Structure solution and refinement – WinGX

Clara Gomes (LAQV-REQUIMTE, FCT-NOVA)

Fátima Minas da Piedade (CQE, FC-ULisboa)

Isabel Cordeiro Santos (C2TN, IST-ULisboa)

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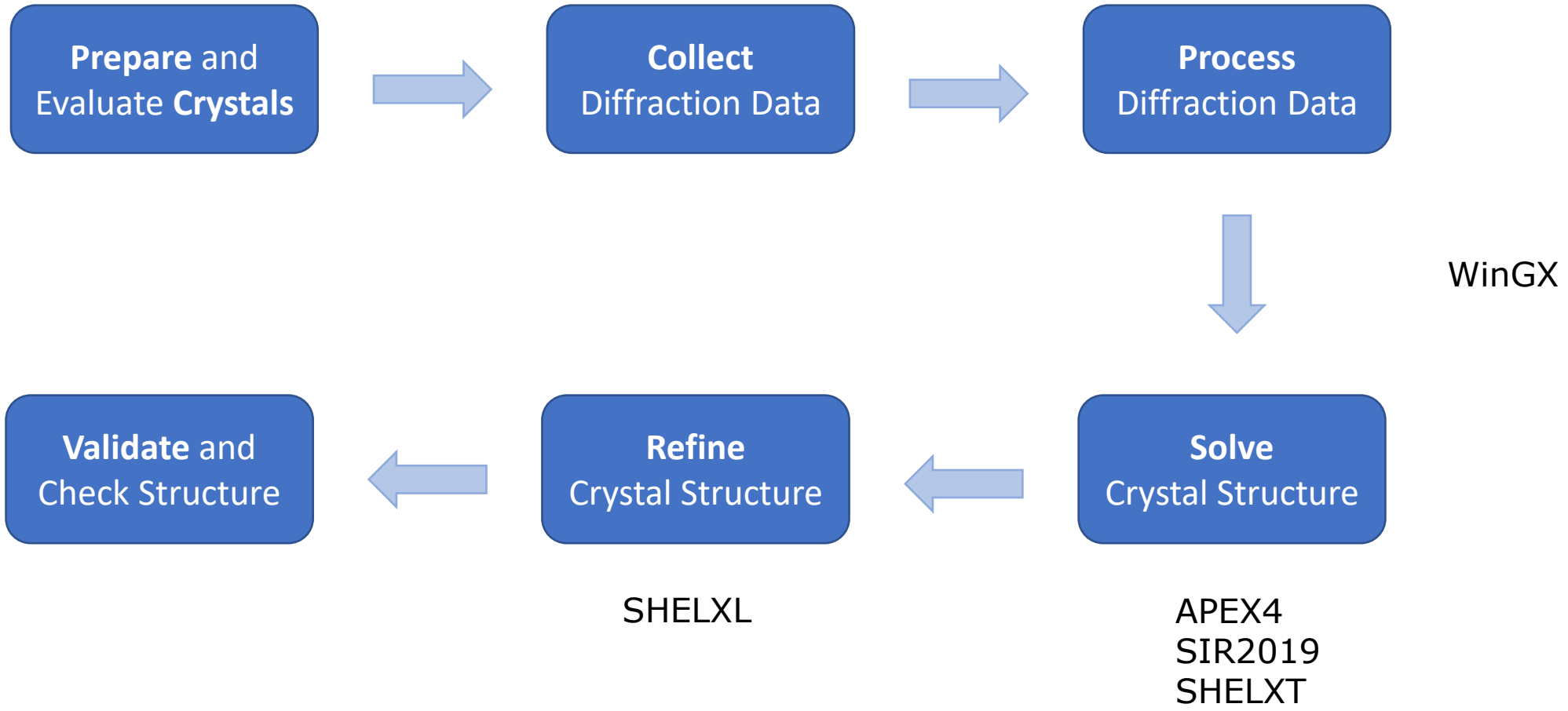
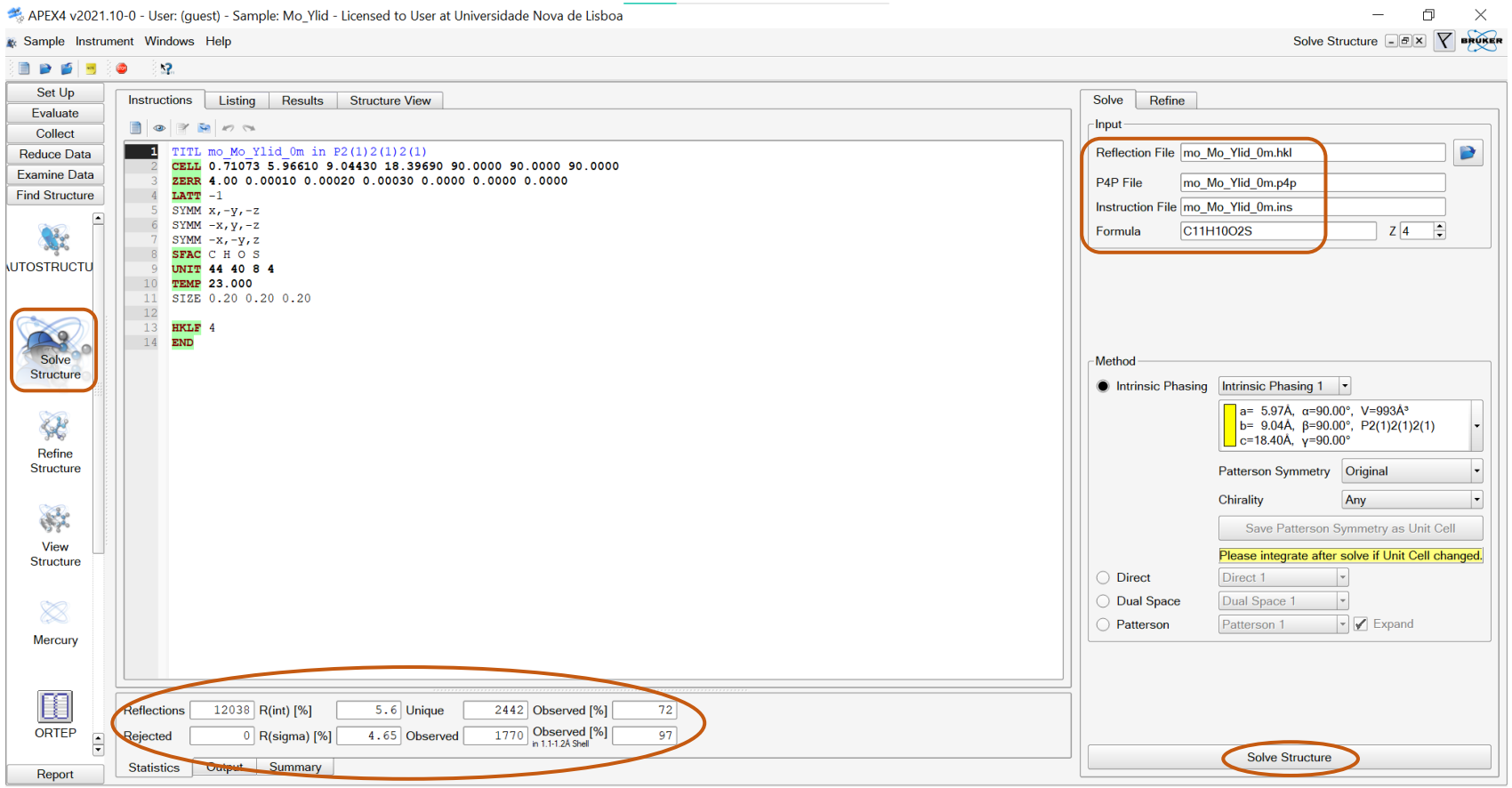
Crystal Structure Determination and Analysis: Step by Step



Structure solution – APEX4

Find Structure



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

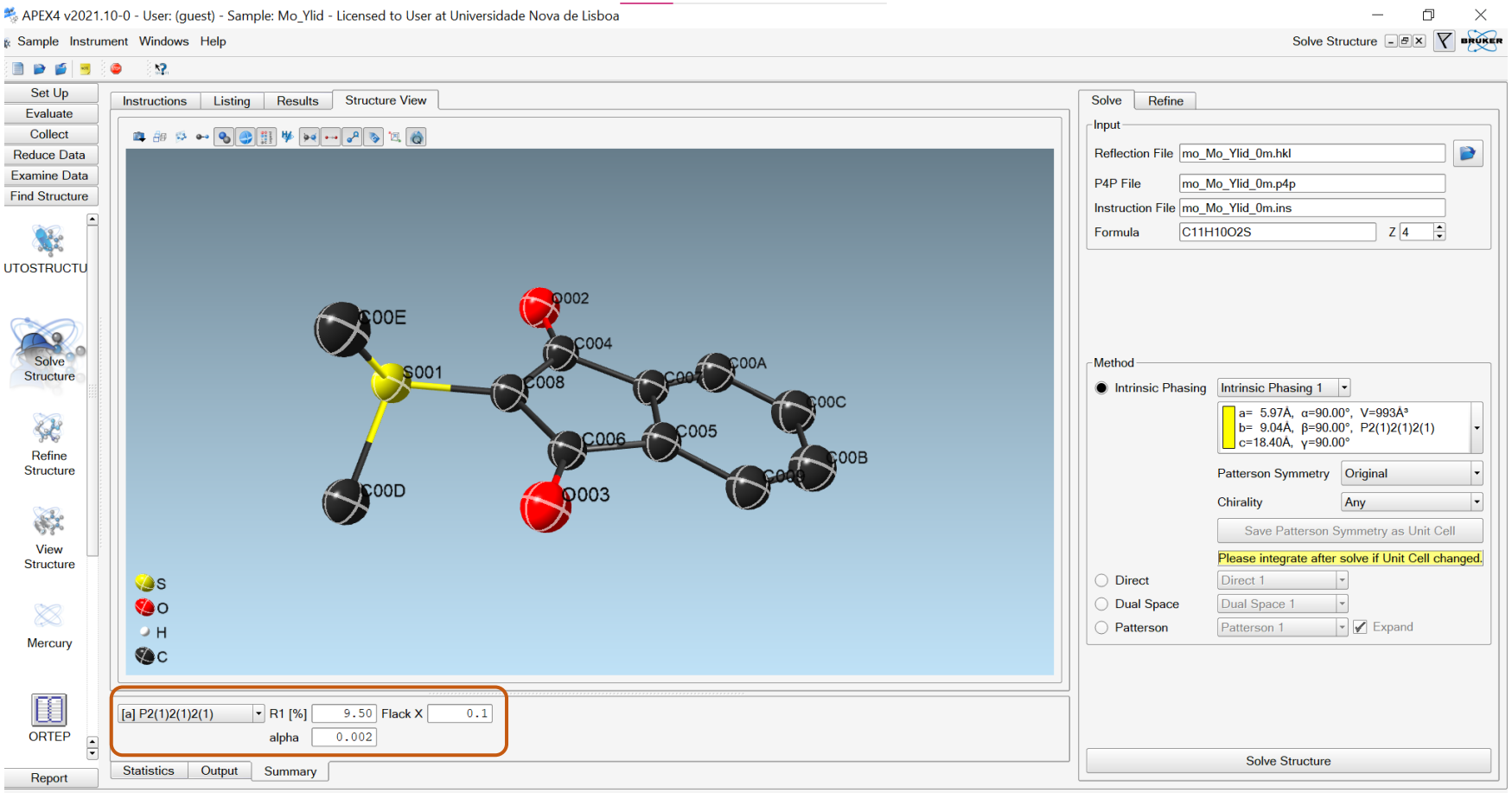
Name.hkl, ***Name.p4p*** (not mandatory) and ***Name.ins***

Available methods:

1) **Intrinsic phasing** (XT) – does not require na 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



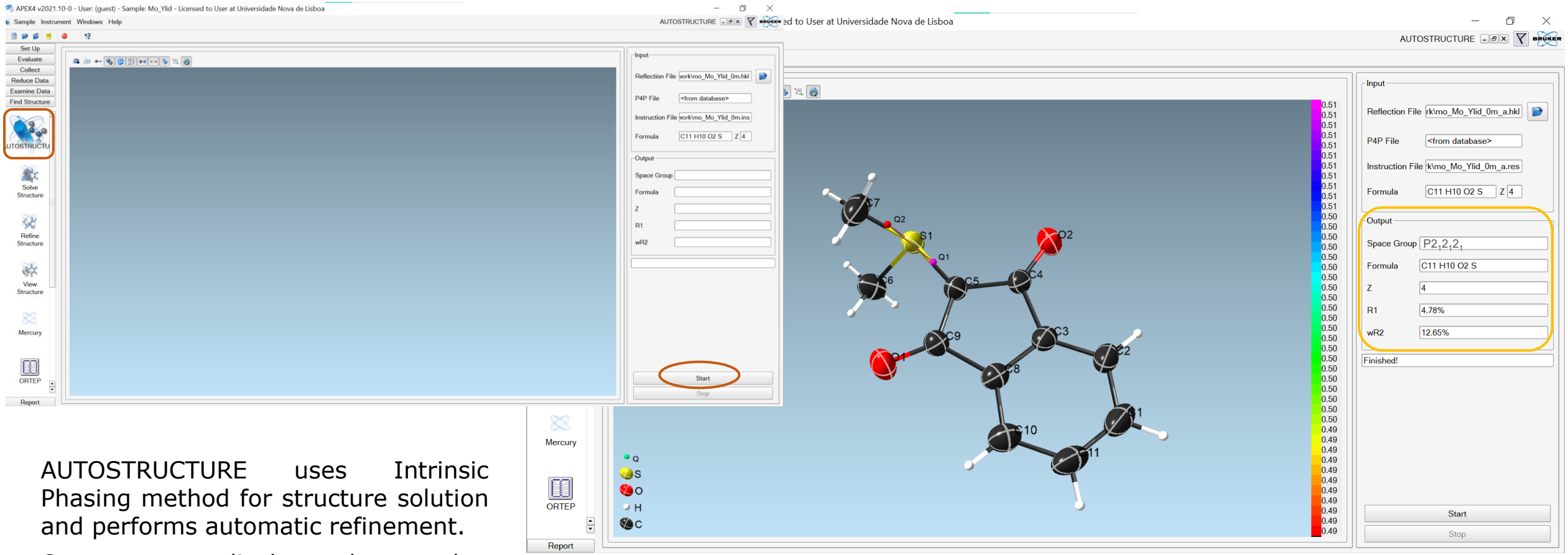
Intrinsic phasing solutions are called ***Name_a(/b/c/…).res***.

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



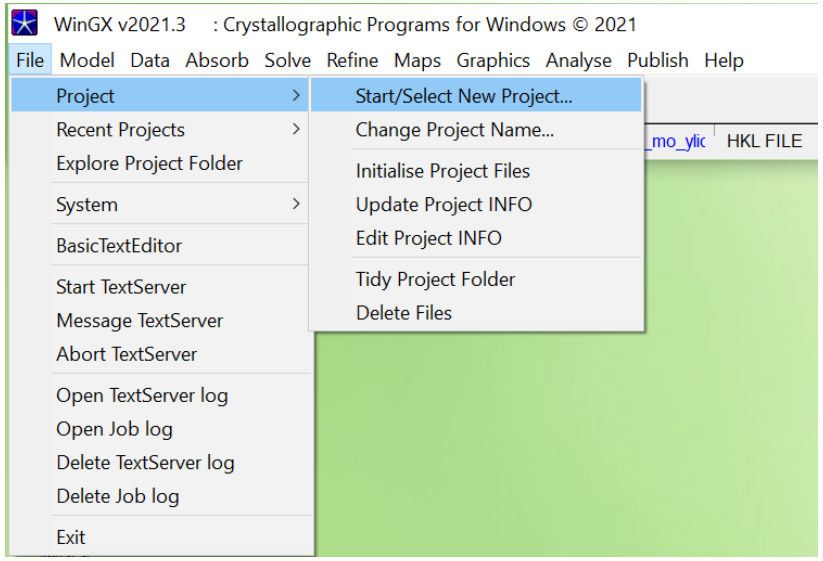
AUTOSTRUCTURE uses Intrinsic Phasing method for structure solution and performs automatic refinement.

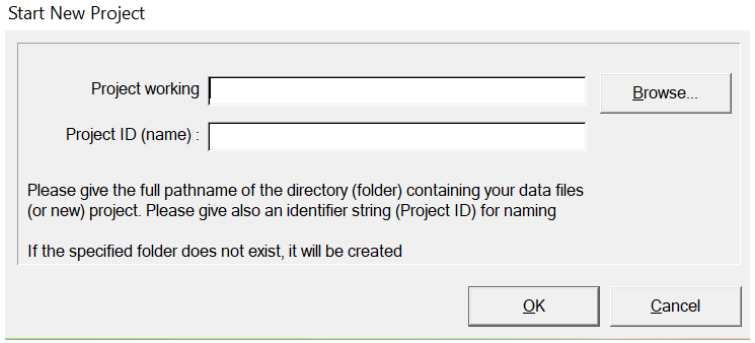
Output area displays the results, including the **space group**, **formula**, **Z**, **R1** and **wR2**.

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
ZERR   16.00   0.0014   0.0027   0.0031   0.000   0.000   0.000
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
UNIT 176 160 32
TEMP 0
SIZE 0.20 0.30 0.60
TREF
HKLF 4
END
```

Unit cell parameters
Z, standard deviations

Lattice type (P = 1, I = 2, etc.). Negative for non-centrosymmetric space groups

symmetry operations for that space group

Structure Factors (element symbols) – the first two are C and H, if presente
number of atoms of each type in the unit cell

Temperature

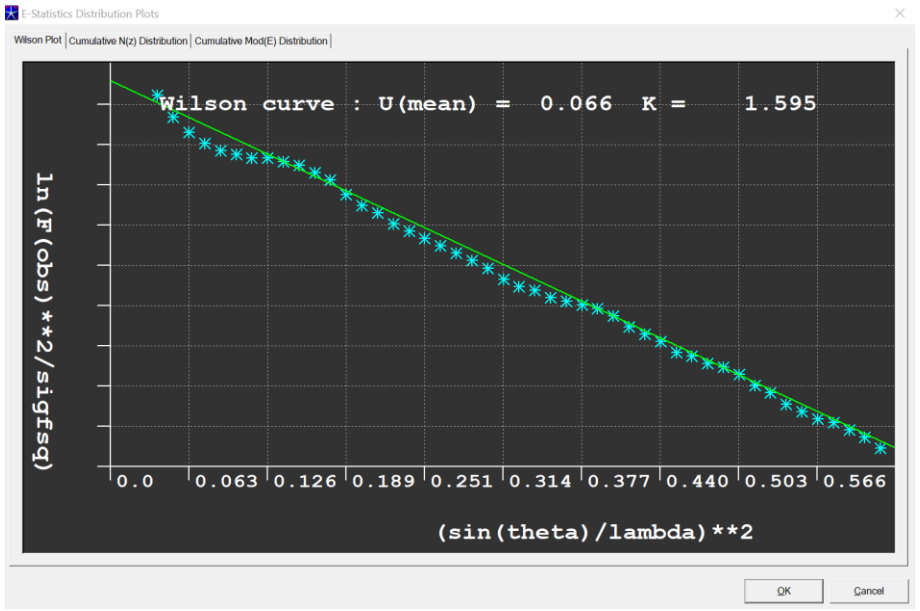
Command for *Direct methods* using XS – omitted once the structure is solved

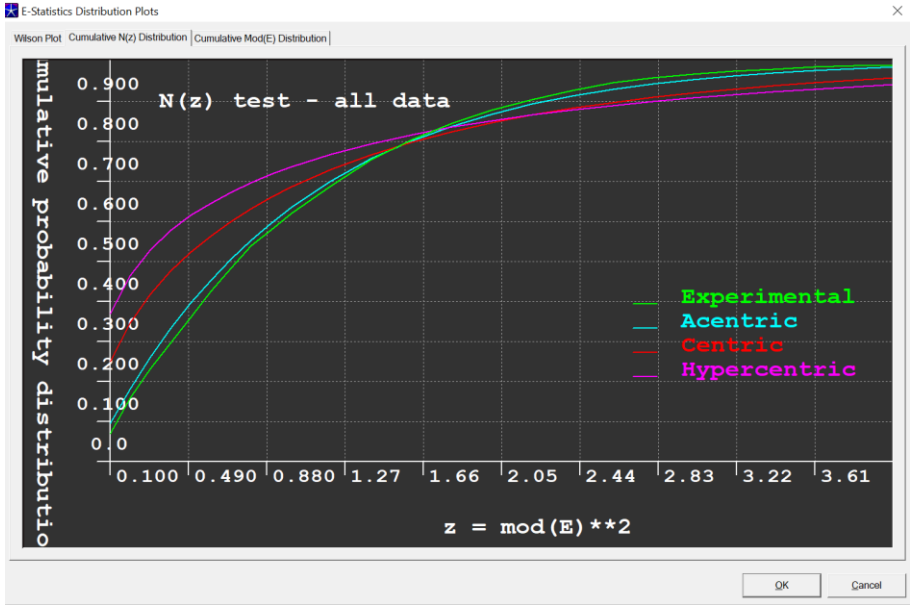
NOTE: The order of the above seven lines must not be changed, and you can't insert any other command lines in here.

SHELXL manual: shelxl_user_guide.pdf (uni-goettingen.de)

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 !

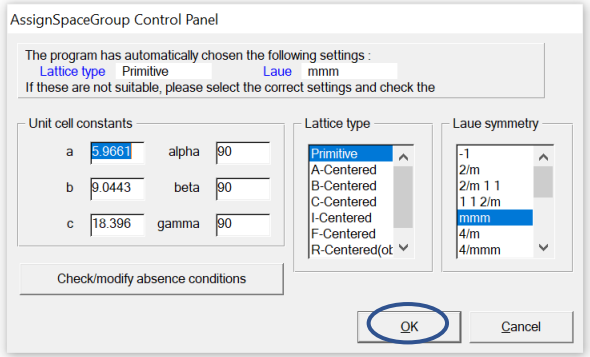
See R. E. Marsh, Acta Cryst (1995) B51, 897.

WinGX – space group assignment

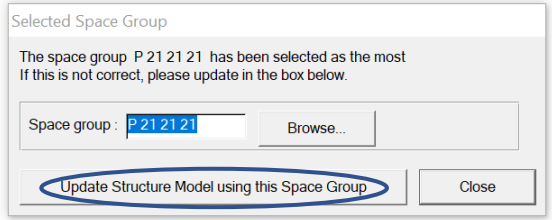
Space Group: Data → HKL TOOL → SpaceGroup

wgxTextServer - HKL Tool

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

Reflection class  <h h l>
                  <I/sig(I)>     R(e)       NRefl
    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+l  =  4n        12.30      1.39         182
    2h+l /=  4n        10.34      1.17         529
    l   =  3n          11.58      1.31         240
    l  /=  3n          10.46      1.19         471
    h+l even           12.30      1.39         351
    h+l odd             9.42      1.07         360

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

Reflection class  <h h 0>
                  <I/sig(I)>     R(e)       NRefl
    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)       NRefl
    No conditions      18.61      2.11          14
    h even             16.57      1.88           6
    h odd              20.14      2.28           8
```

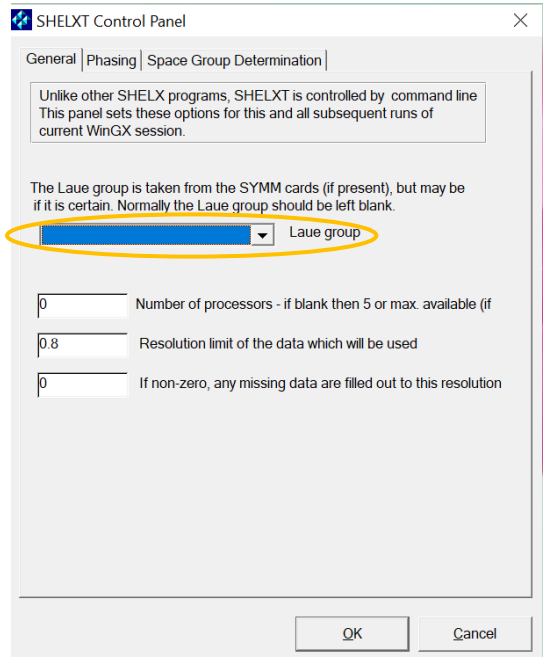



```
    2h+l /=  4n        10.34      1.17         529
    l   =  3n          11.58      1.31         240
    l  /=  3n          10.46      1.19         471
    h+l even           12.30      1.39         351
    h+l odd             9.42      1.07         360

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

Reflection class  <h h 0>
                  <I/sig(I)>     R(e)       NRefl
    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)       NRefl
    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
```



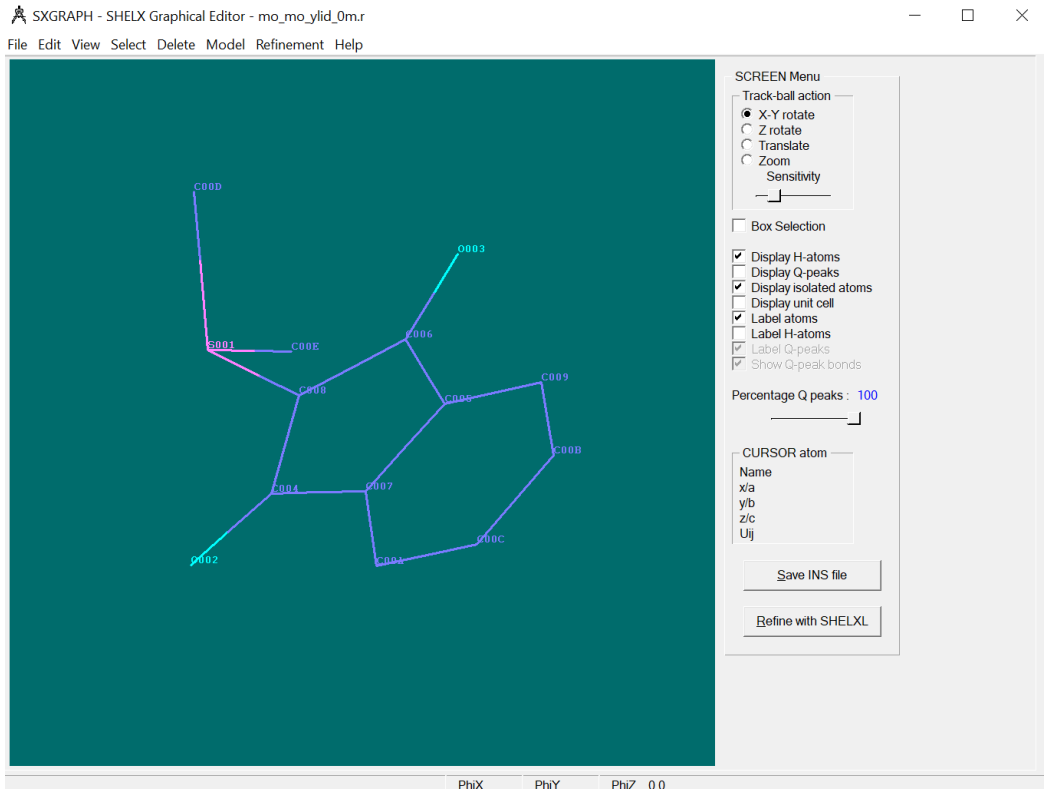
Structure solution – WinGX / SHELXT

Structure Solution: Solve → SHELXT → GUI control



Choose Laue group from drop down menu

```
Mean |E^2-1|    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) Vol/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  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

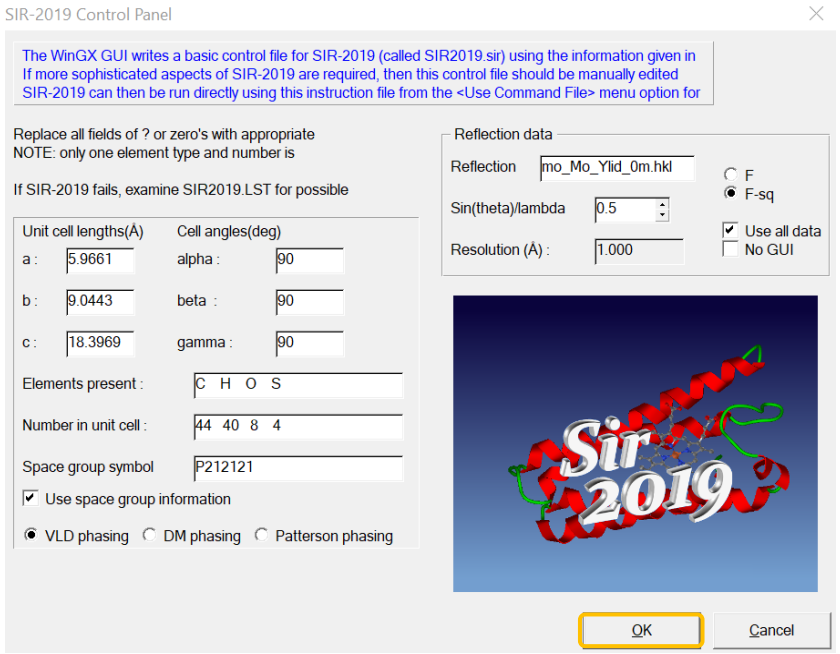


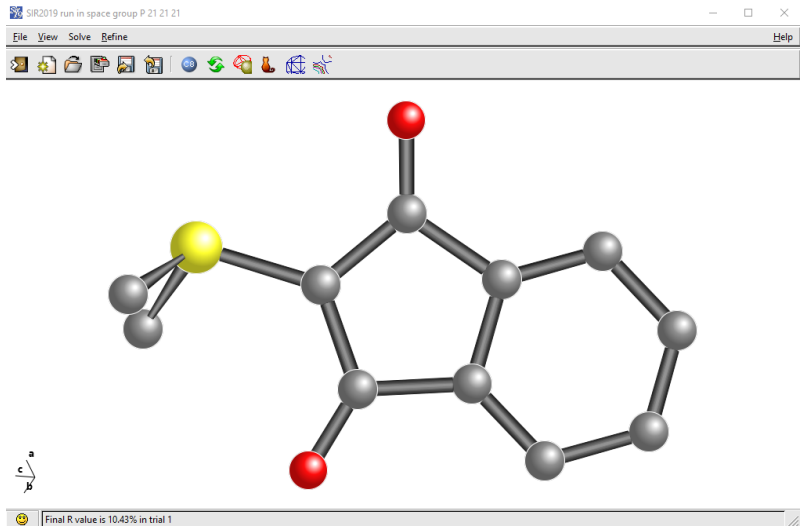
After space group determination and structure solution, a ***name.res*** file is created and the GUI SXGRAPH window opens.

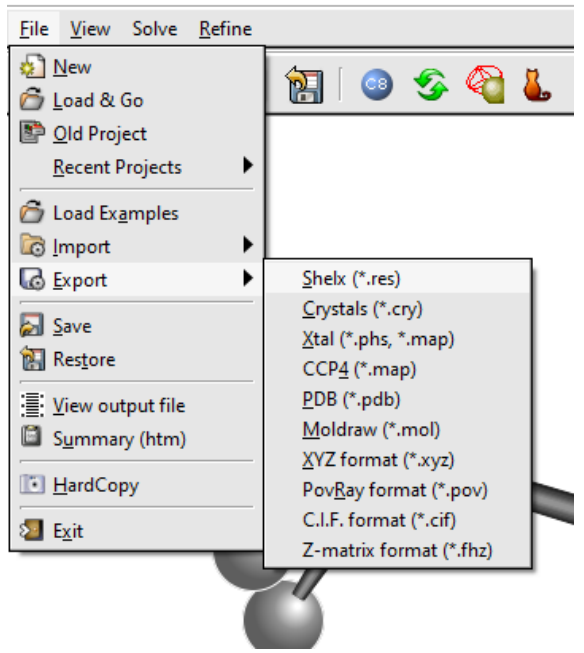
Structure solution – WinGX / SIR2019 (alternative to SHELXT)

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

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



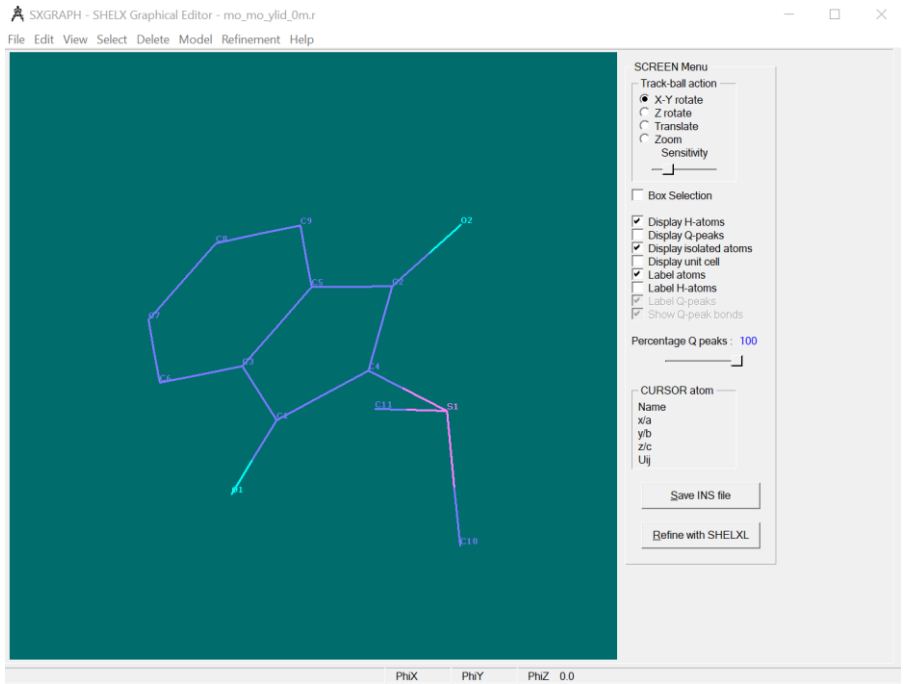




WinGX - Structure refinement (SHELXL)

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
WGHT    0.1            Weighting scheme (default 0.2)
FMAP   2               How to write Q peaks. Default is 2.
PLAN     25            Number of Fourier peaks (Q peaks). Default is 20.
FVAR    1.0            Free Variables.
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

HKLF     4
END
```

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

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).

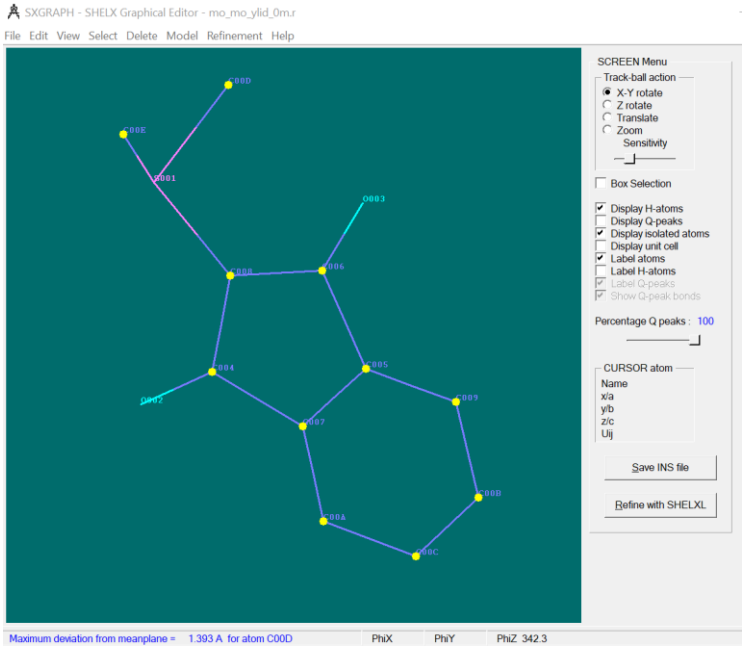
After each single step of model editing (no matter if done graphically or in the text editor) you will use the refinement program SHELXL (**iterative process**). Therefore, at certain stages of the refinement you will have to:

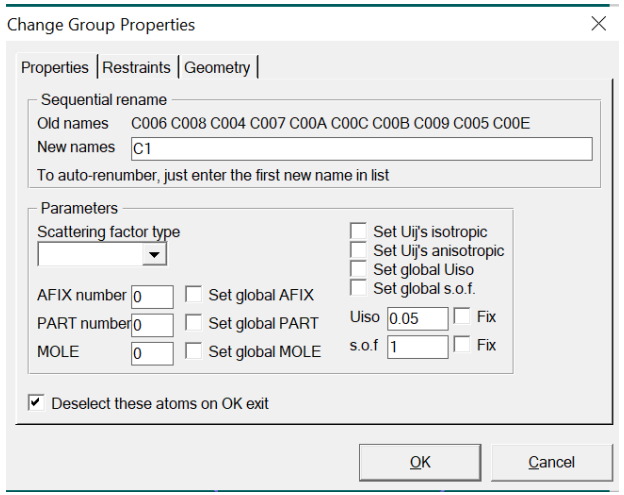
- ✓ **Edit** the instructions file (**INS**) for **SHELXL** and
- ✓ **Analyze** the SHELXL results (**RES**).

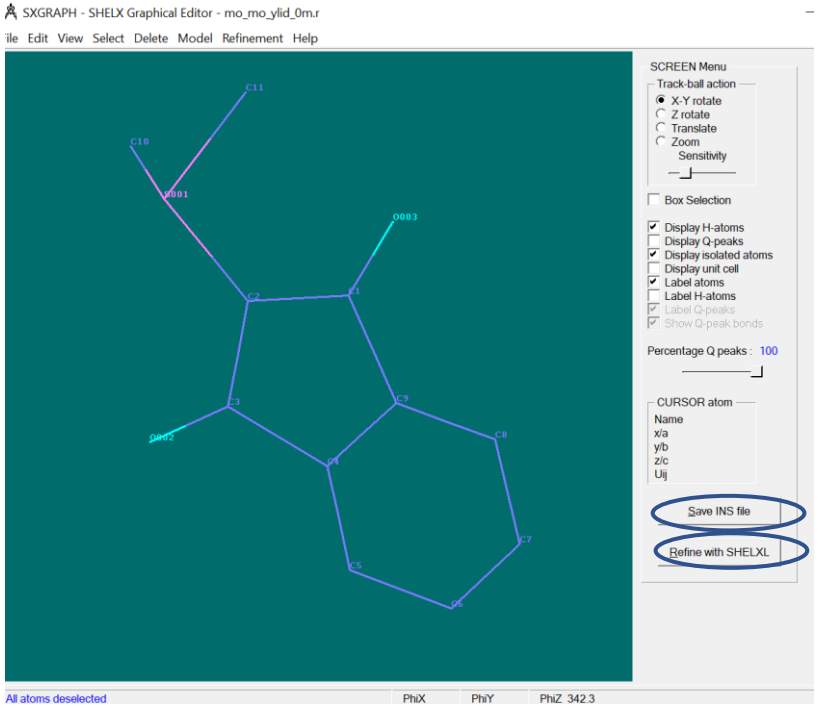
WinGX - Structure refinement step-by-step (SHELXL)

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



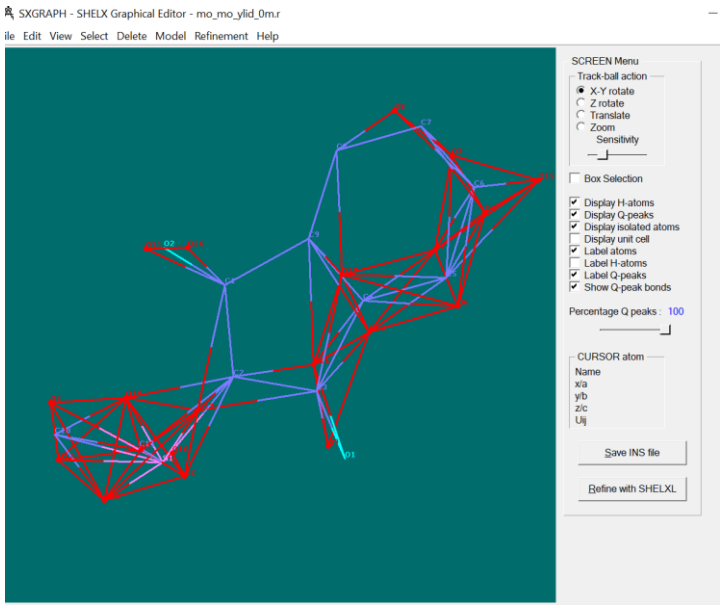




WinGX - Structure refinement step-by-step (SHELXL)

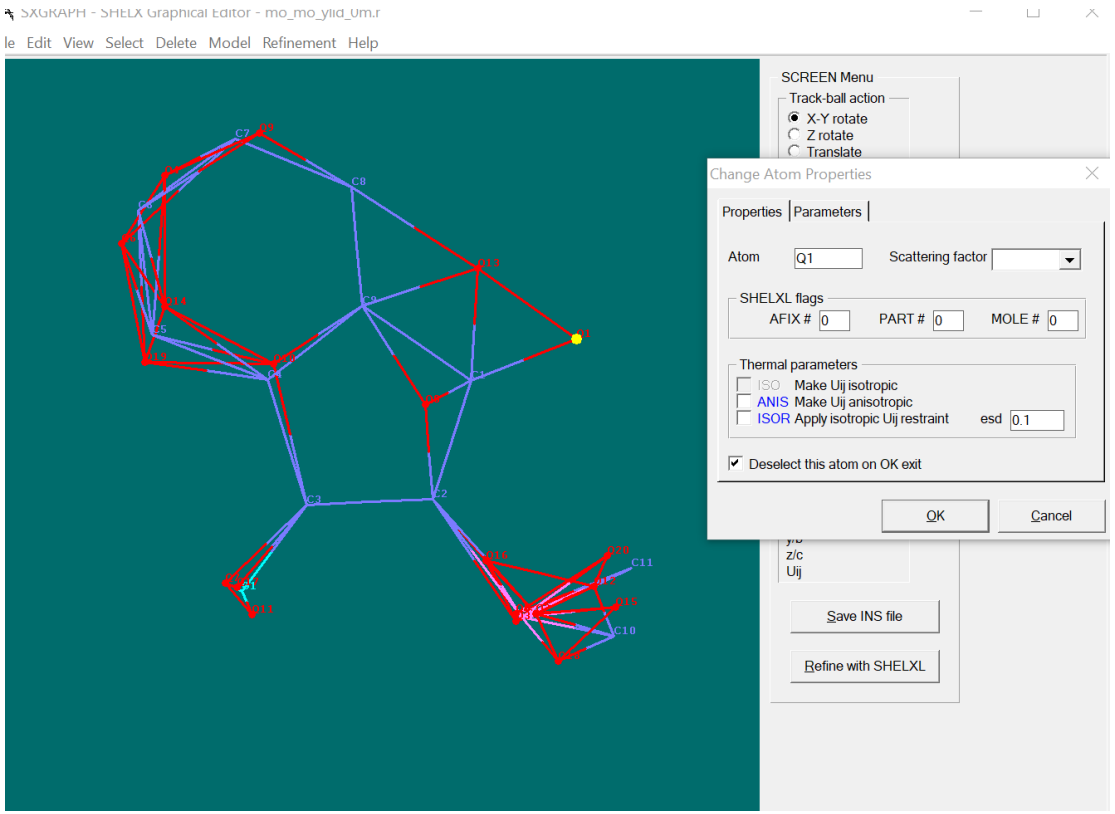
```
Running SHELXL executable : C:\wingx\bin\shelxl.exe


 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
 +  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 A 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 A 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 A 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 A 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 A from S1 ]
Deepest hole   -0.99  at  0.3247  0.6320  0.2360  [  0.46 A 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  +
 ++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

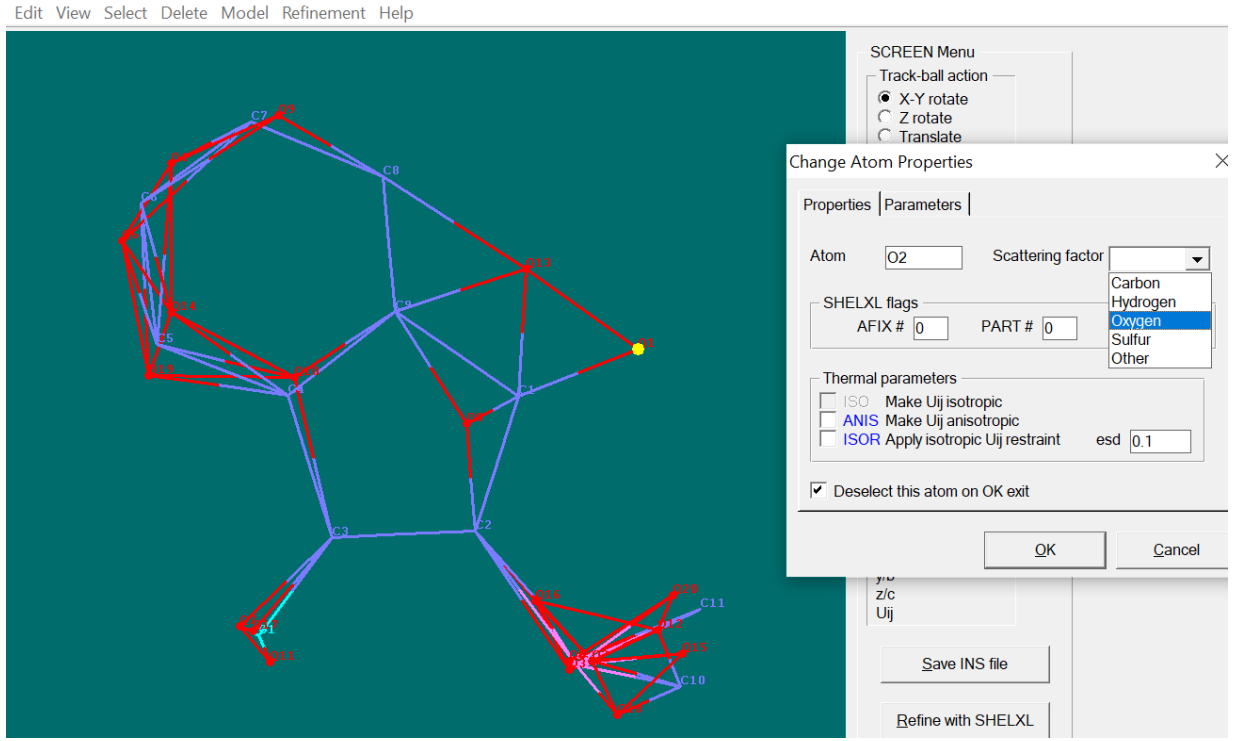
- Check values for **R1**, **wR2**, **GooF/S**, **Max. Shift**, **Maximum**, **Highest and deepest peaks** (values should decrease)



- 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

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

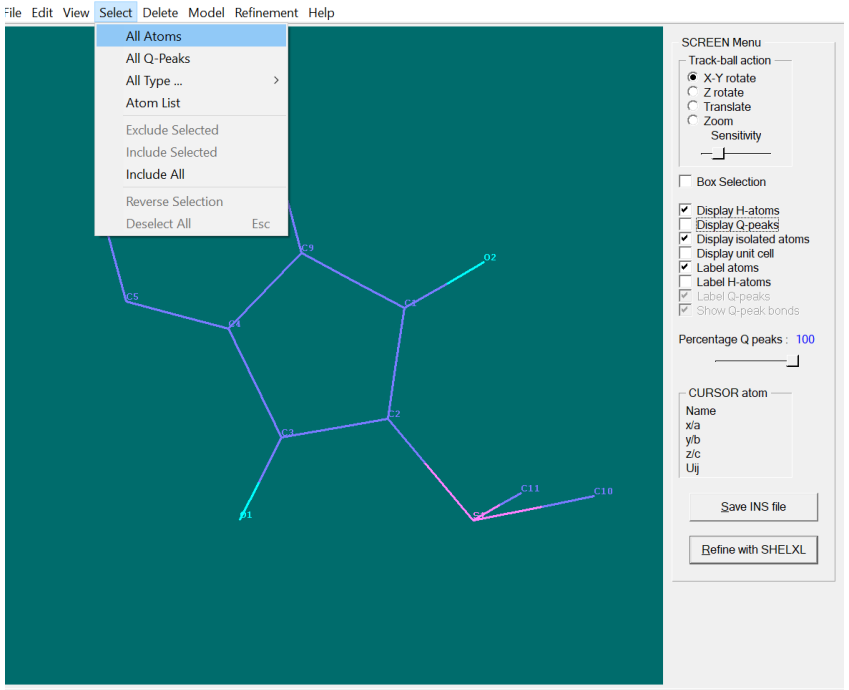


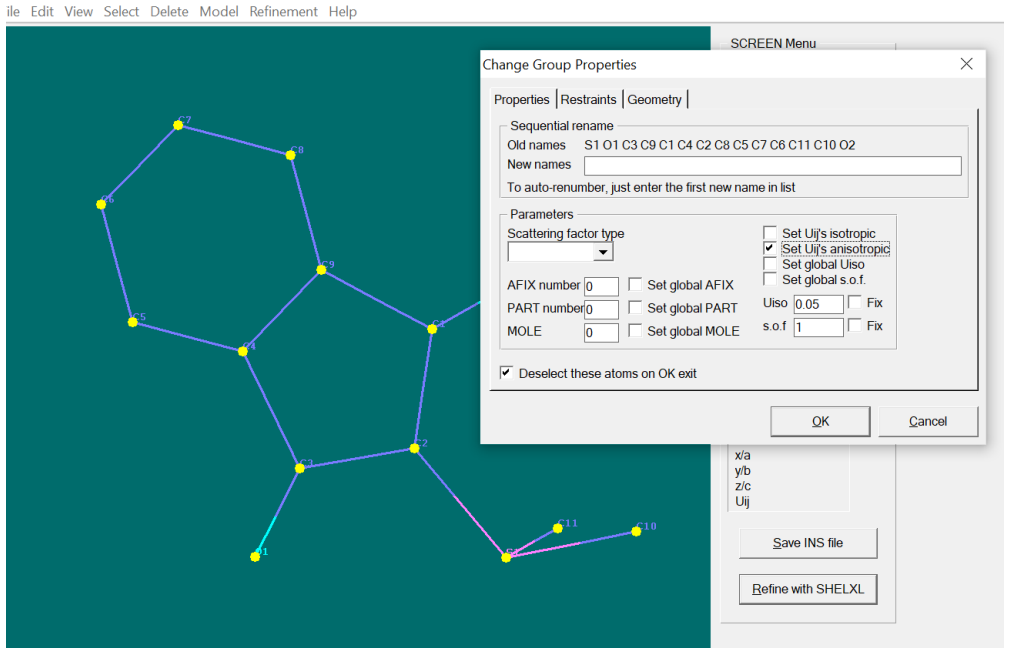


WinGX - Structure refinement step-by-step (SHELXL)

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





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

7th EUROPEAN CRYSTALLOGRAPHIC SCHOOL LISBON | 2022

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
```

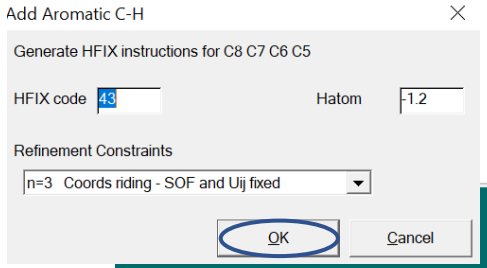
Required for input to shelXle for electron density map analysis

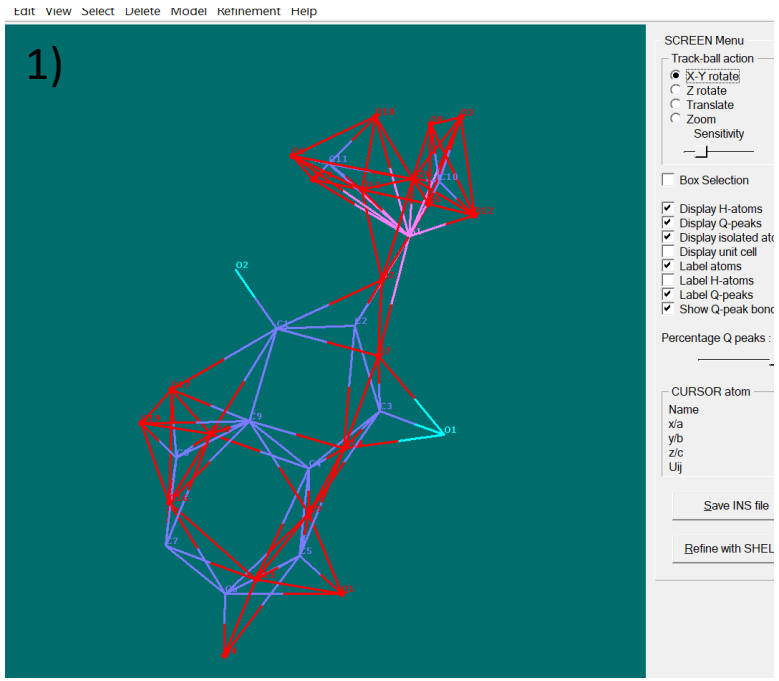
REM name sfac# x y z sof(+10 to fix it) U11 U22 U33 U23 U13 U12 follow

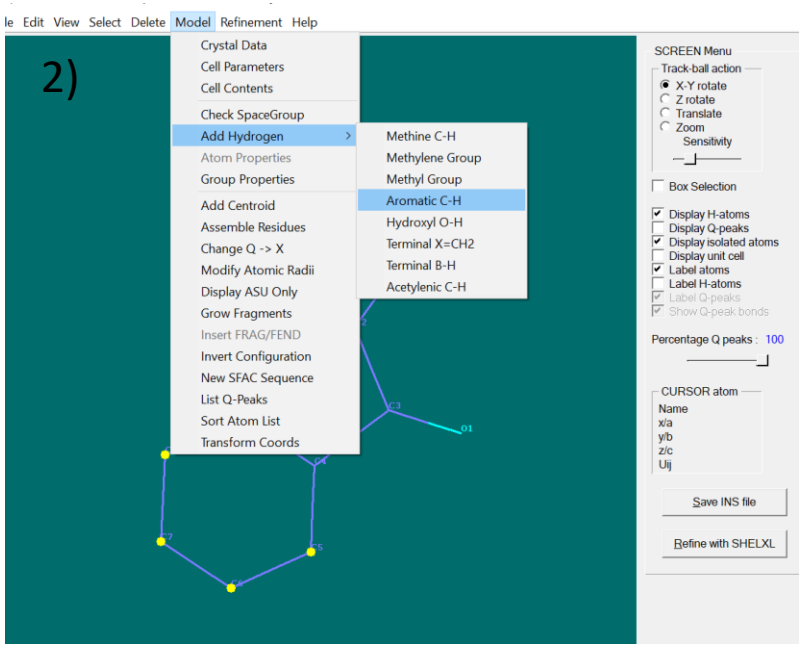
WinGX - Structure refinement step-by-step (SHELXL)

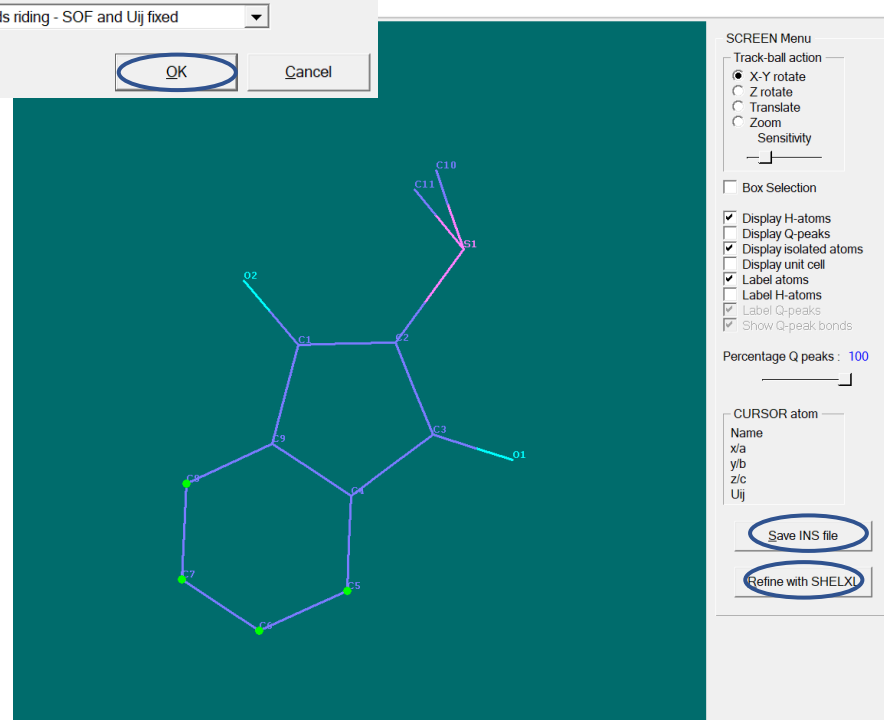
Step 3:

- 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

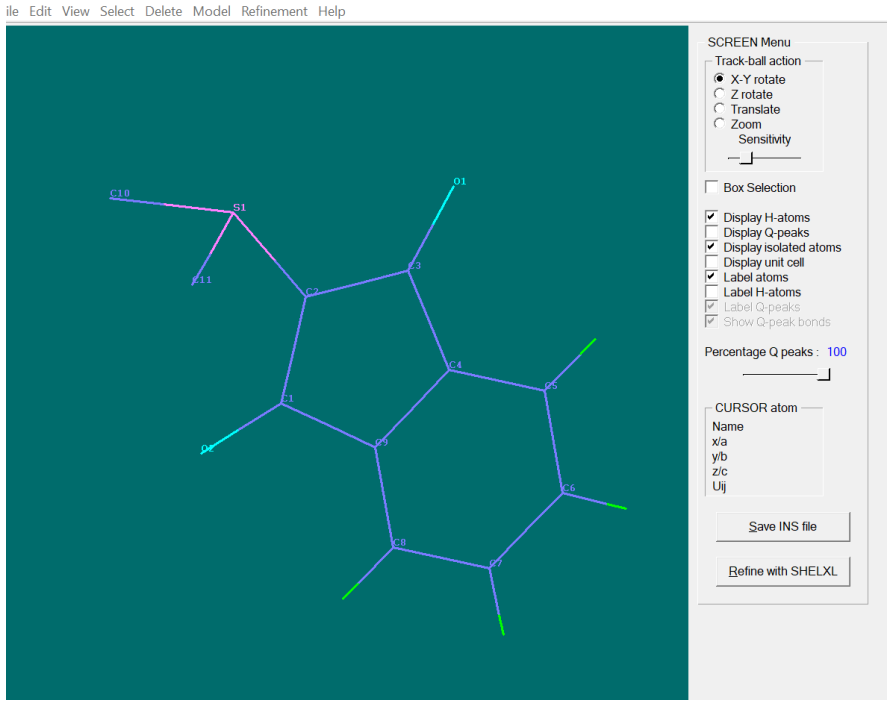








WinGX - Structure refinement step-by-step (SHELXL)



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.

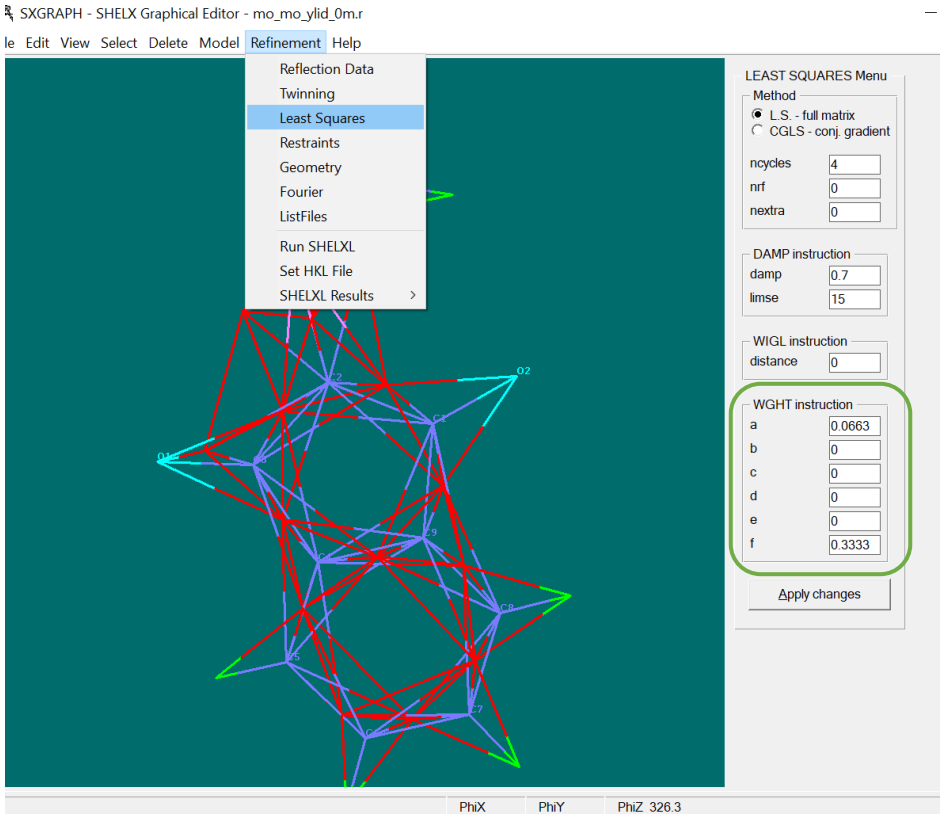
WinGX - Structure refinement step-by-step (SHELXL)

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



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?