TAAM in Olex2 Tutorial

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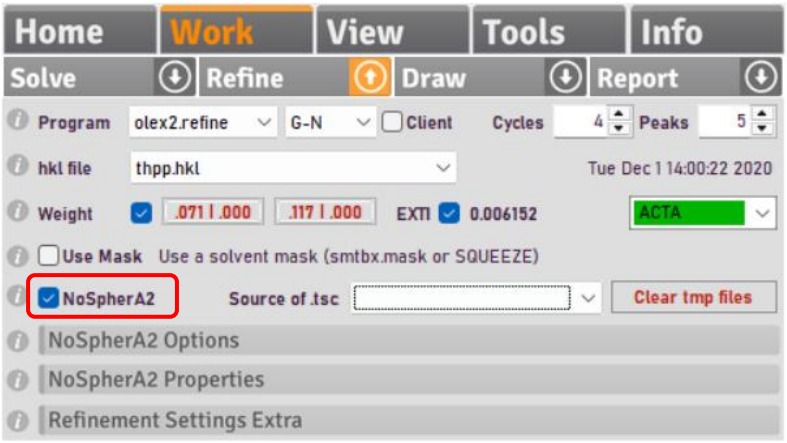
1. Installing DiSCaMB

1.1. Semiautomatic procedure

a. Launch Olex2-1.5 and go to 'Work' → 'Refine'.

b. Select '☑NoSpherA2':



c. From the 'Source of .tsc' select 'Get discambMATTS'. This action will redirect you to the DiSCaMB webpage.

d. On the DiSCaMB webpage, (https://4xeden.uw.edu.pl/software/discamb/), locate and click **Download DiscambMATTS2tsc**. Fill in and submit the form to initiate the automatic download of 'discambMATTS2tsc_v3.009.zip'. Unpack the zip folder.

e. Copy the 'discambMATTS2tsc.exe' file to the folder were Olex2-1.5 is installed. With default Olex2 installation on Windows that woule be 'C:\Program Files\Olex2-1.5' folder. In case of any problems, read README file, which is distributed with 'discambMATTS2tsc.exe'.

f. Restart Olex2-1.5 to complete the setup.

1.2. Manual procedure:

a. Go directly to the the DiSCaMB webpage, (https://4xeden.uw.edu.pl/software/discamb/), locate and click **Download DiscambMATTS2tsc**. Fill in and submit the form. After submitting the link to discambMATTS2tsc_v3.009 download will appear. Click on the link to initiate the automatic download of 'discambMATTS2tsc_v3.009.zip'. Unpack the zip folder.

b. Copy the 'discambMATTS2tsc.exe' file to the folder were Olex2-1.5 is installed. With default Olex2 installation on Windows that woule be 'C:\Program Files\Olex2-1.5' folder. In case of any problems, read README file, which is distributed with 'discambMATTS2tsc.exe'.

c. Open Olex2-1.5.

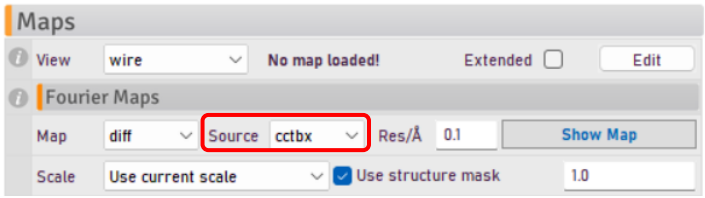
2. Example I: standard case (XRD)

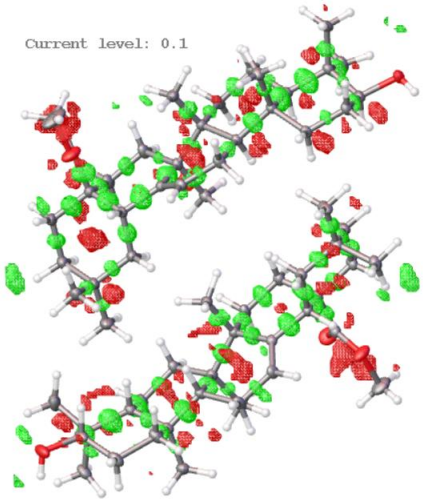
2.1. Preparation

a. Open the structure in Olex2-1.5. Provided data are sourced from *Langer, D., Wicher, B., Bendzinska-Berus, W., Bednarczyk-Cwynar, B. & Tykarska, E. (2022). Acta Cryst. B78, 606-617.*

b. Repeat the last refinement cycle with olex.refine:

c. To inspect the Fourier residual density map, go to the 'Tools → Maps' menu and select 'diff' map option. Choose "Source: cctbx".



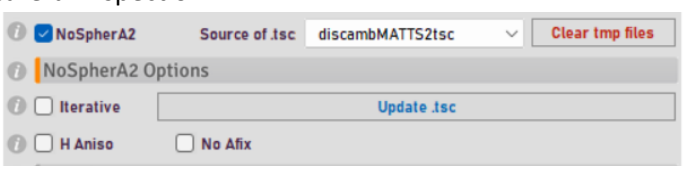


Positive (green) residuals observed on C-C bonds suggest that refinement with the TAAM may improve the structural model.

d. Save files from this step for further comparison with the TAAM refinement.

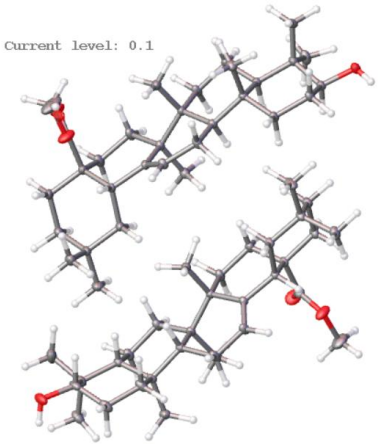
2.2. TAAM refinement

a. In the 'Refine' tab, activate '☑NoSpherA2' and select 'discambMATTStsc' from the drop menu. In 'NoSpherA2 Options' please make sure that '☐H Aniso' is unselected. Anisotropic ADP for hydrogen atoms can be applied in case of TAAM refinement, but they always require careful inspection.



b. Refine until the convergence then repeat the selection of 'discambMATTStsc' and refine again until convergence.

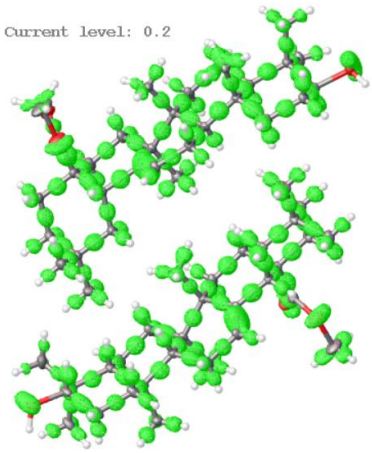
c. Visualize Fourier residual density map.

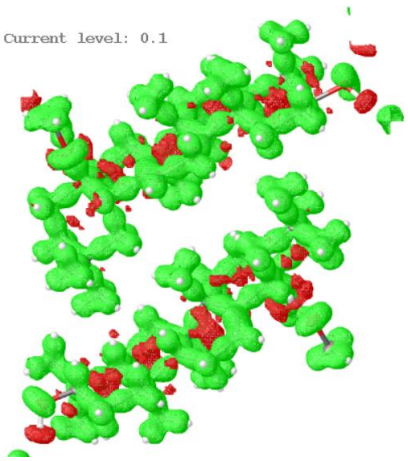


There are no residues visible at the same contour level as visualized for IAM refinement.

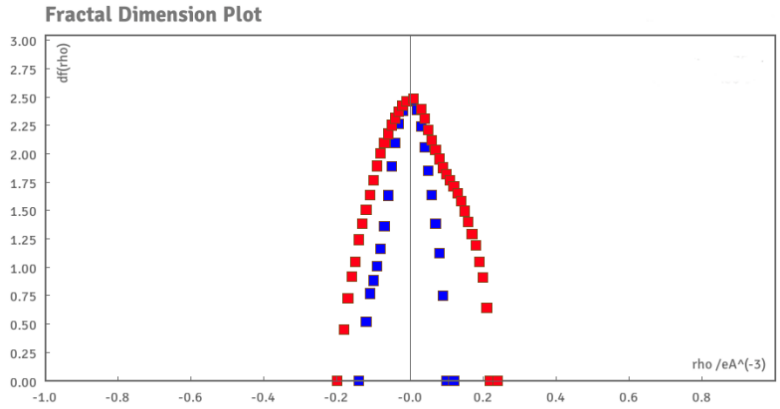
2.3. Analysis of the refinements results

a. Compare refinement statistics (R1, wR2 and other relevant metrics)
b. Evaluate differences in Fourier residual density maps.
c. Visualize and evaluate electron density deformation map: 'Refine' → 'NoSpherA2 Properties' → 'Plot: deformation'





d. Generate fractal dimension plots: 'Info' → 'Reflection statistics' → 'Fractal Dimension'. On the figure below, colors are changed for better comparison.



red: IAM, blue: TAAM

e. Compare X-H bond lengths in '.cif' file. Example values:

	IAM [Å]	TAAM [Å]
C1A-H1Aa	0.978(16)	1.111(12)
C2A-H2Aa	0.986(17)	1.092(12)
O3B-H3Ba	0.83(2)	0.905(14)

f. Check '.log' files:
After TAAM refinement, access the 'discambMATTS2tsc.log' file in the 'olex2/Wfn_job/' directory. This log details atom types and local coordinate systems assigned to the molecule. Any unrecognized atoms will be listed here.

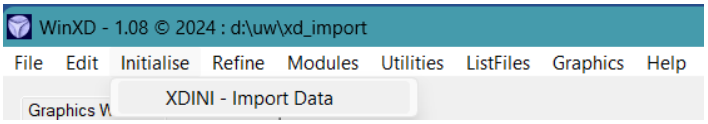
2.4. Preparing '.cif' file for publication:

a. If you used discambMATTS2tsc in your work please add the following text to the resulting .cif file :

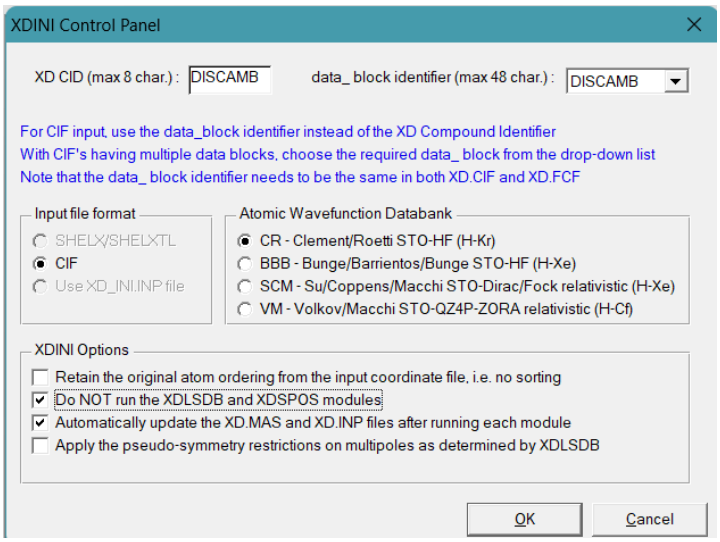
_refine_special_details
;
TAAM / MATTS refinement.
Uses aspherical atomic scattering factors computed by the DiSCaMB library
(Chodkiewicz et.al., J.Appl.Cryst., 2018, 51, 193 - 199)
from multipole model
(Hansen & Coppens, Acta Cryst.A, 1978, 34, 909 - 921)
parametrized using the MATTS2021 data bank
(Jha, et al., J.Chem.Inf.Model., 2022, 62, 3752 - 3765,
Rybicka, et al., J.Chem.Inf.Model., 2022, 62, 3766 - 3783)
Refinement performed with.tsc generated by discambMATTS2TAAMtsc v3.009
;

2.5. Exporting TAAM refinement to XD2024:

a. Locate the '<name>.cif_rho' file in 'olex2/Wfn_job/', which contains multipole parameters for the molecule.
b. Prepare the '.cif_rho' file and '.fcf' files. Change their names according to XD2024 manual (e.g. to 'shelx.cif' and 'shelx.fcf')
c. Run XD2024, set the working directory, and run 'Initialize' → 'XDINI'.



d. In the XDINI interface, select '☑Do NOT run the XDLSDB and XDSPOS modules':



e. Click Ok
f. Verify the presence ‘xd.inp’ and ‘xd.mas’ files in the working directory and review their contents.

3. Example II: Disorder (XRD)

3.1. Preparation

a. Open the structure in Olex2-1.5. Provided data are sourced from
Yi-Hua, Y., Kai-Li, Y., Qian, L., Xu-Guang, M. & Shou-Xin, L. (2019). Chin. J. Struct. Chem. 38, 2077–2082
TAAM refinement has also been presented in
Jha, K. K., Kleemiss, F., Chodkiewicz, M. L. & Dominiak, P. M. (2023). J. Appl. Cryst. 56, 116-127.

b. Repeat the last refinement cycle with olex.refine:

Notice the presence of AFIX constraints.

3.2. TAAM refinement

a. In the 'Refine' tab select '☑NoSpherA2' and '☑Update Table: discambMATTS'.
Unselect '☐H Aniso' and '☐No Afix'.
Note: Removing AFIX constraints for the terminal alkenyl chain may lead to incorrect geometry.

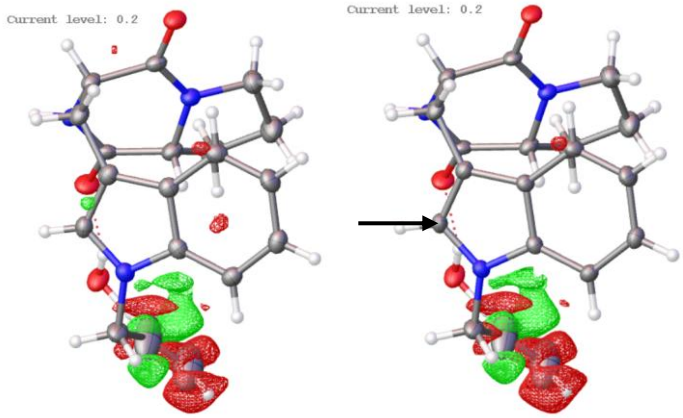
b. Refine until the convergence then repeat the 'Update Table' step and refine again until convergence.

TAAM does not improve the structure, with increased maximum/minimum Fourier residuals and barely lowered R1. This suggests fundamental problems in the structure.

3.3. Analysis of the refinements results

a. Visualize Fourier residual density map.

IAM TAAM



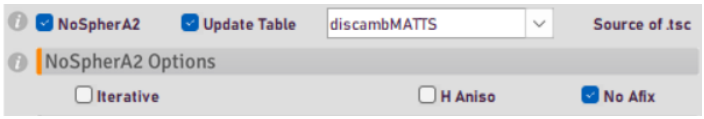
They reveal unmodeled disorder in the terminal alkenyl chain, evident even in IAM refinement.

3.4. Modeling disorder (TAAM)

a. Select atoms C23,C24,H23,H24a,H24b and navigate to 'Work' → 'Toolbox Work' → 'Modelling and Disorder Tools'. Click 'Split' chosing 'SAME RIGU' option and move a little.

b. Return to the 'Refine' tab and select '☑NoSpherA2' and '☑Update Table: discambMATTS'. Refine until convergence. Then apply anisotropic ADP to disordered atoms, "Update Table' again and refine until convergence.

c. Remove AFIX constraints by selecting '☑No Afix' option:



WARNING: if the alkenyl group deviates from planarity, the proper atom type cannot be assigned. Check the 'discambMATTS2tsc.log' file in './olex2/Wfn_job/' for details on unassigned atom type.

d. Similarly, model disorder in IAM refinement and remove AFIX constraints via 'Tools' → 'Hydrogen Atoms' → 'Free ALL H'.

3.5. Further analysis

Below there are figures based on IAM and TAAM refinements with modeled disorder and no AFIX.

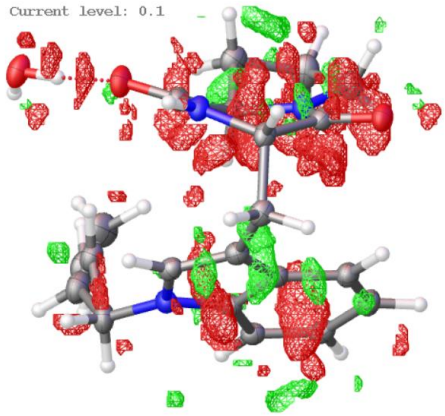
a. Compare refinement statistics (R1, wR2, etc.)

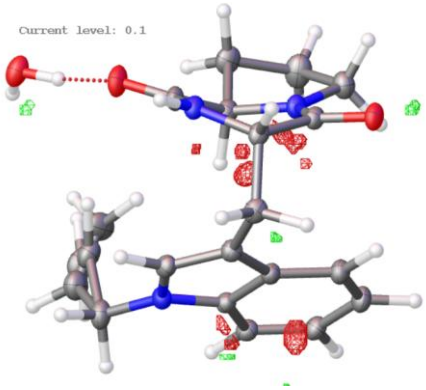
IAM:

TAAM:

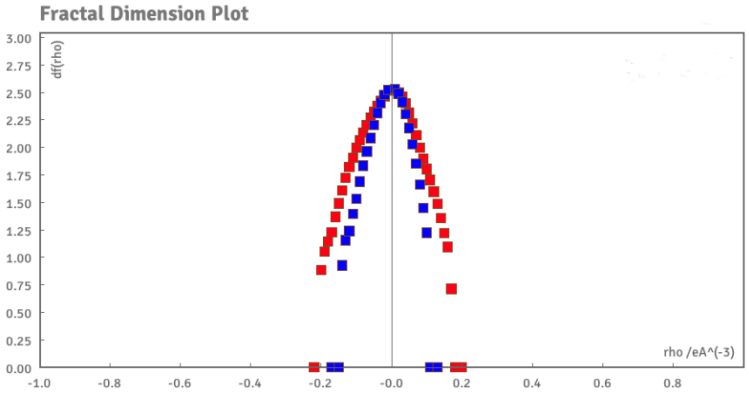
b. Compare Fourier residual density maps.

IAM TAAM





c. Compare fractal dimension plots:



red: IAM, blue: TAAM

d. Investigate X-H bond lengths in '.cif' file. Example values:

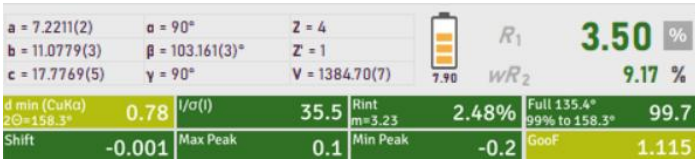
	IAM [Å]	TAAM [Å]
O1W-H1Wa	0.85(3)	0.92(2)
N12-H12	0.87(2)	0.969(16)
C7-H7	0.935(18)	1.071(15)
C17-H17a	0.95(2)	1.080(15)

4. Example III: Anharmonicity

4.1. Preparation

a. Open the structure in Olex2-1.5. Provided data are sourced from *Zwolenik, A. & Makal, A. (2025). IUCrJ, 12, 23-35.*

b. Repeat the last refinement cycle with olex.refine:

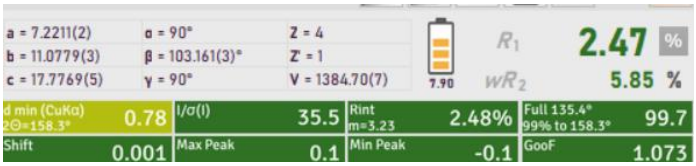


Anharmonic ADPs for oxygen atoms, as noted in the publication, are already applied and can be inspected in the '.res' file.

4.2. TAAM refinement

a. In the 'Refine' tab, activate '☑NoSpherA2' and select '☑Update Table: discambMATTS'. As hydrogen atoms are refined anisotropicaly and there are no AFIX constraints applied, you can leave 'NoSpherA2 Options' unchanged.
The existing anharmonic ADPs for oxygen atoms require no additional adjustments when using NoSpherA2 with DiSCaMB.

b. Refine until the convergence, then repeat the 'Update Table' step and refine again until convergence.

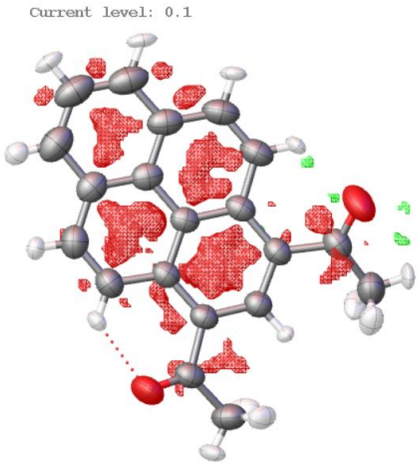


4.3. Analysis of the refinements results

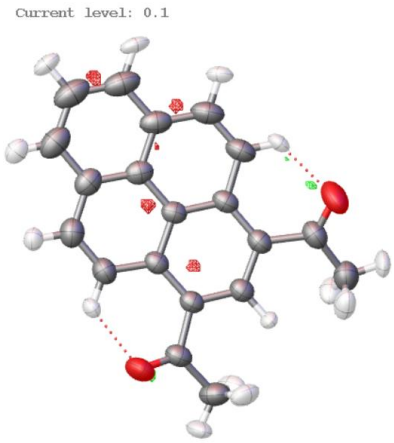
a. Compare refinement statistics (R1, wR2 and other relevant metrics)

b. Visualize and compare Fourier residual density map.

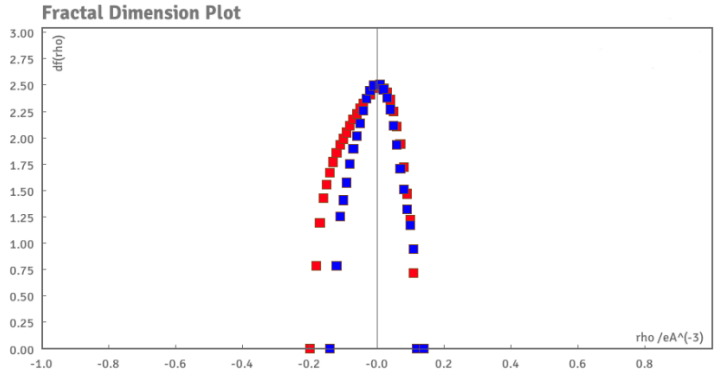
IAM



TAAM



c. Generate and compare fractal dimension plots. On the figure below, colors are changed for better comparison.



red: IAM, blue: TAAM

d. Compare X-H bond lengths in '.cif' file. Example values:

	IAM [Å]	TAAM [Å]
C3-H3	0.988(14)	1.052(12)
C6-H6	1.015(19)	1.076(16)
C14-H14	1.004(13)	1.088(11)
C18-H18a	1.02(2)	1.050(18)

5. Example IV: standard case (3DED)

5.1. Preparation

a. Open the structure in Olex2-1.5. Provided data are sourced from
Kumar, A., Jha, K. K., Olech, B., Goral, T., Malinska, M., Wozniak, K. & Dominiak, P. M. (2024). Acta Cryst. C80, 264-277.

b. Repeat the last refinement cycle with olex.refine:

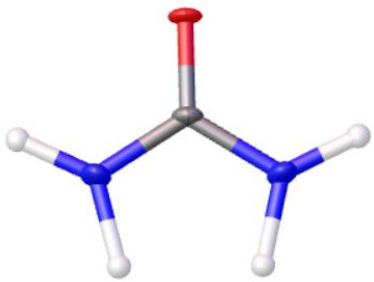
Note the appearance of the 'ED SFAC' option:

5.2. TAAM refinement

a. In the 'Refine' tab select '☑NoSpherA2' and '☑Update Table: discambMATTS'.
In 'NoSpherA2 Options' unselect '☐H Aniso'.

b. Refine until the convergence then repeat the 'Update Table' step and refine again until convergence.

WARNING! Refinement must be performed on asymmetric unit. The '.tsc' file is calculated for the asymmetric unit, and attempting refinement on a larger fragment (e.g., the full molecule) will result in an error.



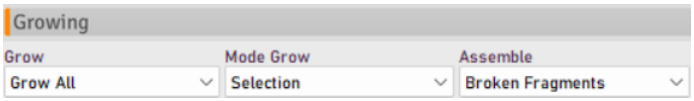
```
Refinement finished at: 2025-02-18 17:09:19.154487
Using tabulated atomic form factors

+++ STARTING olex2.refine +++++ C:\Program Files\Olex2-1.5/cctbx
Using 6 threads. Using OpenMP: False.
Number of atoms in model and table are not matching!
Error!:
Insufficient number of scatterers in .tsc file!
Did you forget to recalculate after adding or deleting atoms?
```

5.3. Analysis of the refinements results

a. Compare refinement statistics (R1, wR2 and other relevant metrics)

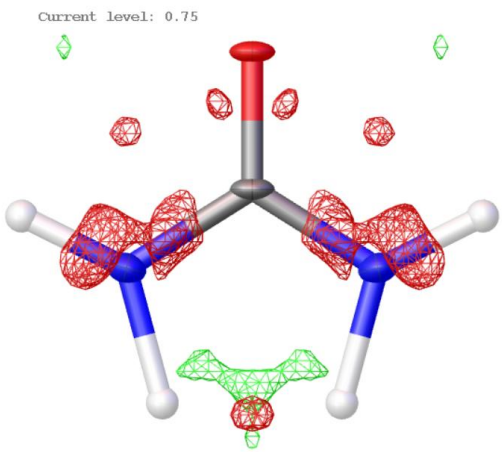
b. Visualize and compare Fourier residual density map.

To extend the view, go to 'Work' →'Toolbox Work'→'Growing' and select 'Grow all':

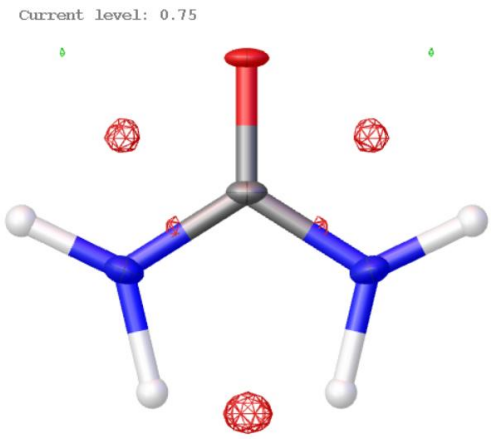


Then visualize Fourier residual density map.

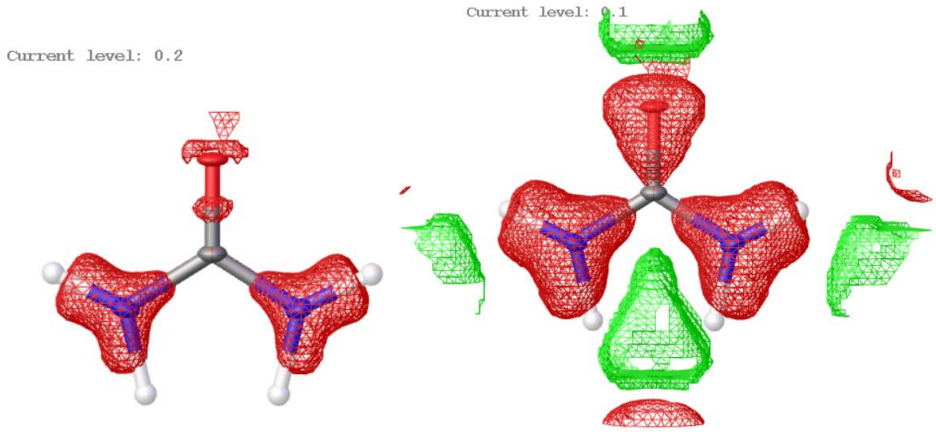
IAM



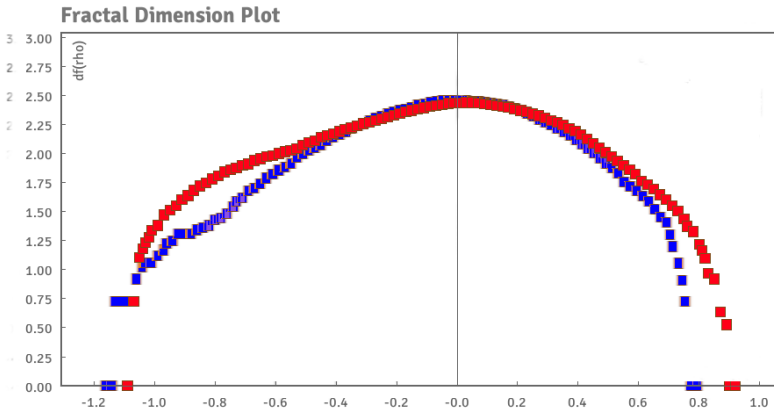
TAAM



e. Visualize and evaluate electrostatic potential deformation map



f. Generate and compare fractal dimension plots. On the figure below, colors are changed for better comparison.



red: IAM, blue: TAAM

c. Compare X-H bond lengths in '.cif' file:

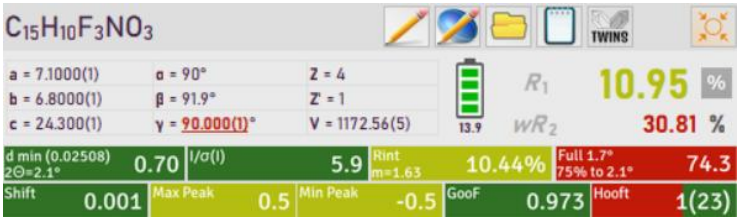
	IAM [Å]	TAAM [Å]
N1-H1a	1.06(3)	1.05(2)
N1-H1b	1.27(7)	1.17(4)

6. Example V: Worse data (3DED)

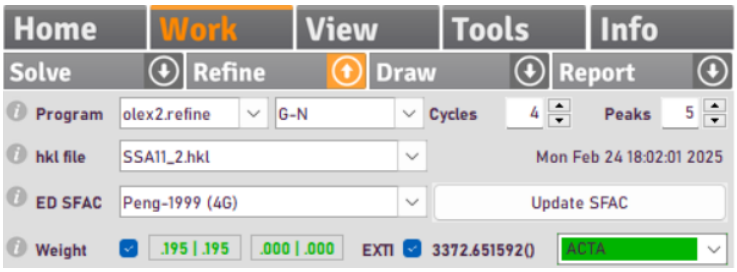
6.1. Preparation

a. Open the structure in Olex2-1.5. Provided data are sourced from
Hawash, M.; Al-Smadi, D.; Kumar, A.; Olech, B.; Dominiak, P.M.; Jaradat, N.; Antari, S.; Mohammed, S.; Nasasrh, A.; Abualhasan, M.; et al. Biomolecules *(2023),* 13, *1486.*

b. Repeat the last refinement cycle with olex.refine:



Note the appearance of the 'ED SFAC' option and enabled 'EXTI' option.



c. AFIX constraints are implemented. Note that X-H bond lengths are fixed to averaged neutron positions. This is the correct choice for 3DED structures, both IAM and TAAM refinements. Below some examples from '.res' file:
AFIX 43 1.083
AFIX 43 1.027
AFIX 23 1.091
Removing them results in inappropriate bond lengths and angles.

6.2. TAAM refinement

a. In the 'Refine' tab select '☑NoSpherA2' and '☑Update Table: discambMATTS'.
Do not select '☐H Aniso' and '☐No Afix'.

b. Refine until the convergence then repeat the 'Update Table' step and refine again until convergence.

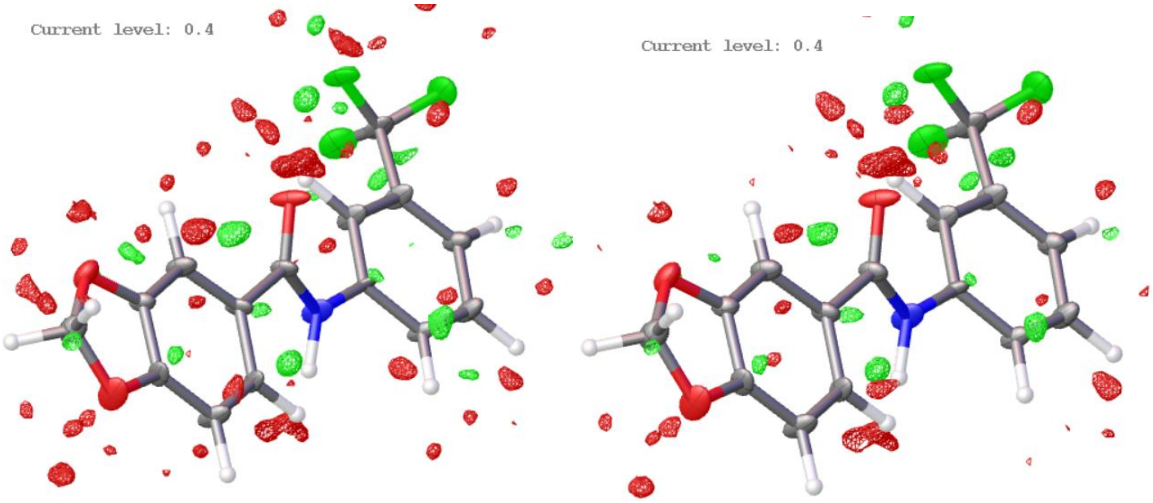
c. Note: Removing AFIX constraints is possible but may lead to incorrect hydrogen atom positions. For consistency, retain AFIX in subsequent comparisons.

6.3. Analysis of the refinements results

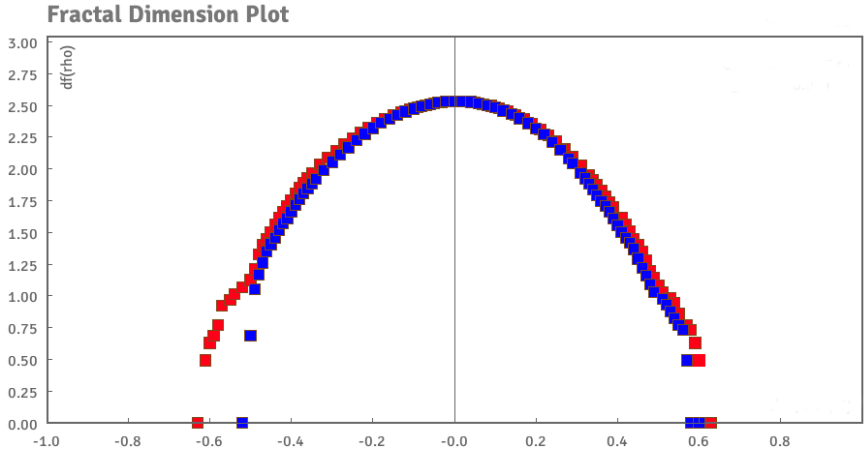
a. Compare refinement statistics (R1, wR2 and other relevant metrics)

b. Visualize and compare Fourier residual density map.

IAM TAAM



c. Generate and compare fractal dimension plots. On the figure below, colors are changed for better comparison.



red: IAM, blue: TAAM