21 21 21
CELL 0.71073 5.9661 9.0443 18.3969 90.000 90.000 90.000
ZERR 4 0.0001 0.0002 0.0003 0.000 0.000 0.000
```

```
LATT -1
SYMM 1/2-X, -Y, 1/2+Z
SYMM 1/2+X, 1/2-Y, -Z
SYMM -X, 1/2+Y, 1/2-Z
SFAC C H O S
UNIT 44 40 8 4
```

L.S. 10 Number of least square refinement cycles to run

ACTA Writes a cif file.

BOND \$H Writes bond distances and angles (including for H atoms) to the cif file.

CONF

TEMP 23.0

SIZE 0.20 0.20 0.20 Weighting scheme (default 0.2)

WGHT 0.1 How to write Q peaks. Default is 2.

FMAP 2 Number of Fourier peaks (Q peaks). Default is 20.

PLAN 25 Free Variables.

FVAR 1.0

S1	4	0.30911	0.18147	0.74051	11.00000	0.03598
O1	3	-0.16486	0.30225	0.67647	11.00000	0.05038
O2	3	0.34014	-0.08947	0.62924	11.00000	0.05054
C1	1	-0.06705	0.19484	0.64965	11.00000	0.03398
C2	1	0.18814	0.00031	0.62536	11.00000	0.03523
C3	1	-0.14245	0.11592	0.58259	11.00000	0.03502
C4	1	0.13646	0.12322	0.67275	11.00000	0.03482
C5	1	0.01049	0.00085	0.56751	11.00000	0.03547
C6	1	-0.32380	0.14069	0.53817	11.00000	0.04653
C7	1	-0.34885	0.04940	0.47656	11.00000	0.05297
C8	1	-0.20028	-0.06187	0.46199	11.00000	0.05293
C9	1	-0.01406	-0.08791	0.50787	11.00000	0.04622
C10	1	0.33791	0.37755	0.72867	11.00000	0.04718
C11	1	0.15067	0.17908	0.82280	11.00000	0.05429

Columns:

- 1) Atom name,
- 2) refers to the SFAC line,
- 3-5) Fractional coordinates (x, y, z),
- 6) occupancy of the atom.
- 7) Displacement Parameter U

```
HKLF 4
END
```

WinGX - Structure refinement (SHELXL)

- A **parameter** is *any value that is refined*. For example, it can be:
 - A fractional atom position (x-coordinate of an atom)
 - A thermal parameter of an atom (how much does the atom move)
 - An occupancy factor (how much of an atom is in that position)
 - etc
- **Three parameters** are needed to define an atom **position** (x, y, z)
- **Six parameters** are needed to define thermal libration (defined as a symmetric three by three matrix or tensor)
- Usually, about 10-20 more data (independent diffraction spots) are needed as one would like to refine parameters



WinGX - Structure refinement step-by-step (SHELXL)

Two major tasks are involved: **1)** building of a plausible chemical model by interpretation of electron density and, **2)** the mathematical refinement of structural parameters (given by the model) to optimize the fit of the model to experimental data.

The single steps are normally carried out in the following order:

Step 1: Correction of element assignment and labelling of all atoms except for hydrogen atoms – results from structure solution.

Step 2: Refining anisotropic displacement of non-hydrogen atoms.

Step 3: Identification, placement and refinement of the hydrogen atoms.

Step 4: Adjusting the weighting scheme.

Step 5: Final adjustments and looking at special structural features like crystal packing and hydrogen bonds (if present).

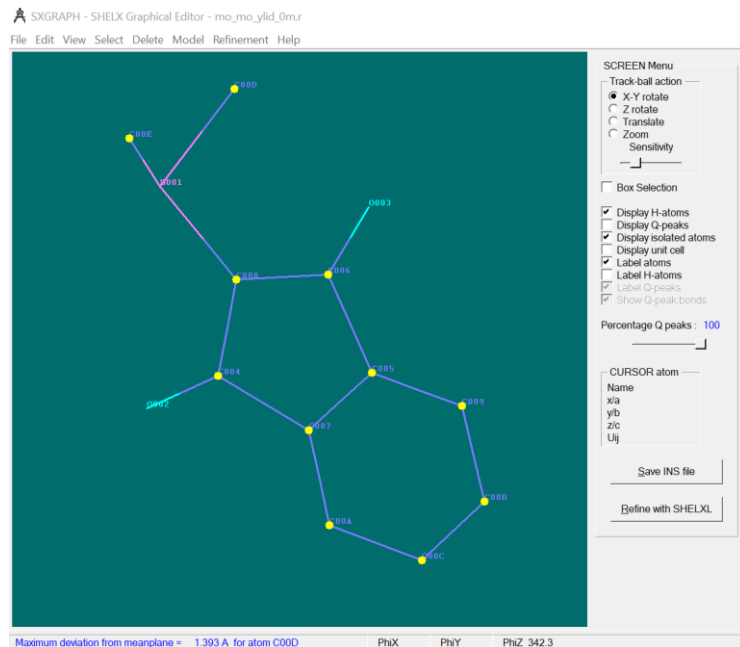
After each single step of model editing (no matter if done graphically or in the text editor) you will use the refinement program SHELXL (**iterative process**). Therefore, at certain stages of the refinement you will have to:

- ✓ **Edit** the instructions file (**INS**) for **SHELXL** and
- ✓ **Analyze** the SHELXL results (**RES**).

WinGX - Structure refinement step-by-step (SHELXL)

Step 1:

- Check element assignment
- Rename atoms accordingly – select atoms in increasing label order (yellow), right-mouse click (a new window opens), introduce new names (if atoms are all of the same type, only the first name is required).
- Rename the remaining atoms.
- Save INS + Refine with SHELXL



Change Group Properties

Properties | Restraints | Geometry

Sequential rename

Old names C006 C008 C004 C007 C00A C00C C00B C009 C005 C00E

New names C1

To auto-renumber, just enter the first new name in list

Parameters

Scattering factor type

AFIX number 0 Set global AFIX

PART number 0 Set global PART

MOLE 0 Set global MOLE

Set Uij's isotropic

Set Uij's anisotropic

Set global Uiso

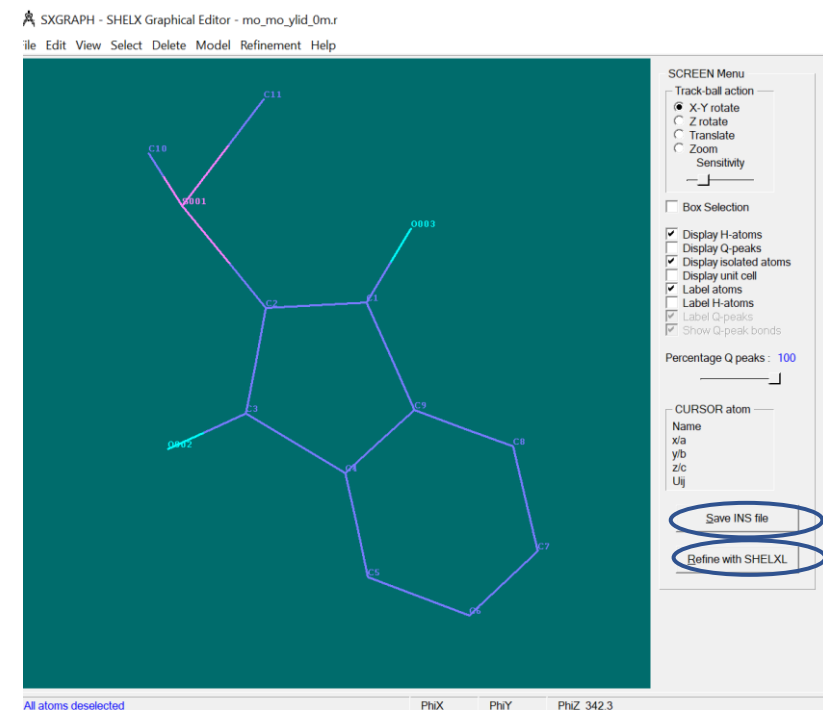
Set global s.o.f.

Uiso 0.05 Fix

s.o.f 1 Fix

Deselect these atoms on OK exit

OK Cancel



Running SHELXL executable : C:\wingx\bin\shelxl.exe

```

+++++
+ SHELXL - CRYSTAL STRUCTURE REFINEMENT - MULTI-CPU VERSION +
+ Copyright(C) George M. Sheldrick 1993-2018 Version 2018/3 +
+ shelx started at 10:49:16 on 11-Jul-2022 +
+++++
  
```

```

Read instructions and data
Data: 4117 unique, 0 suppressed R(int) = 0.0534 R(sigma) = 0.0629
Systematic absence violations: 0 Bad equivalents: 6
wR2 = 0.3004 before cycle 1 for 4117 data and 57 / 57 parameters
GooF = S = 1.120; Restrained GooF = 1.120 for 0 restraints
Mean shift/esd = 0.225 Maximum = 0.853 for y C4 at 10:49:17
Max. shift = 0.006 Å for C6 Max. dU = 0.000 for O1
wR2 = 0.3001 before cycle 2 for 4117 data and 57 / 57 parameters
GooF = S = 1.123; Restrained GooF = 1.123 for 0 restraints
Mean shift/esd = 0.098 Maximum = -0.434 for x C6 at 10:49:17
Max. shift = 0.003 Å for C6 Max. dU = 0.000 for C5
wR2 = 0.3000 before cycle 3 for 4117 data and 57 / 57 parameters
GooF = S = 1.124; Restrained GooF = 1.124 for 0 restraints
Mean shift/esd = 0.016 Maximum = -0.126 for x C6 at 10:49:17
Max. shift = 0.001 Å for C6 Max. dU = 0.000 for C5
wR2 = 0.3000 before cycle 4 for 4117 data and 57 / 57 parameters
GooF = S = 1.124; Restrained GooF = 1.124 for 0 restraints
Mean shift/esd = 0.006 Maximum = -0.046 for x C6 at 10:49:17
Max. shift = 0.000 Å for C6 Max. dU = 0.000 for C7
wR2 = 0.3000 before cycle 5 for 4117 data and 0 / 57 parameters
GooF = S = 1.124; Restrained GooF = 1.124 for 0 restraints
wR2 = 0.3000, GooF = S = 1.124, Restrained GooF = 1.124 for all data
R1 = 0.1005 for 2841 Fo > 4sig(Fo) and 0.1286 for all 4117 data
Flack x = 0.025(223) by classical fit to all intensities
Flack x = 0.055(41) from 920 selected quotients (Parsons' method)
0 atoms may be split and 0 atoms NPD
R1 = 0.1231 for 2417 unique reflections after merging for Fourier
Highest peak 1.71 at 0.7439 0.1708 0.2428 [ 0.46 Å from S1 ]
Deepest hole -0.99 at 0.3247 0.6320 0.2360 [ 0.46 Å from S1 ]
  
```

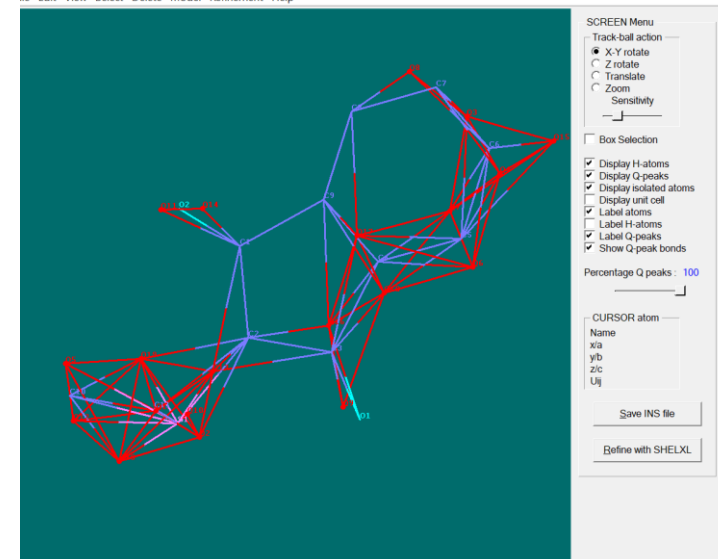
Please cite: G.M. Sheldrick (2015) "Crystal structure refinement with SHELXL", Acta Cryst., C71, 3-8 (Open Access) if SHELXL proves useful.

```

+++++
+ shelx finished at 10:49:17 Total elapsed time: 1.06 secs +
+++++
  
```

SXGRAPH - SHELX Graphical Editor - mo_mo_ylid_0m.r

File Edit View Select Delete Model Refinement Help



- Open SXGRAPH. After the refinement cycle the **atom positions** and thermal parameters of already defined atoms will be **optimized** and positions of still missing atoms will be suggested (new Q peaks – red dots). Decide which might be actual atoms. If some correspond to missing atoms, rename them (as previous renaming atoms).
- Save INS + Refine with SHELXL

- Check values for **R1, wR2, GooF/S, Max. Shift, Maximum, Highest and deepest peaks** (values should decrease)

WinGX - Structure refinement step-by-step (SHELXL)

SXGRAPH - SHELX Graphical Editor - mo_mo_yiid_um.r

le Edit View Select Delete Model Refinement Help

SCREEN Menu
Track-ball action
 X-Y rotate
 Z rotate
 Translate

Change Atom Properties

Properties | Parameters |

Atom Q1 Scattering factor

SHELXL flags
 AFIX # 0 PART # 0 MOLE # 0

Thermal parameters
 ISO Make Uij isotropic
 ANIS Make Uij anisotropic
 ISOR Apply isotropic Uij restraint esd 0.1

Deselect this atom on OK exit

OK Cancel

Save INS file

Refine with SHELXL

Edit View Select Delete Model Refinement Help

SCREEN Menu
Track-ball action
 X-Y rotate
 Z rotate
 Translate

Change Atom Properties

Properties | Parameters |

Atom O2 Scattering factor

SHELXL flags
 AFIX # 0 PART # 0 MOLE # 0

Thermal parameters
 ISO Make Uij isotropic
 ANIS Make Uij anisotropic
 ISOR Apply isotropic Uij restraint esd 0.1

Deselect this atom on OK exit

OK Cancel

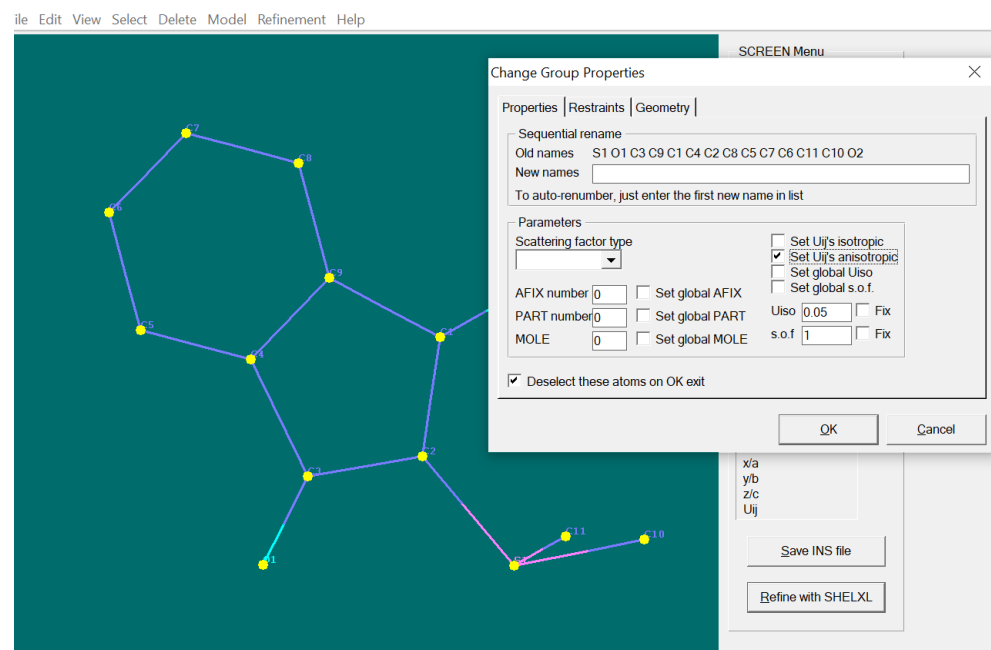
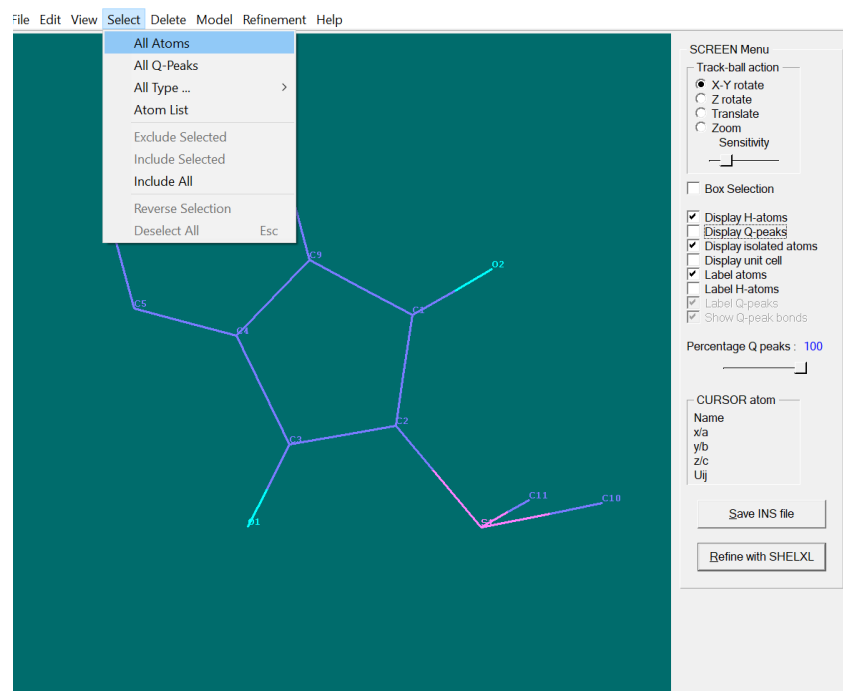
Save INS file

Refine with SHELXL

WinGX - Structure refinement step-by-step (SHELXL)

Step 2:

- Select all non-H atoms (yellow)
- Right-mouse click (a new window opens), Set Uij's anisotropic, OK.
- Save INS + Refine with SHELXL
- Inspect the refinement parameters



RES and/or INS files (after anisotropic refinement)

```
TITL shelxt_a.res in P2(1)2(1)2(1)
shelx.res
created by SHELXL-2018/3 at 11:17:06 on 11-Jul-2022
CELL 0.71073 5.9661 9.0443 18.3969 90.000 90.000 90.000
ZERR 4.00 0.0001 0.0002 0.0003 0.000 0.000 0.000
LATT -1
SYMM 1/2 - X, - Y, 1/2 + Z
SYMM - X, 1/2 + Y, 1/2 - Z
SYMM 1/2 + X, 1/2 - Y, - Z
SFAC C H O S
UNIT 44 40 8 4
MERG 2
FMAP 2
PLAN 20
SIZE 0.200 0.200 0.200
BOND
LIST 6
L.S. 4
TEMP 23.00
WGHT 0.185200 0.305800
FVAR 0.87046
S1 4 0.190202 0.318503 0.740501 11.00000 0.03712 0.03042 =
0.03735 0.00393 0.00830 0.00486
O1 3 0.156841 0.589202 0.628824 11.00000 0.05580 0.03872 =
0.05067 0.00694 0.00803 0.01464
C3 1 0.310706 0.499299 0.625234 11.00000 0.04115 0.02877 =
0.03122 0.00073 0.00154 -0.00103
C9 1 0.641182 0.385958 0.582571 11.00000 0.03644 0.03540 =
0.03311 -0.00126 0.00290 -0.00777
C1 1 0.566755 0.304868 0.649802 11.00000 0.03371 0.03085 =
0.03530 0.00098 0.00093 -0.00163
C4 1 0.490213 0.497767 0.567648 11.00000 0.04793 0.03028 =
0.02993 -0.00239 0.00437 -0.00610
C2 1 0.364638 0.375544 0.672396 11.00000 0.03535 0.03036 =
0.03430 0.00406 0.00355 0.00010
C8 1 0.824306 0.359215 0.537913 11.00000 0.04314 0.05279 =
0.04265 -0.00282 0.00879 -0.00317
C5 1 0.513948 0.589059 0.507666 11.00000 0.07359 0.04003 =
0.03213 0.00281 0.00637 -0.00422
C7 1 0.850798 0.450376 0.476954 11.00000 0.06390 0.05250 =
0.04622 -0.00670 0.02494 -0.01207
C6 1 0.701591 0.561342 0.462257 11.00000 0.08429 0.04612 =
0.03689 -0.00002 0.01706 -0.01333
C11 1 0.165805 0.122060 0.729145 11.00000 0.04791 0.03110 =
0.06020 -0.00083 0.01013 -0.00666
C10 1 0.350684 0.322267 0.822933 11.00000 0.08324 0.05538 =
0.03491 0.00313 -0.00949 -0.00405
O2 3 0.664765 0.197256 0.676606 11.00000 0.04087 0.04461 =
0.05402 0.01080 0.00104 0.01106
HKLF 4
```

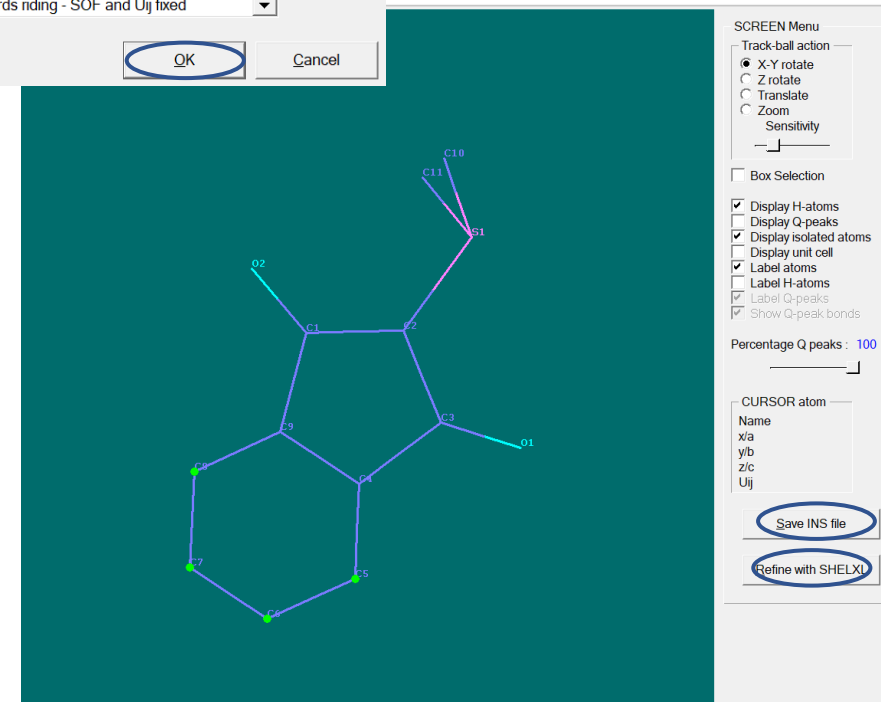
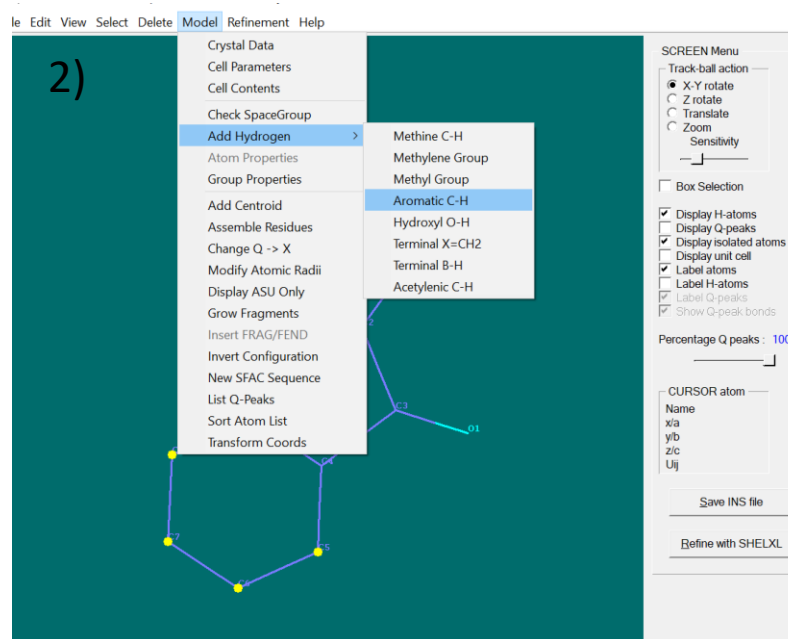
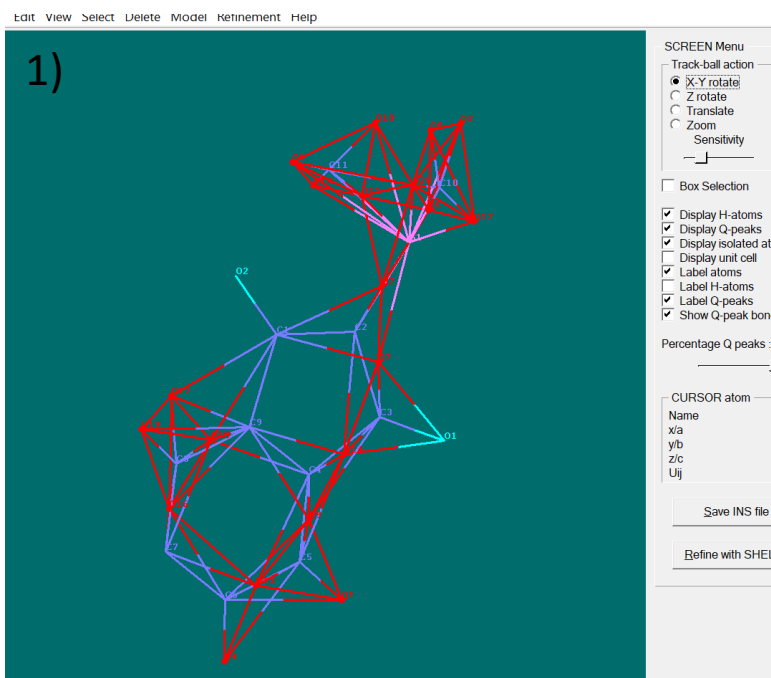
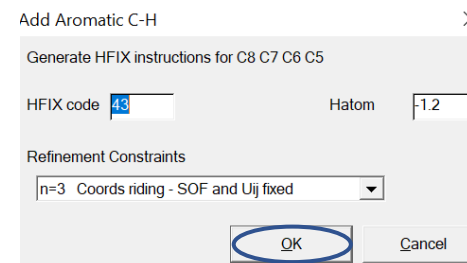
Required for input to shelXle for electron density map analysis

REM name sfac# x y z sof(+10 to fix it) U11 U22 U33 U23 U13 U12 follow

WinGX - Structure refinement step-by-step (SHELXL)

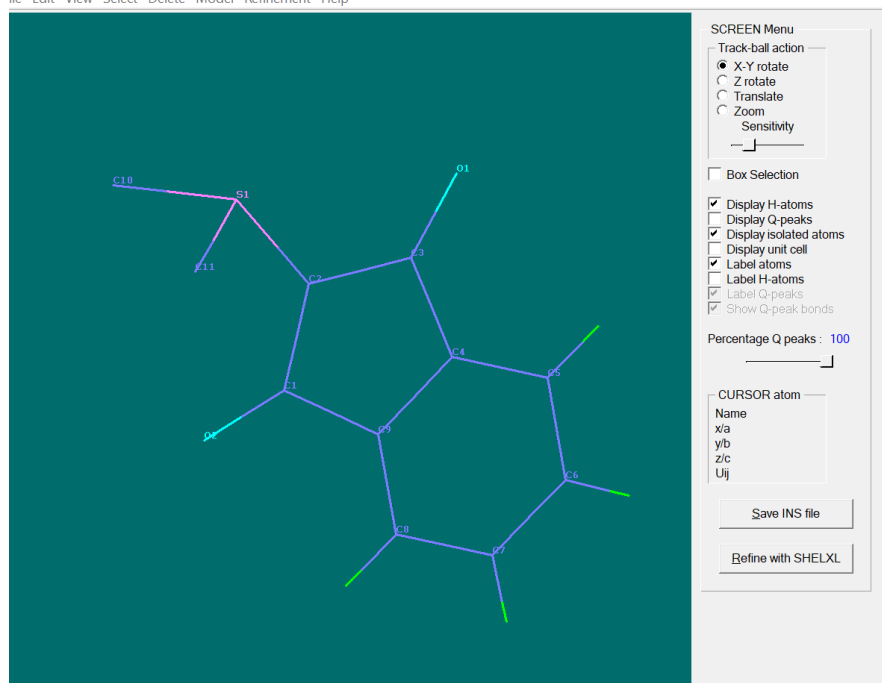
Step 3:

- Depending on the quality of crystal data, it is possible to: **1)** locate H-atoms or **2)** add H-atoms in calculated positions
- Exceptions: N-H atoms and hydride-atoms are ALWAYS located in the electron density map.
- It is preferable to use option **2)**
- Select all C atoms that correspond to the same type of H-atom (yellow)
- Model → Add Hydrogen → select type of H to add.
- New window opens, click OK if no changes are required
- Save INS + Refine with SHELXL and Inspect the refinement parameters



WinGX - Structure refinement step-by-step (SHELXL)

File Edit View Select Delete Model Refinement Help



Hydrogen Atoms: Common HFIX commands

C(sp ³)H	HFIX 13	(saturated tertiary alkanes)
C(sp ²)H	HFIX 43	(alkenes, benzenes, etc)
N(sp ²)H	HFIX 43	(amides, planar secondary amines, usually conjugated to unsaturated atoms)
C(sp ²)H ₂	HFIX 93	(terminal alkenes)
N(sp ²)H ₂	HFIX 93	(planar primary amines, usually conjugated to unsaturated atoms)
C(sp ³)H ₂	HFIX 23	(saturated secondary alkanes)
N(sp ³)H ₂ ⁺	HFIX 23	(protonated secondary amines)
C(sp ³)H ₃	HFIX 33	(methyl group, not rotating)
C(sp ³)H ₃ ⁺	HFIX 137	(methyl group, rotating)
N(sp ³)H ₃ ⁺	HFIX 33	(protonated primary amine, not rotating)
N(sp ³)H ₃ ⁺	HFIX 137	(protonated primary amine, rotating)
O(sp ³)H	HFIX 83	(alcohol, not rotating)
O(sp ³)H	HFIX 147	(alcohol, rotating)
O(sp ³)H	HFIX 148	(alcohol, rotating, O-H distance refined)

Step 3:

- Repeat the process for the remaining types of H-atoms
- The U_{iso} values are usually tied to the atom the H atom is bonded to, being set to 1.2 (for O) or 1.5 (all others) times U_{eq} of the carrier atom.

WinGX - Structure refinement step-by-step (SHELXL)

Step 4:

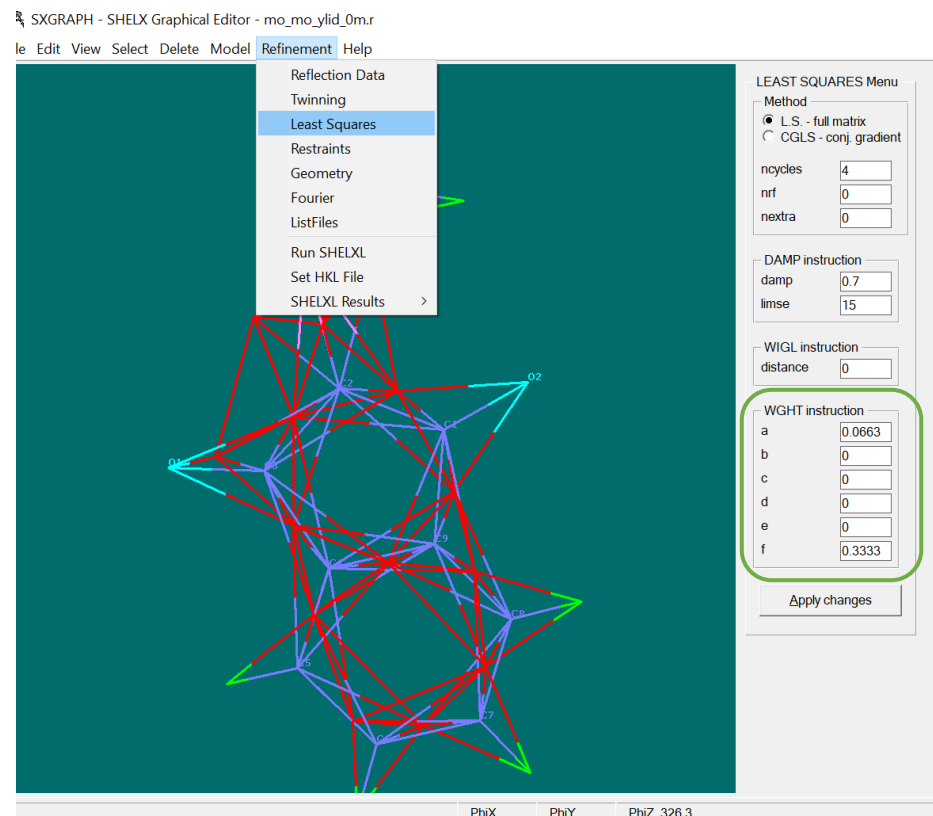
- the internal **weighting parameters a and b** affect (mainly) the wR2 and GooF value. These two parameters are controlled by the WGHT instruction in the INS file. The **recommended values** for WGHT are always **found** in the **latest RES file**, directly **after the END instruction**:

```
REM shelxt_a.res in P2(1)2(1)2(1)
REM wR2 = 0.1284, GooF = S = 0.814, Restrained GooF = 0.814 for all data
REM R1 = 0.0487 for 2841 Fo > 4sig(Fo) and 0.0746 for all 4117 data
REM 127 parameters refined using 0 restraints
```

END

WGHT	0.0663	0.0000
------	--------	--------

- To update the values on WinGX:
Refinement → Least Squares
- Apply changes, SAVE INS and refine with SHELXL



SXGRAPH - SHELX Graphical Editor - mo_mo_ylid_0m.r

le Edit View Select Delete Model Refinement Help

Reflection Data
Twinning
Least Squares
Restraints
Geometry
Fourier
ListFiles

Run SHELXL
Set HKL File
SHELXL Results >

LEAST SQUARES Menu
Method
 L.S. - full matrix
 CGLS - conj. gradient

ncycles 4
nrf 0
nexta 0

DAMP instruction
damp 0.7
limse 15

WIGL instruction
distance 0

WGHT instruction
a 0.0663
b 0
c 0
d 0
e 0
f 0.3333

Apply changes

PhiX PhiY PhiZ 326.3

WinGX - Structure refinement step-by-step (SHELXL)

Step 5:

❖ Final adjustments:

- Add a line **ACTA** (a refinement cycle will now create a cif file)
- Add **BOND \$H** or change BOND to BOND \$H (a refinement cycle will now add X-H bonds and H-X-H angles to the cif file)
- Add a line **CONF** (a refinement cycle will now add dihedral angles to the cif file)
- Add a line **HTAB** (possible hydrogen bonds will be suggested at the end of the next res file)
- **OMIT** reflections (Refinement → Reflection data → Omit instruction): **s** should be 0 or -2. A positive value is not allowed in combination with ACTA. OMIT reflections allows to exclude outliers.

- Update the formula ("**CELL CONTENTS menu**" → Update UNIT card). Continue refinement procedure.

- Add **HTAB** and **EQIV** lines from bottom of res file (check each of the HTAB lines. Some might be meaningless). Continue refinement procedure.



WinGX – Final checklist

- Did you do everything?
- Is the refinement finished? How can you see that?
- Are all non-hydrogen atoms refined anisotropically? How can you check this?
- Does the ORTEP plot look ok? Use Mercury or ShelXle.
- Are all the important entries in the INS file (e.g. TEMP, SIZE, CONF, ACTA, BOND)?
- Has the weighting scheme been adjusted?
- Have the hydrogen bonds been treated correctly, if the structure contains any (HTAB, EQIV)?
- Are the UNIT and Z in ZERR right?
- Are there any warnings or error messages in the .lst file or during refinement?
- Is the wavelength correct?
- Is the structure model chemically reasonable? Do the charges cancel out? Do bond lengths and angles make sense?