

Tutorial

MoProSuite : Crystallographic software for charge density refinement

<http://crm2.univ-lorraine.fr/lab/software/mopro/download-mopro/>

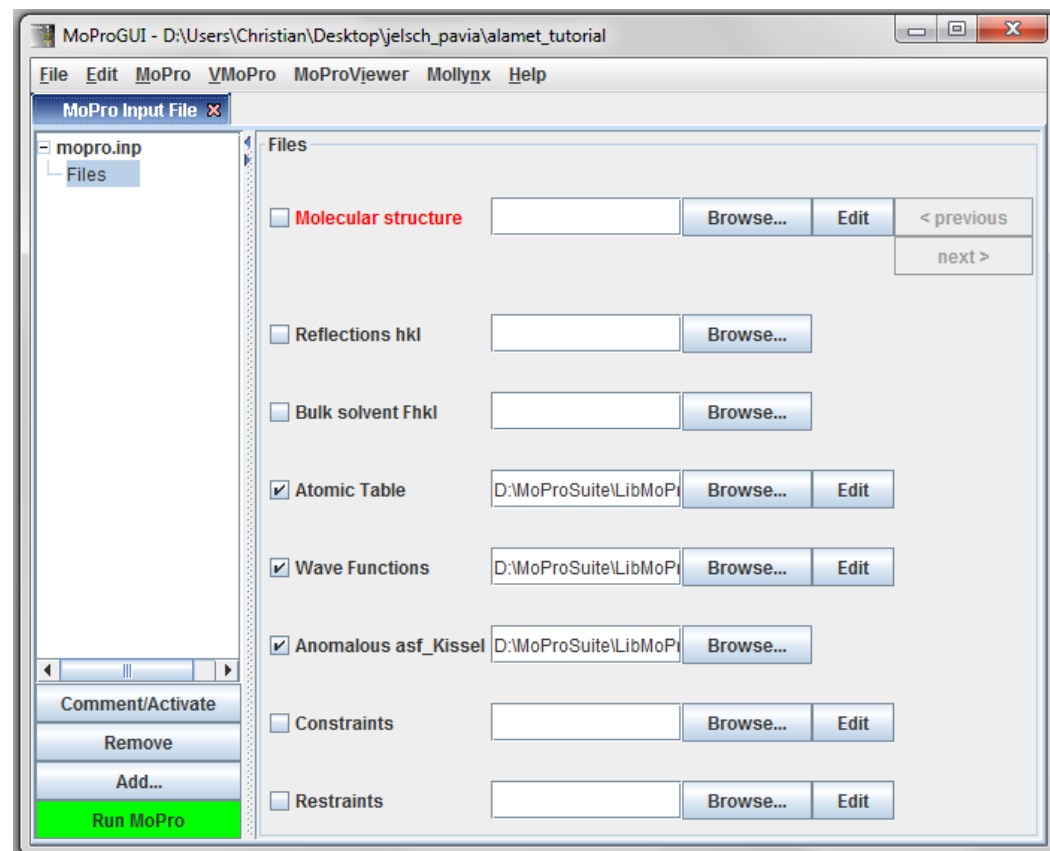
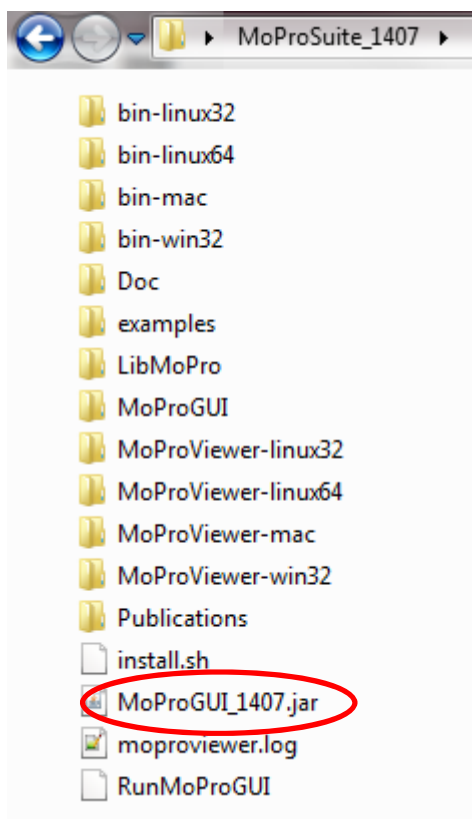
Christian Jelsch. CNRS. Université de Lorraine. France;

Available on windows, linux & mac.

July 2014

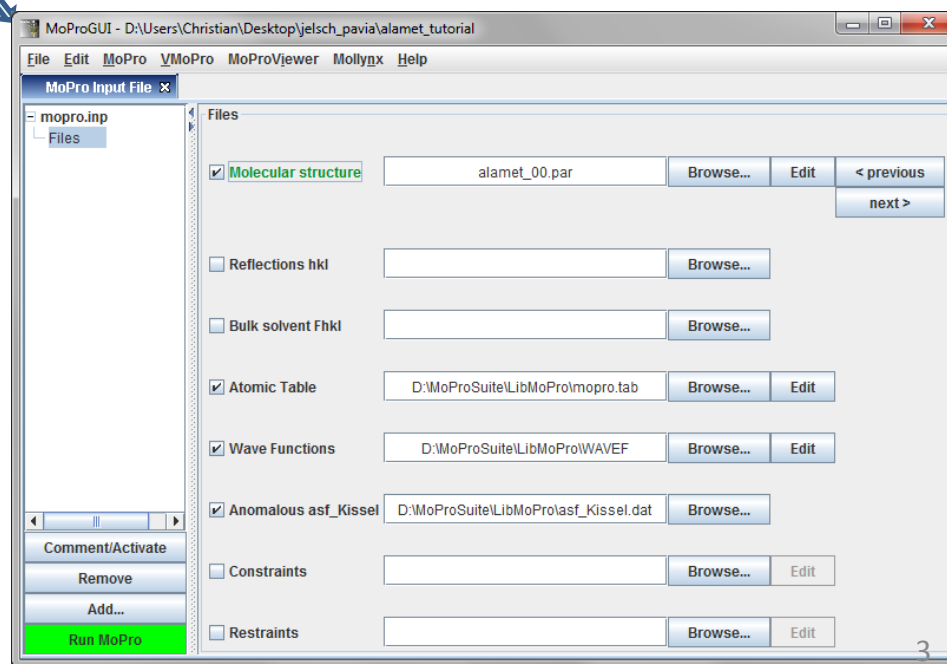
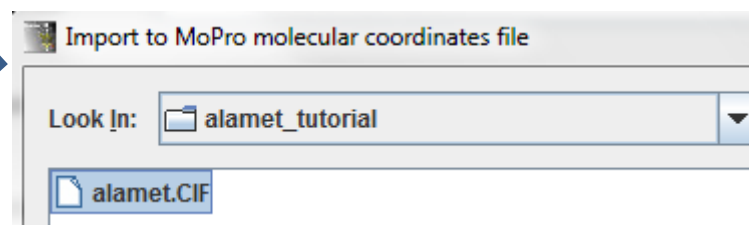
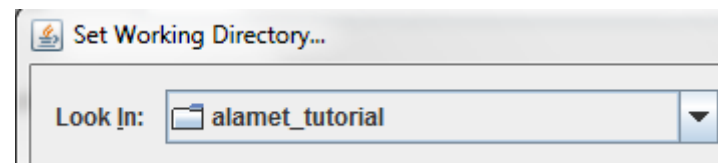
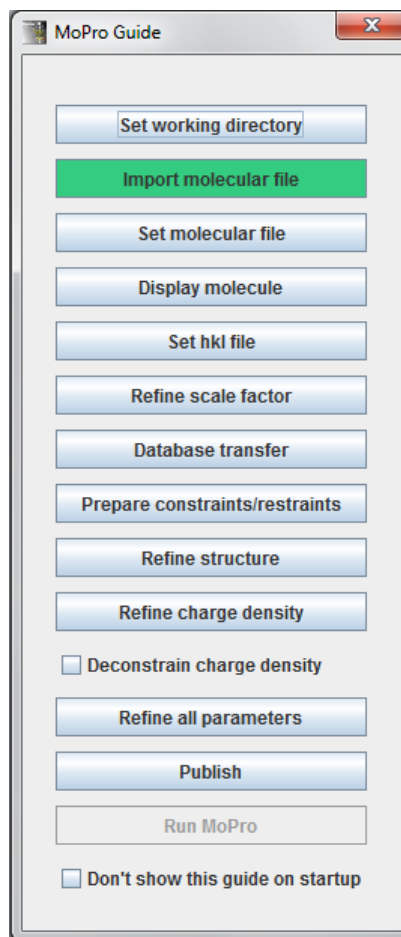
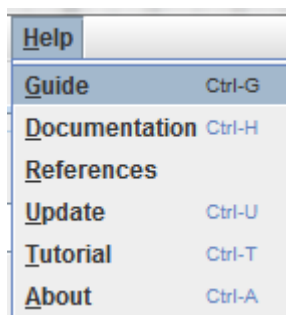
Step 1 Launch MoProGUI

MoPro Graphical User Interface

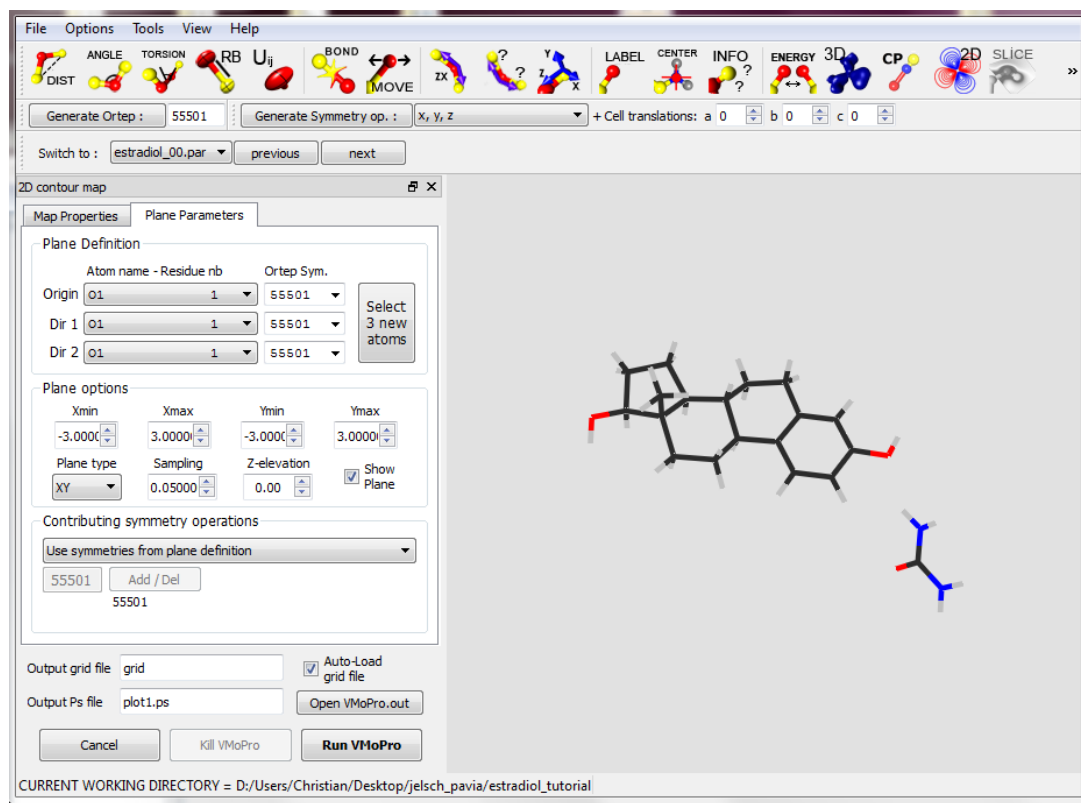
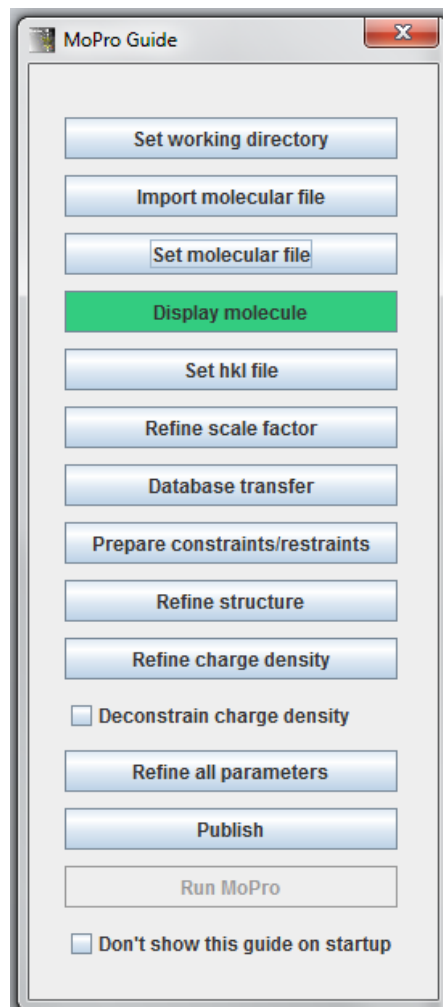


Requires JAVA, which can be downloaded at:
<https://www.java.com/fr/download/>

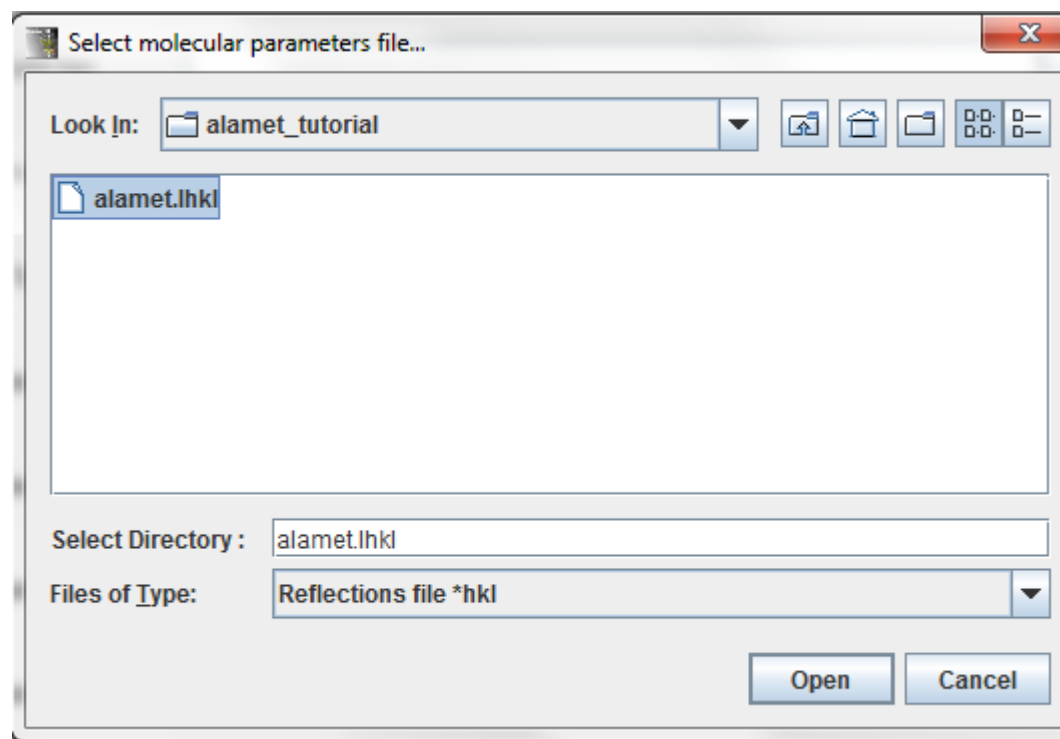
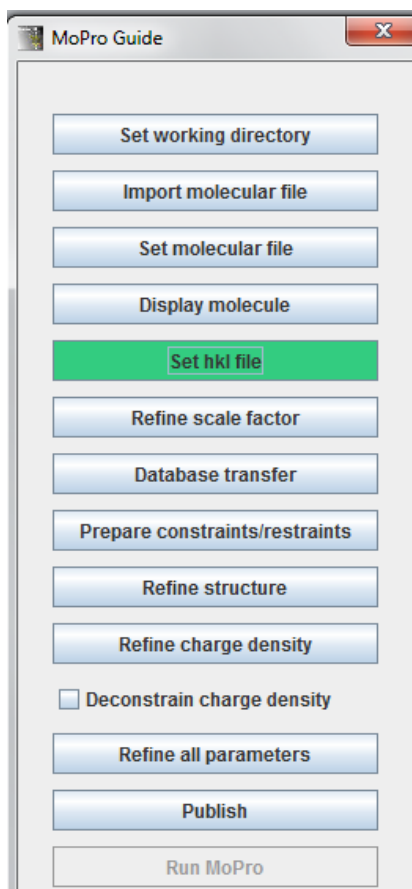
Step 2 initial steps : follow the GUIDE



Step 3 Display molecule with MoProViewer

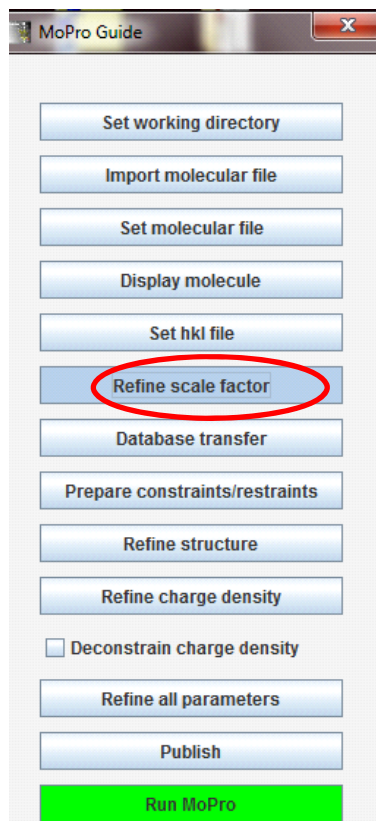


Step 4 Select reflections file

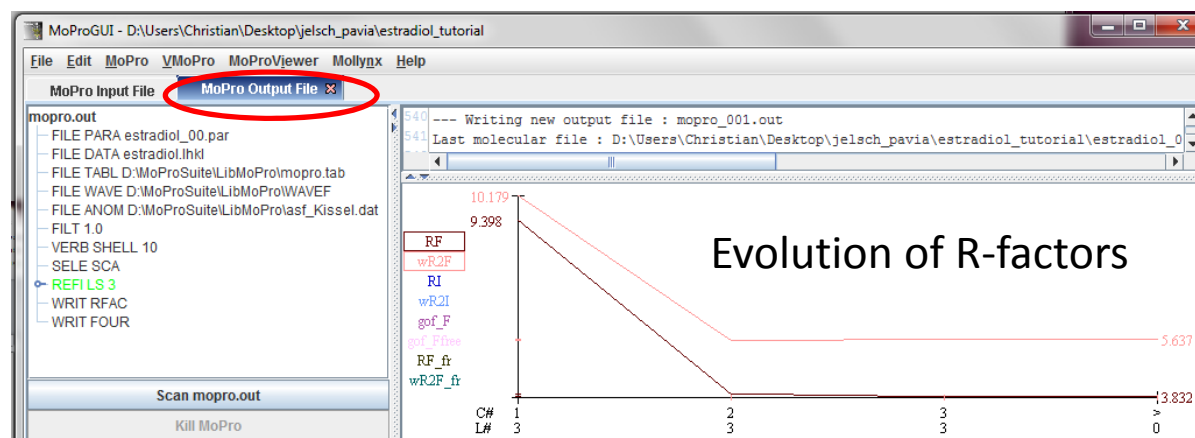
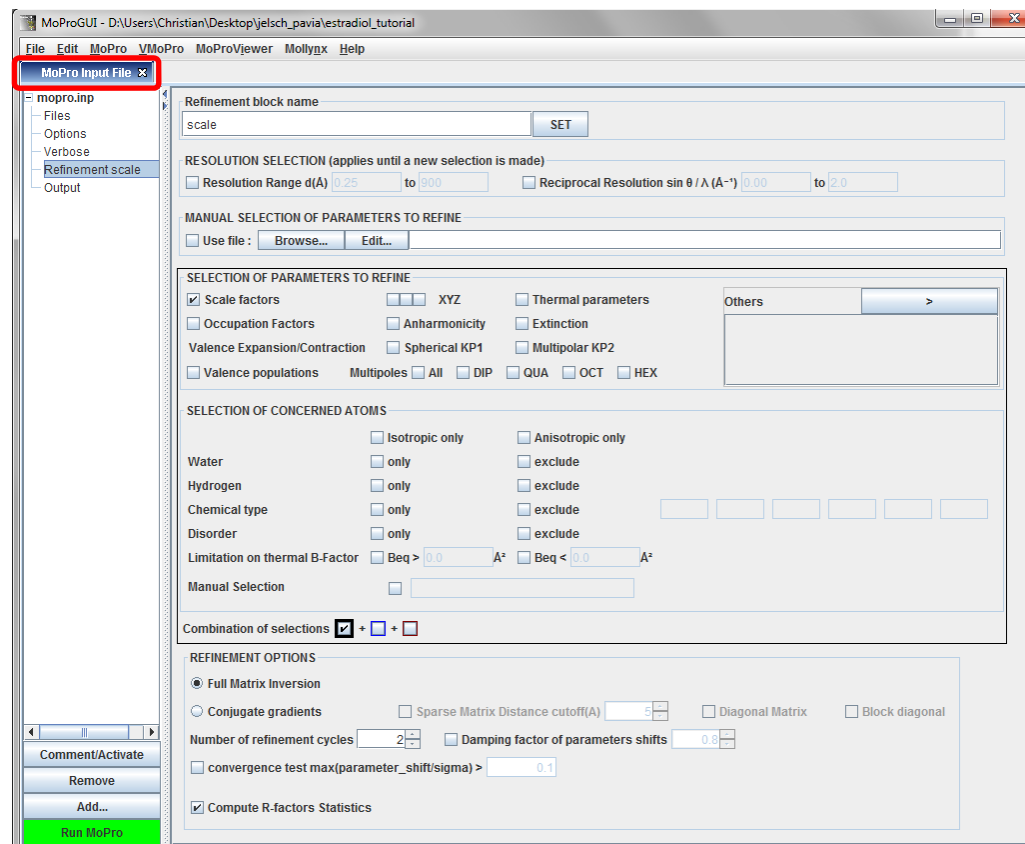


h k l lobs sigma(lobs)

Step 5 Refine SCALE factor

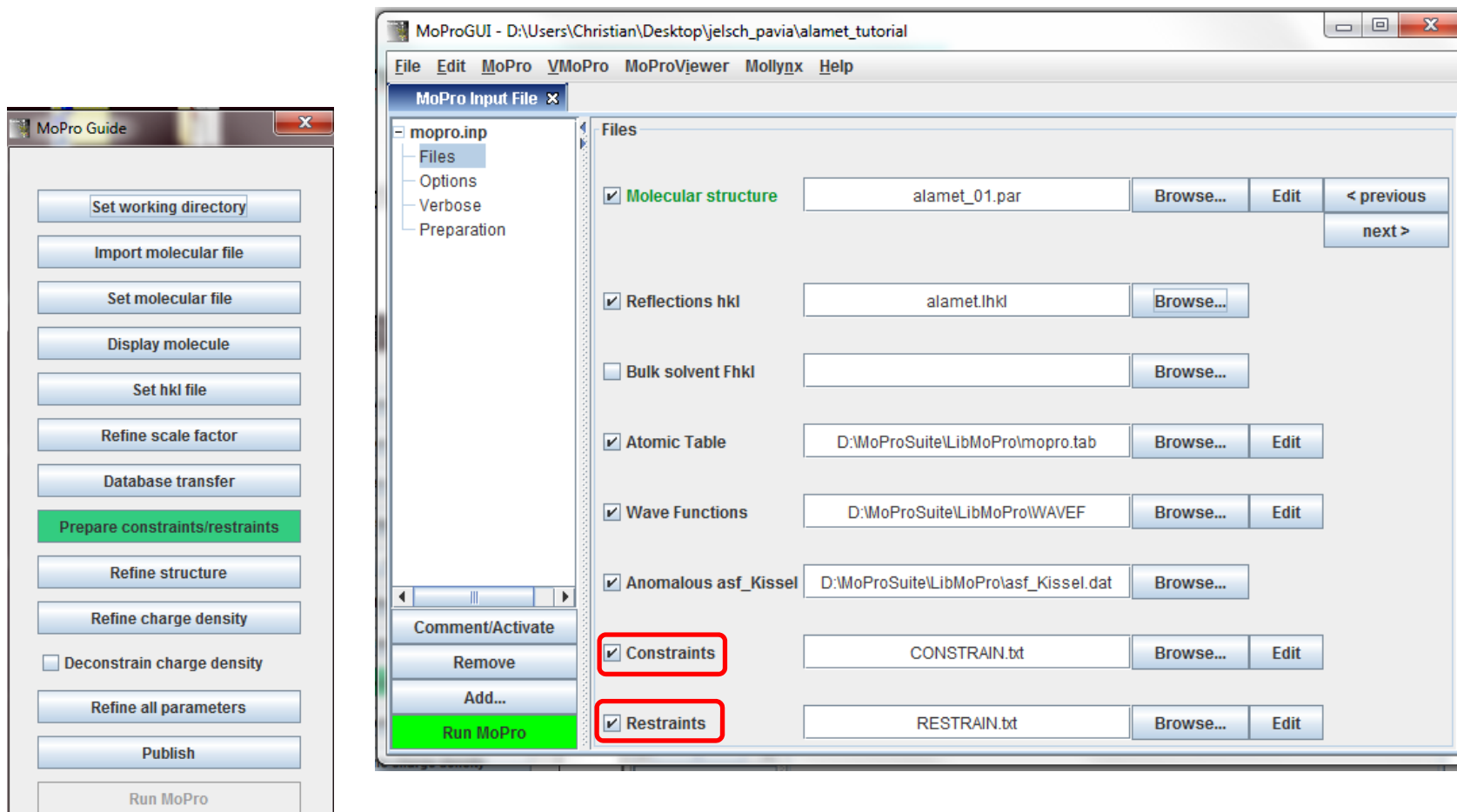


Click on
"mopro Input file"
&
"mopro Output file"



Step 6 Preparation of constraints & restraints

e.g. H-X distances for Hydrogen atoms

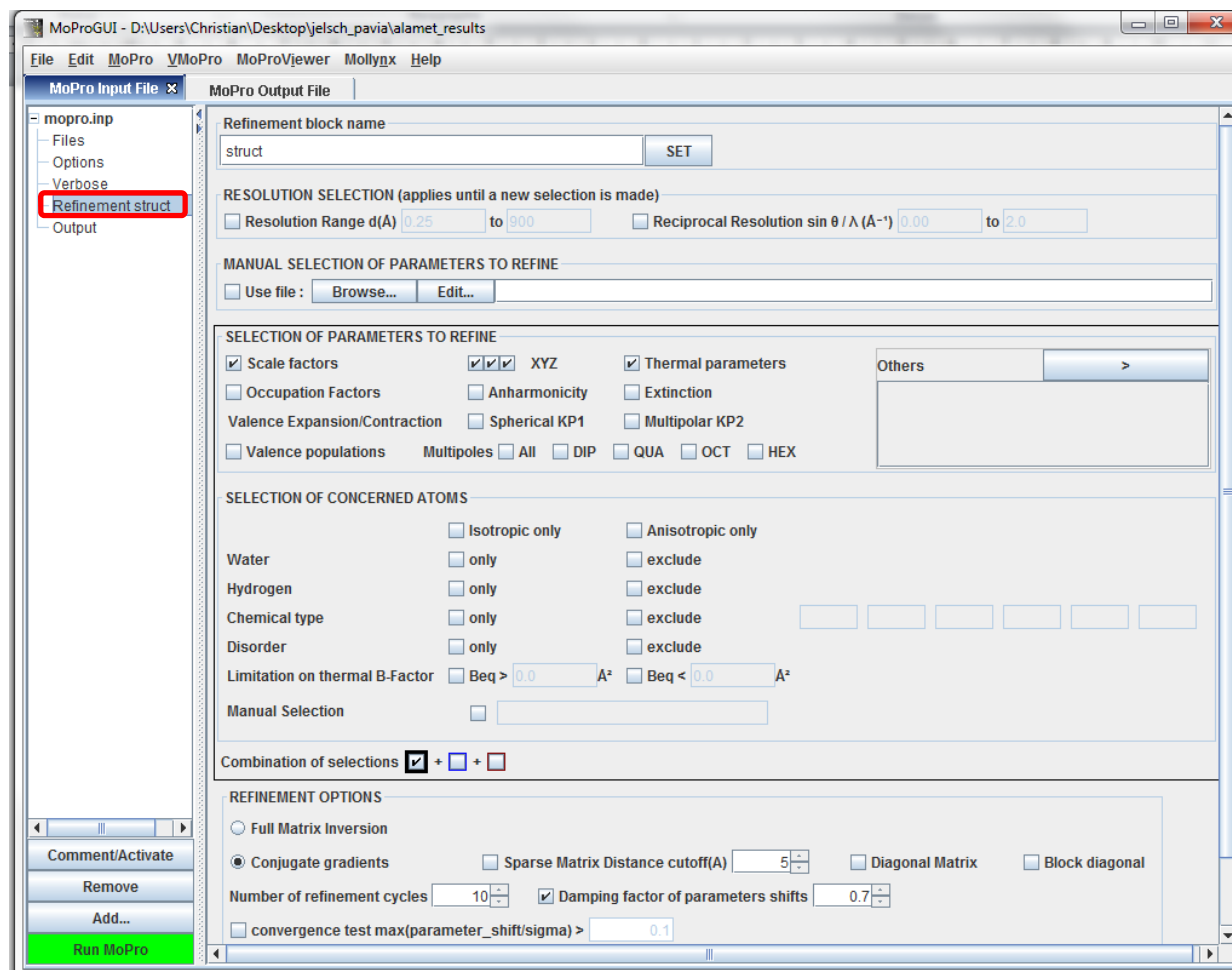
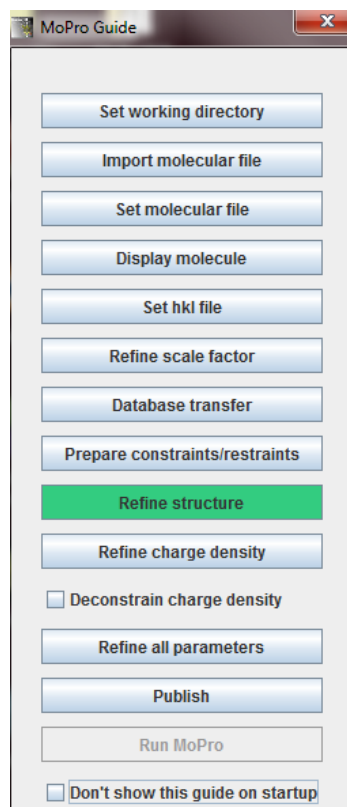


Edit and have a look at the generated
CONSTRAIN.txt & RESTRAIN.txt files

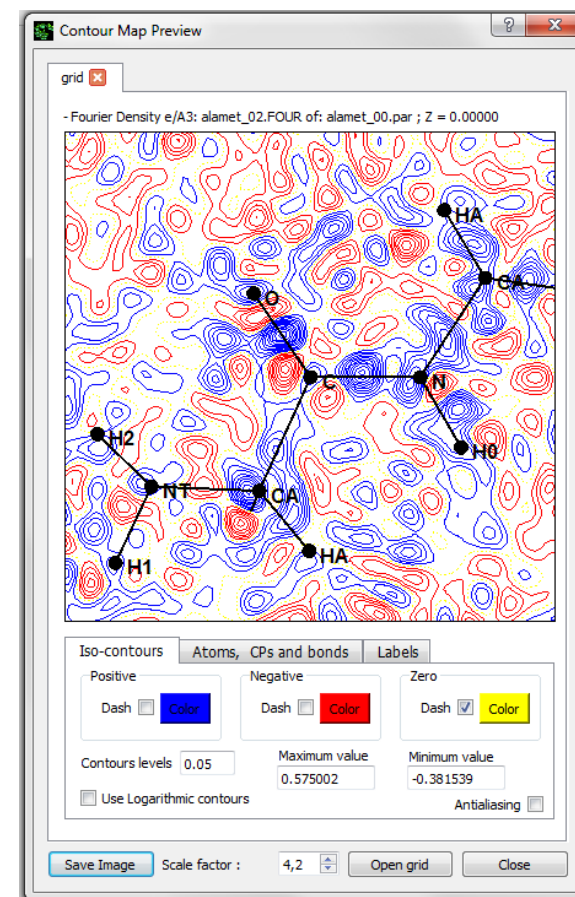
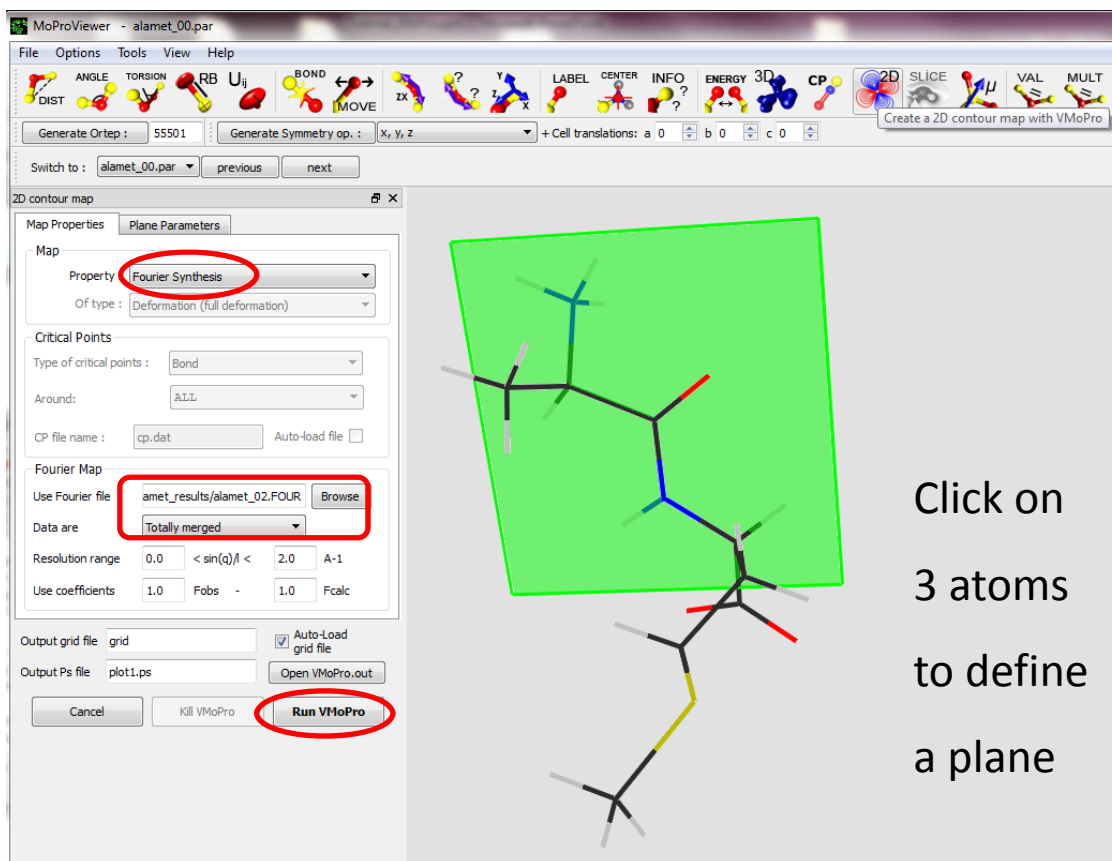
Step 7 Refine structure

SCA XYZ UIJ *are selected*

SCA factor, Positions & Thermal motion parameters



Step 8 Compute a Fourier Residual Electron Density 2D map



2D map:

Select Map Property : Fourier Synthesis

Select Fourier file : alamet_02.FOUR

Data are Merged

Click on "Run VMoPro" to start calculation

Step 9 HIGH ORDER refinement of structure

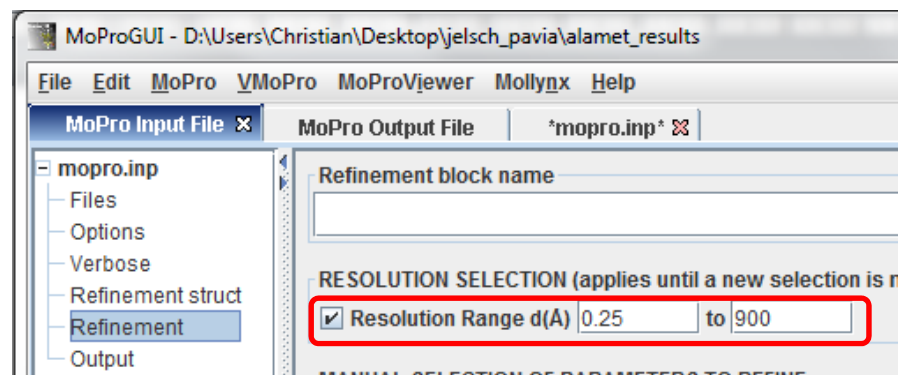
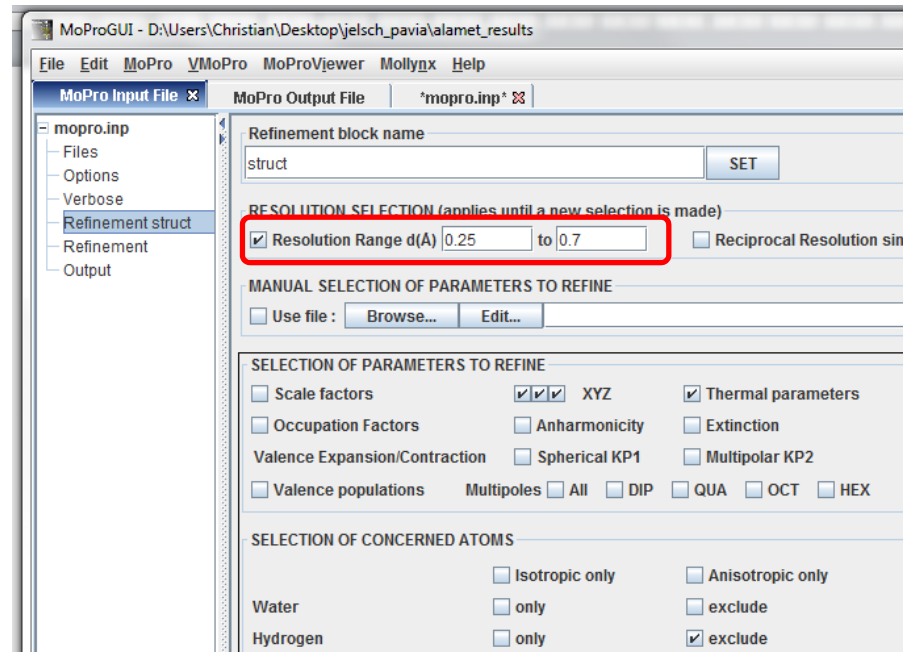
High resolution data $d < 0.7 \text{ \AA}$,
Hydrogen not refined

The MoPro commands

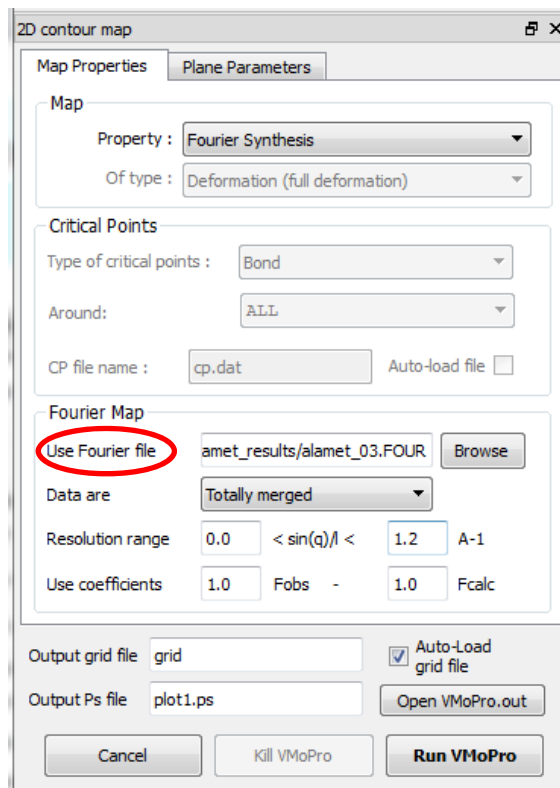
! <refinement> structural
RESO 0.25 0.7
SELE XYZ UIJ NOH
REFI CG 10 DAMP 0.7
WRIT RFAC

RESO 0.25 900
WRIT FOUR

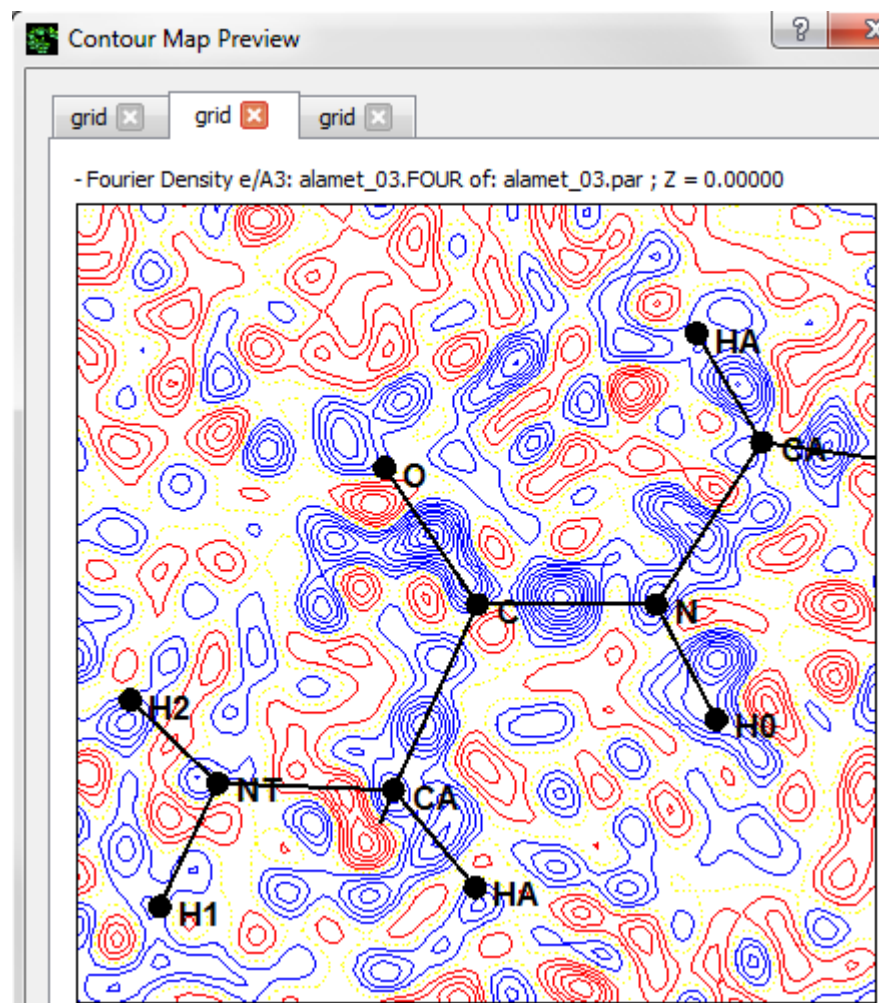
Write a Fourier reflections file
for all resolution



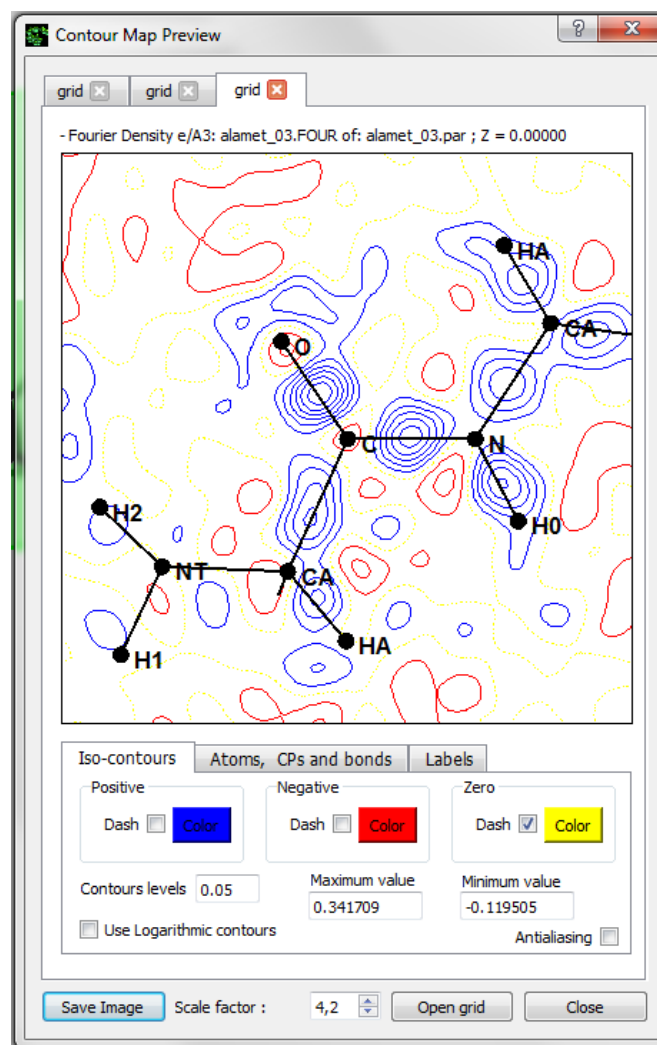
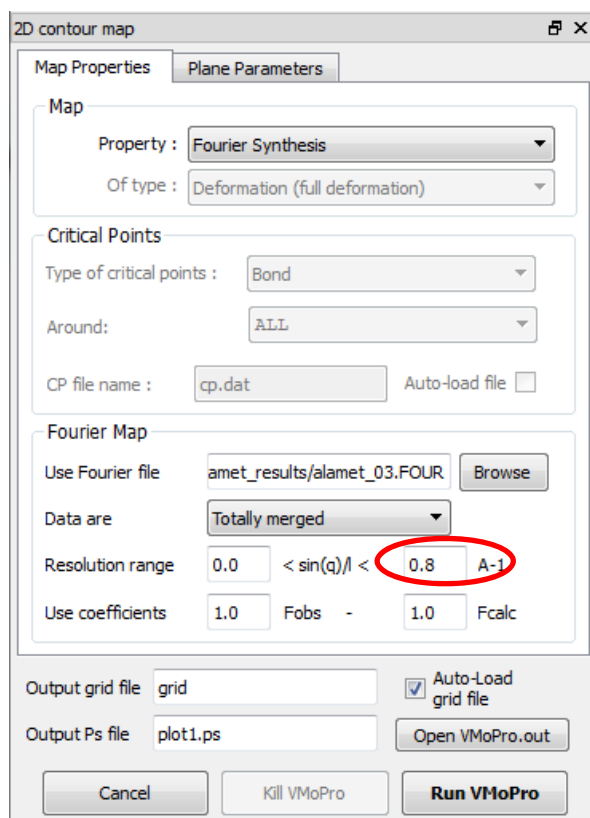
Step 10 Compute a Fourier Residual Electron Density after High-Order refinement



Stronger residual electron density
visible on covalent bonds



Step 11 Compute a Fourier Residual map at LOW resolution after High-Order refinement

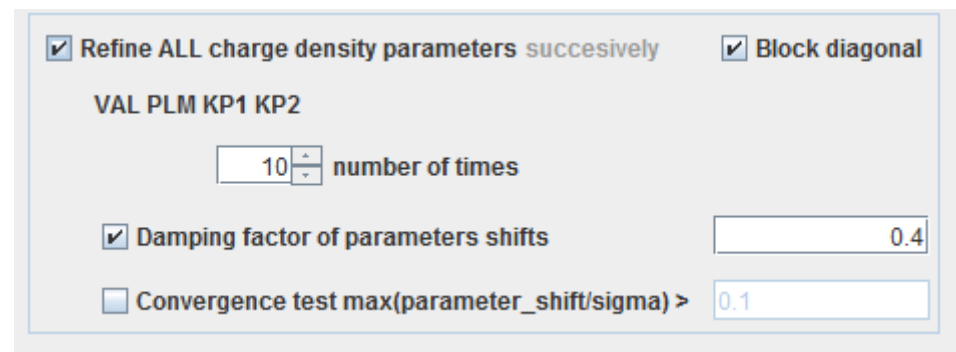
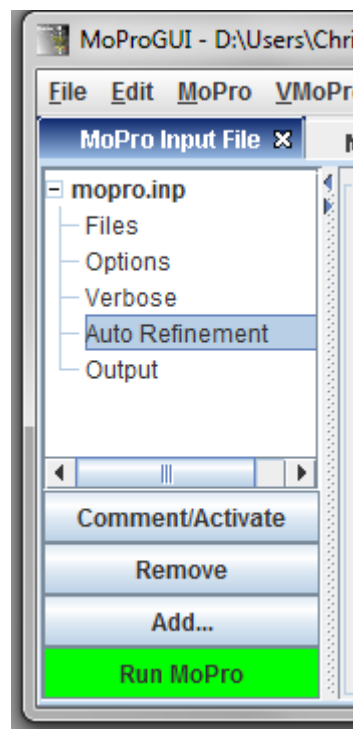
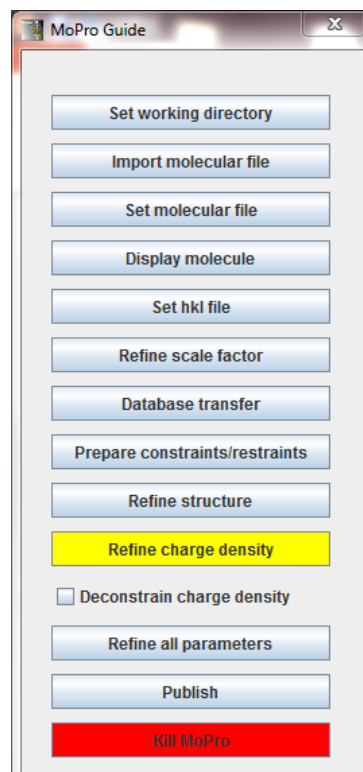


$$\sin \theta / \lambda < 0.8 \text{ \AA}^{-1}$$

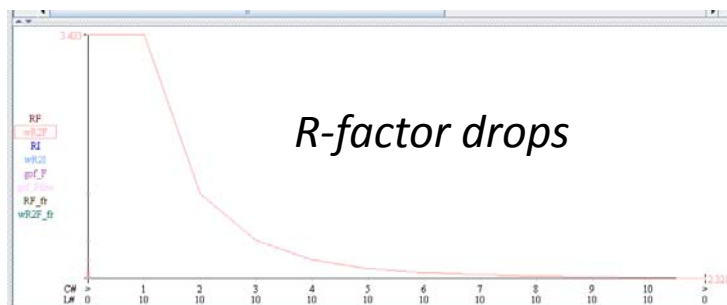
resolution
 $d > 0.6 \text{ \AA}$

Bonding
Electron
Density
is clearly
visible

Step 12 Refinement of charge density



- * Block diagonal: variables are decorrelated and can be refined together
- * Damping to avoid divergence



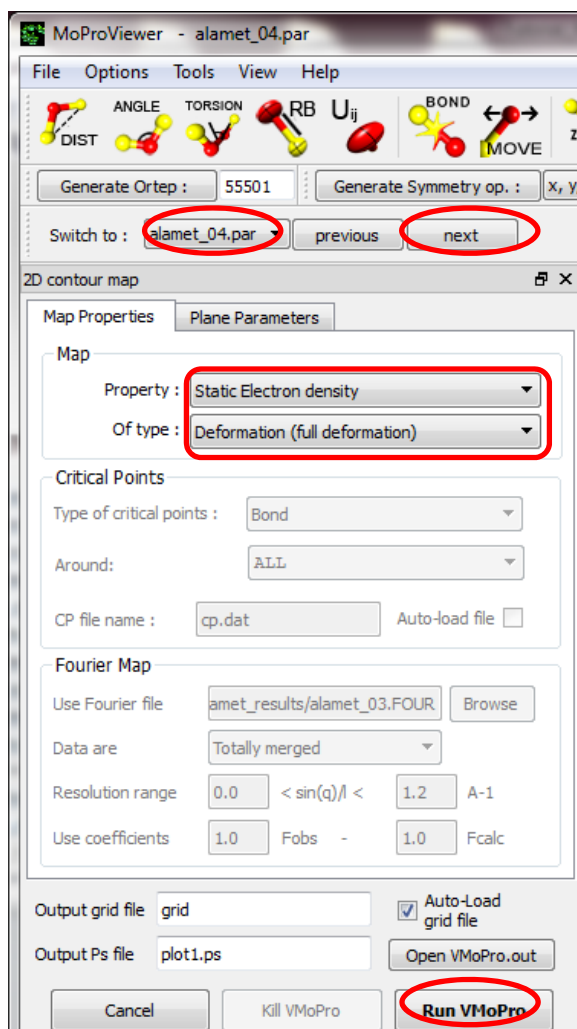
Have a look at resulting molecular .par file

ATOMS 30

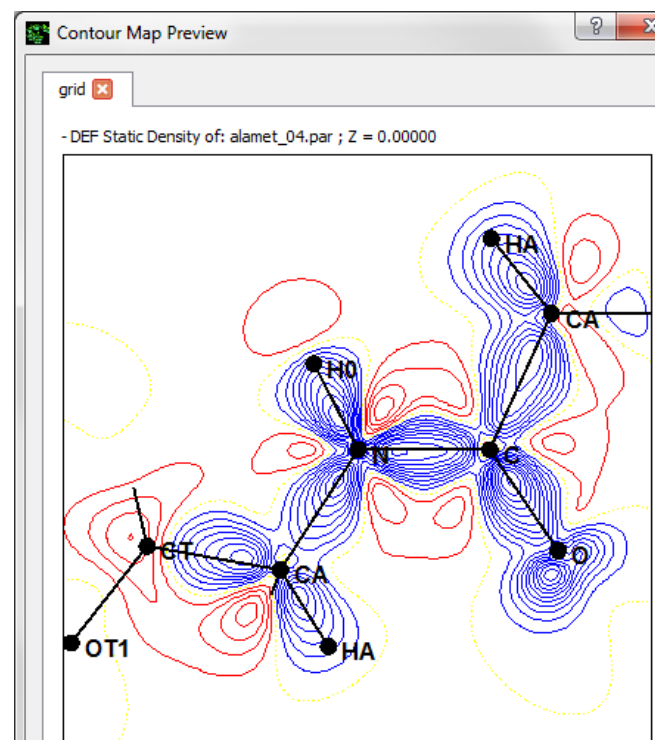
```

ATOM 1 NT ala 1 0.427429 0.050030 0.613817 1.0000 1 N
ZX CA H1 OCT K1 VO MO QO
UANI 0.013163 0.010518 0.009881 -.000244 0.005418 -.000507
5.07251 0. 0. 0. -.017 -.030 0. 0. 0.
0.173 0. 0. 0. 0. 0.128 0.
XYZ Pval =valence Plm = multipole populations
    
```

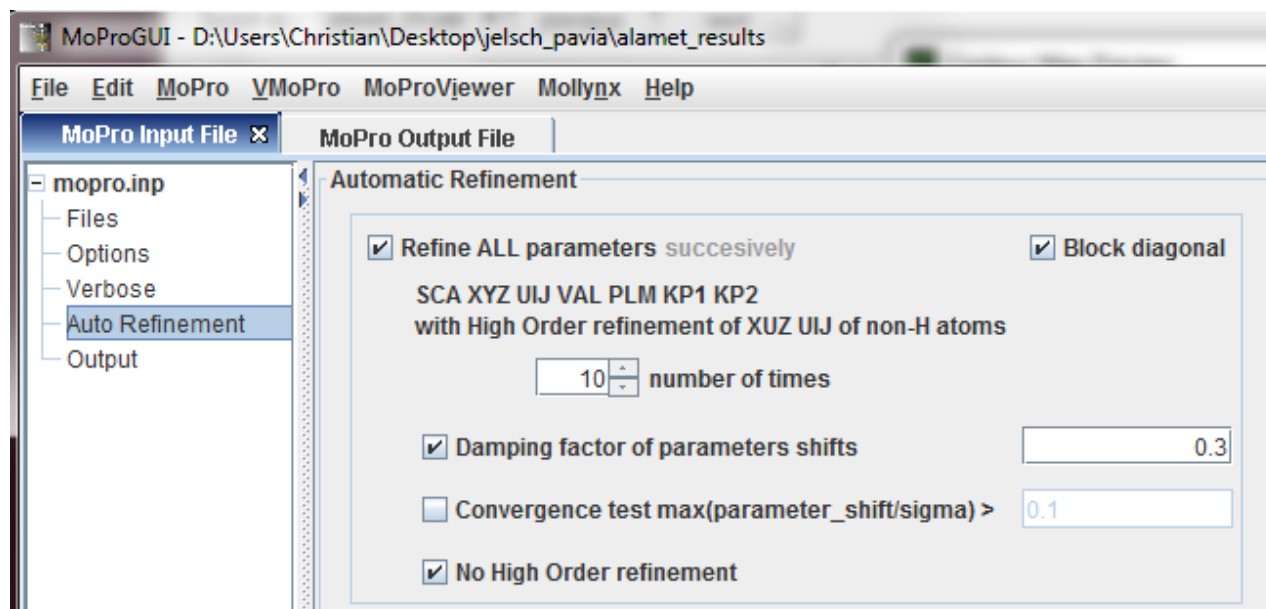
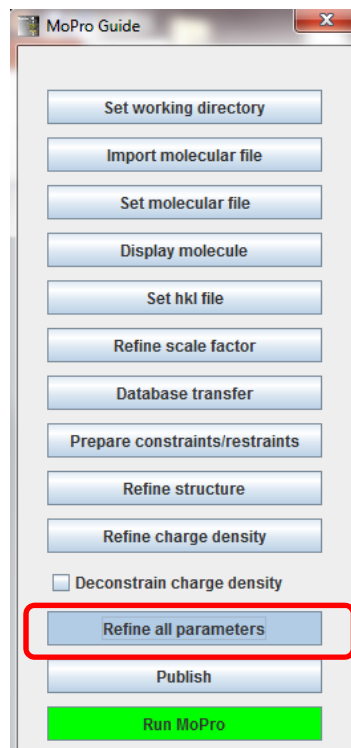
Step 13 Compute a Static Deformation Electron Density map



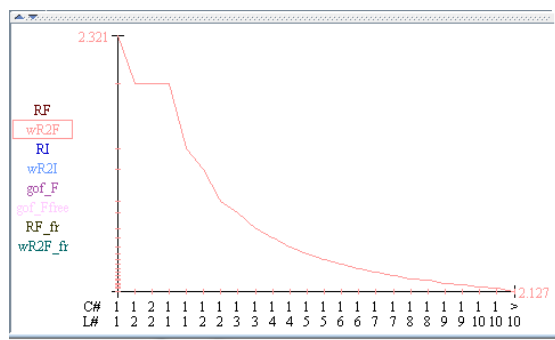
Choose appropriate molecular file



Step 14 Refinement of all parameters



- Block diagonal & damp : refine all parameters together
- Else : refine them successively

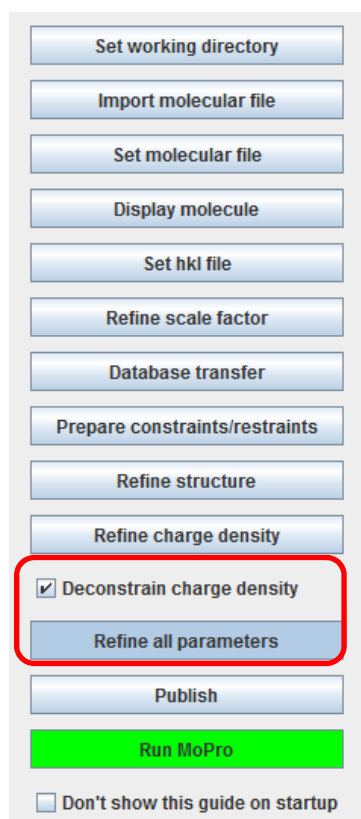


Further R-factor drop

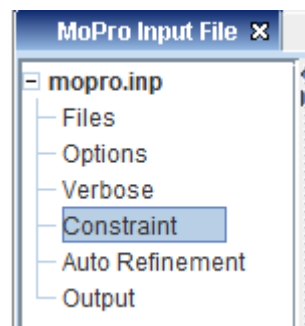
Step 15 Deconstrain charge density

Removes

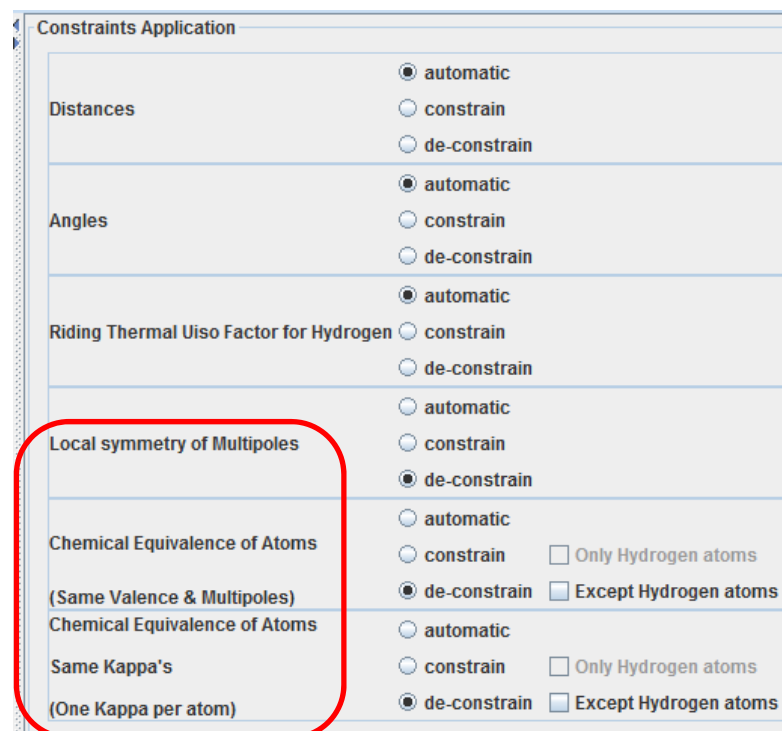
- Local symmetry of multipoles
- Atoms equivalencies : similar atoms have same charge density



A vertical stack of buttons in the MoPro main interface. The buttons are: 'Set working directory', 'Import molecular file', 'Set molecular file', 'Display molecule', 'Set hkl file', 'Refine scale factor', 'Database transfer', 'Prepare constraints/restraints', 'Refine structure', 'Refine charge density', 'Deconstrain charge density' (checked and highlighted with a red box), 'Refine all parameters', 'Publish', and a green 'Run MoPro' button. At the bottom is a checkbox 'Don't show this guide on startup'.



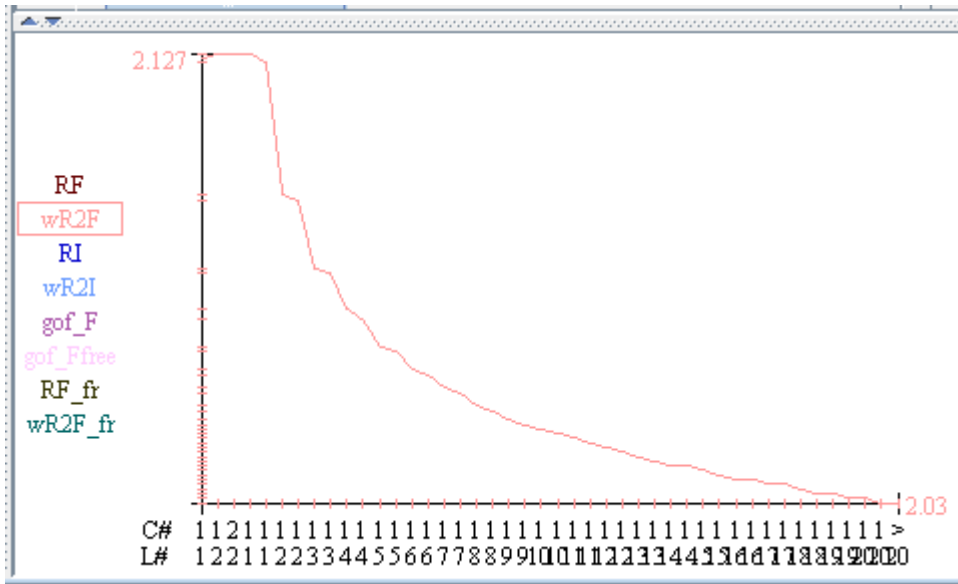
The 'MoPro Input File' menu is open, showing a tree structure. The 'Constraint' option is highlighted with a blue box. The menu items are: 'mopro.inp', 'Files', 'Options', 'Verbose', 'Constraint', 'Auto Refinement', and 'Output'.



The 'Constraints Application' dialog box is shown. It contains several sections with radio button options. The 'Local symmetry of Multipoles' section is highlighted with a red box and has 'de-constrain' selected. Other sections include 'Distances', 'Angles', 'Riding Thermal Uiso Factor for Hydrogen', 'Chemical Equivalence of Atoms (Same Valence & Multipoles)', and 'Same Kappa's (One Kappa per atom)'. Each section has 'automatic', 'constrain', and 'de-constrain' options, with 'automatic' selected for most and 'de-constrain' for the highlighted section.

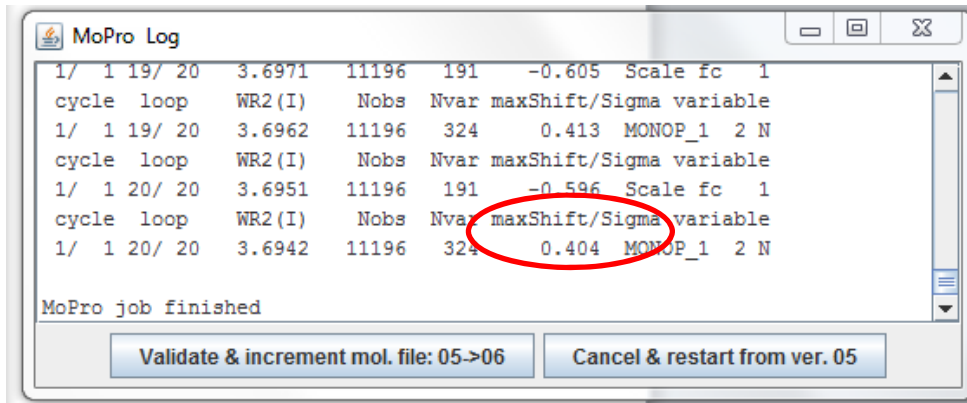
Section	automatic	constrain	de-constrain	Other options
Distances	<input checked="" type="radio"/>	<input type="radio"/>	<input type="radio"/>	
Angles	<input checked="" type="radio"/>	<input type="radio"/>	<input type="radio"/>	
Riding Thermal Uiso Factor for Hydrogen	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>	
Local symmetry of Multipoles	<input type="radio"/>	<input type="radio"/>	<input checked="" type="radio"/>	
Chemical Equivalence of Atoms (Same Valence & Multipoles)	<input type="radio"/>	<input type="radio"/>	<input checked="" type="radio"/>	<input type="checkbox"/> Only Hydrogen atoms <input type="checkbox"/> Except Hydrogen atoms
Same Kappa's (One Kappa per atom)	<input type="radio"/>	<input type="radio"/>	<input checked="" type="radio"/>	<input type="checkbox"/> Only Hydrogen atoms <input type="checkbox"/> Except Hydrogen atoms

Step 16 Refinement of all parameters till convergence

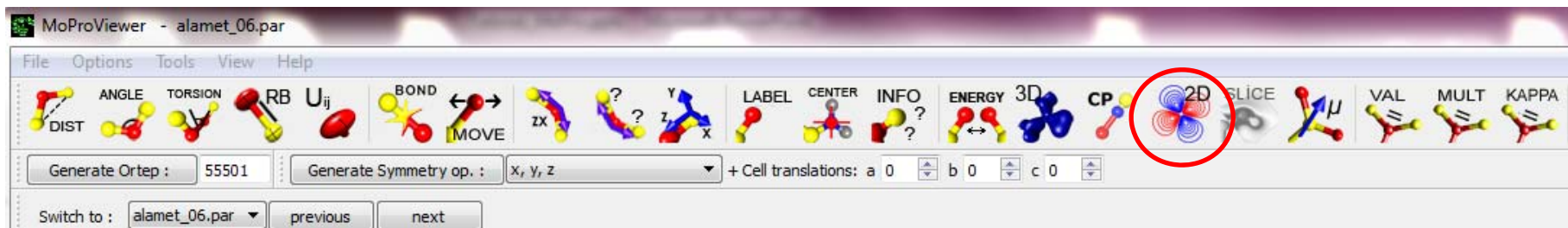


Check R-factor evolution

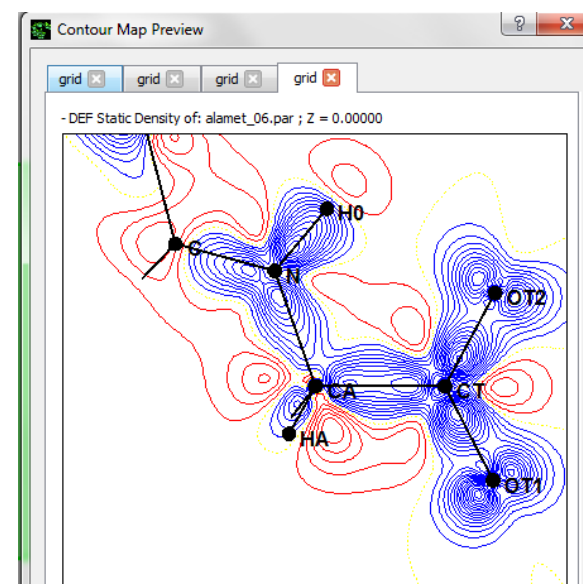
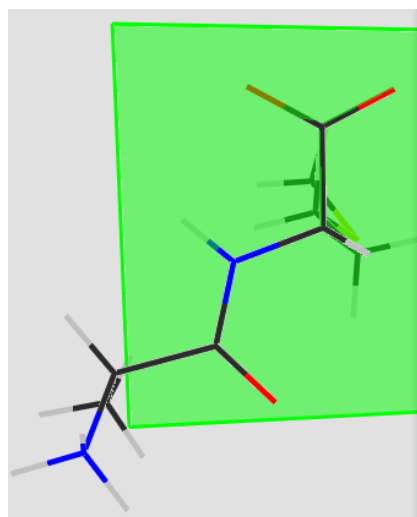
Check max Shift/sigma



Step 17 Check Static Deformation Electron Density 2D maps



Choose
Triplets of connected atoms
to define planes



Step 18 Fast Fourier Transform 3D map

MoProViewer - alamet_06.par

File Options Tools View Help

ANGLE TORSION RB U_{ij} BOND MOVE zx y z x LABEL CENTER INFO ENERGY 3D CP 2D SLICE μ VAL MULT KAPPA

Generate Ortep : 55501 Generate Symmetry op. : X, Y, Z + Cell translations: a 0 b 0 c 0

Switch to : alamet_06.par previous next

3D isosurface map

Map Properties 3D Box Parameters Surface Hirshfeld

Map

Property : Fast Fourier Transform

Of type : Static Electron density
Electrostatic Potential
Fourier Synthesis
Fast Fourier Transform
Laplacian of Electron density
Surface

Fourier Map

Use Fourier file : amet_results/alamet_06.FOUR Browse

Data are : Totally merged

Resolution range : 0.0 < $\sin(\theta)/\lambda$ < 2.0 A⁻¹

Use coefficients : 1.0 Fobs - 1.0 Fcalc

Oversampling : 3.0

Output file name : gridmap .xplor

0/119 Auto-Load map

Cancel Kill VMoPro Run

3D Maps Manager

gridmap.xplor_0

Map File : D:/Users/Christian/Desktop/jelsch_pavia/alamet_results/gridmap.xplor

REMARK -- Fourier Density (e/A³): alamet_06.FOUR of alamet_06.par

Isosurface 1

Show ☒ Isovalue : 0.244 Update

Cull back faces ☒ Two faces lightning ☐ Invert normals ☐ Filled ☒ Transp. : 0 Color

Color isosurface 1 according to : None

Limit surface ☐ print property on surface : print

Isosurface 2

Show ☒ Isovalue : -0.244 Update

Cull back faces ☒ Two faces lightning ☐ Invert normals ☐ Filled ☒ Transp. : 0 Color

Parameters

Maximum : 0.550
Minimum : -0.620
Average : 0.000
Std. dev. : 0.081
Nx, Ny, Nz : 176 74 212
Dimensions : 13.1x5.3x15.9

Extend (fractional coordinates)

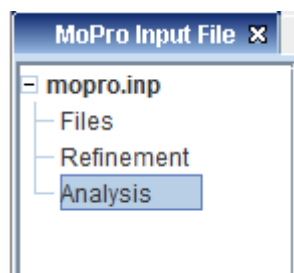
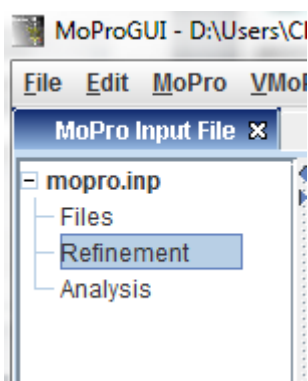
X : 0.00 - 1.00
Y : 0.00 - 1.00
Z : 0.00 - 1.00

Fit to View Use PBC Show Box

Delete Map

Residual Electron Density map in unit cell

Step 19 Stereochemical analysis



Refinement block name SET

RESOLUTION SELECTION (applies until a new selection is made)

☐ Resolution Range d(A) to ☐ Reciprocal Resolution $\sin \theta / \lambda$ (

MANUAL SELECTION OF PARAMETERS TO REFINE

☐ Use file :

SELECTION OF PARAMETERS TO REFINE

☒ Scale factors ☒ XYZ ☒ Thermal parameters

☐ Occupation Factors ☐ Anharmonicity ☐ Extinction

Valence Expansion/Contraction ☐ Spherical KP1 ☐ Multipolar KP2

☐ Valence populations Multipoles ☐ All ☐ DIP ☐ QUA ☐ OCT ☐ HEX

Analysis

Molecular Geometry

☒ Distances

☐ Angles

☐ Dihedral angles

☐ Plane

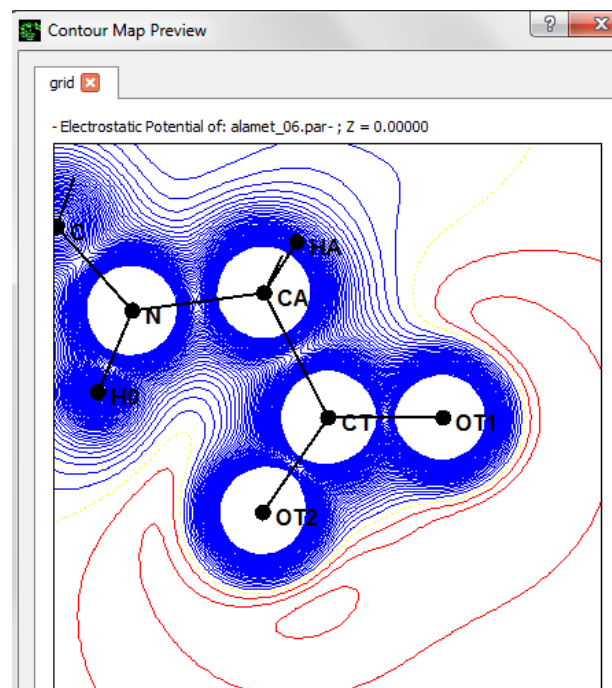
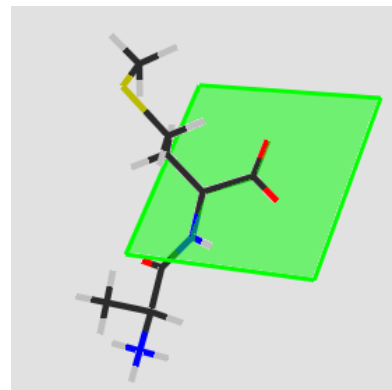
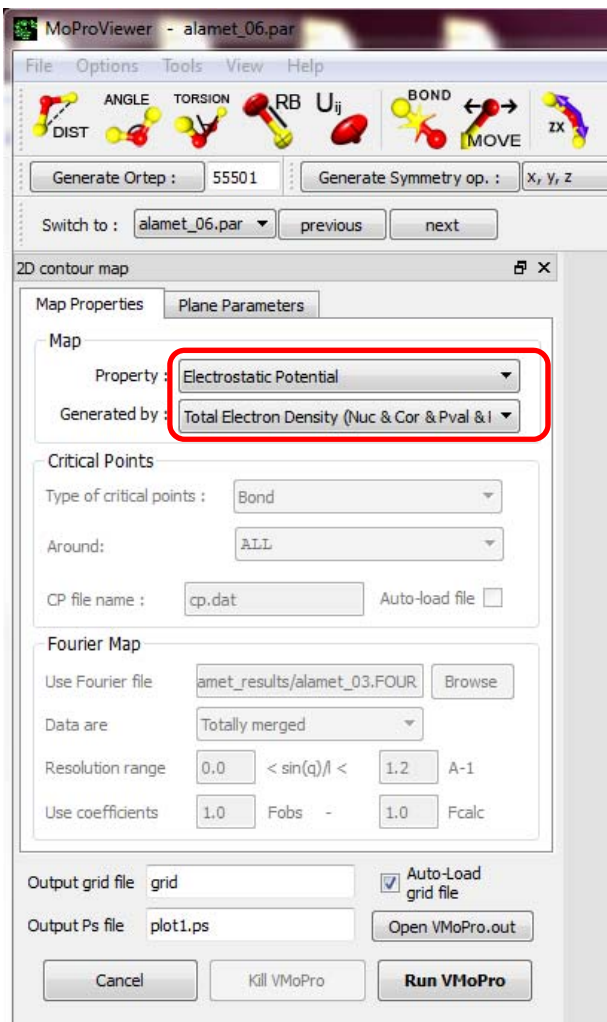
☐ Chiral volumes

Molecular Connectivity

Refine
at first
structure
to obtain
sigmas
of distances

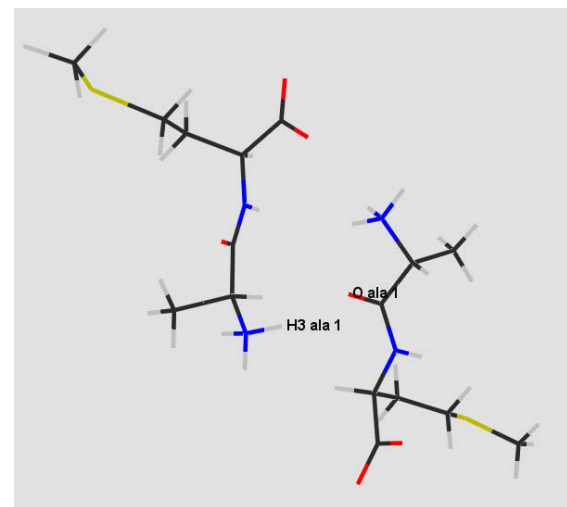
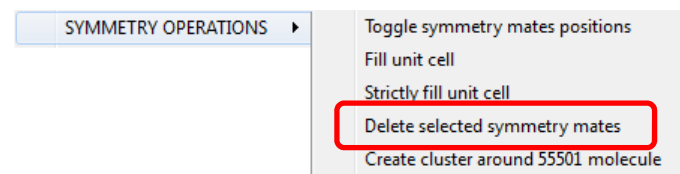
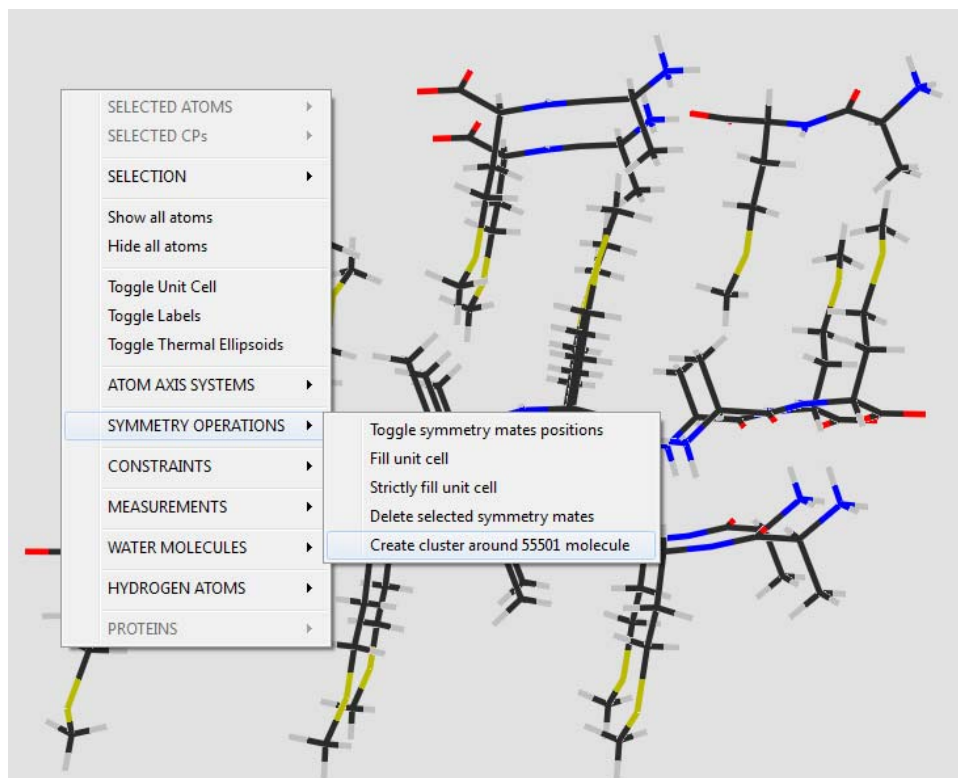
										refined		
ATOM1				ATOM2				SYM2	DIST	sigDIST	xyz	xyz
NT	ala	1	--	CA	ala	1			1.481091	0.001594	+++	+++
NT	ala	1	--	H2	ala	1			1.030136	0.001247	+++	+++
NT	ala	1	--	H3	ala	1			1.034194	0.002353	+++	+++
NT	ala	1	--	H1	ala	1			1.036034	0.001297	+++	+++
CA	ala	1	--	C	ala	1			1.525269	0.000535	+++	+++

Step 20 2D map of Electrostatic Potential



Step 21 Generate a dimer in MoProViewer

Click right on view

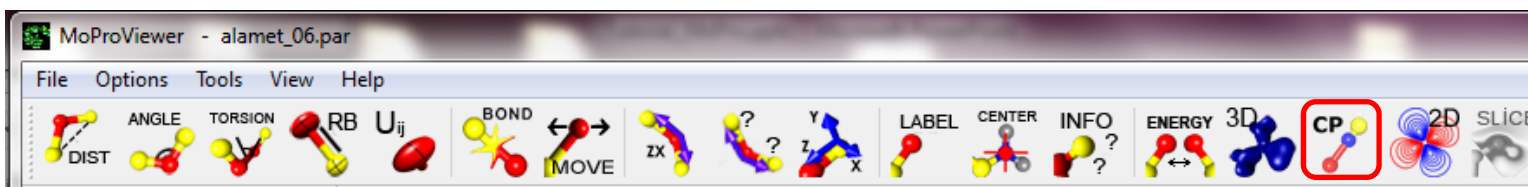


Keep a dimer like 55501 + 65602

Ortep symmetry code
65602

Translation $1\mathbf{a} + 0\mathbf{b} + 1\mathbf{c}$ & symmetry #02
 $6 \rightarrow 1 \quad 5 \rightarrow 0 \quad 4 \rightarrow -1$

Step 22 Search Intermolecular critical points



Critical Points Search

VMoPro MoProViewer

Critical Points Search using VMoPro

Property type

Property : Static Electron density

Of type : Total electron density (Cor & Pval & P00 & Plm)

Contributing symmetry operations

Use all currently active symmetric molecules

Add / Del : 55501 Generate Molecules

55501

Search options

Type of critical points : Intermolecular

H3

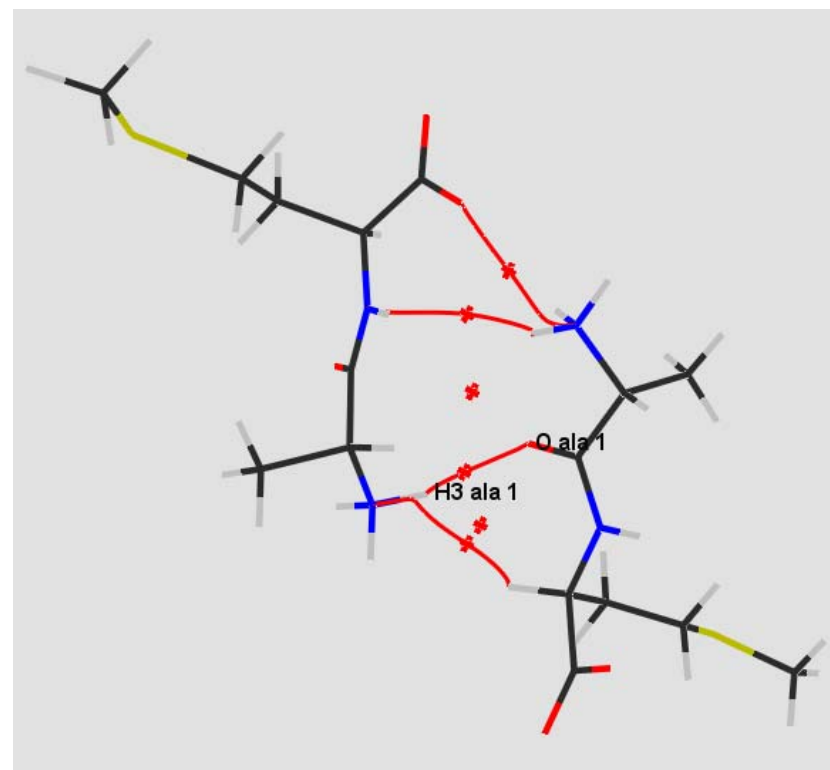
Search options

Number of iterations: 900

Output file name: cp.dat Auto-load ☒

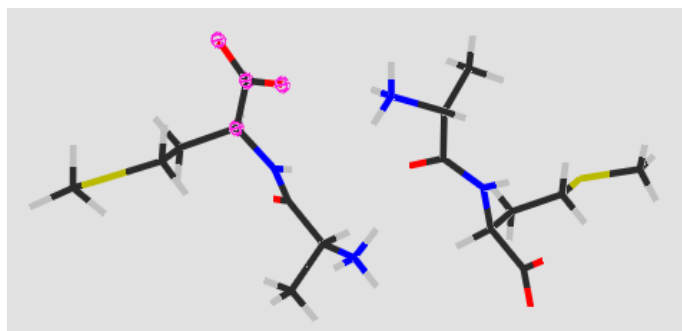
Cancel Kill VMoPro Run VMoPro

Search
around
atom
H3 only
(fast
calculation)



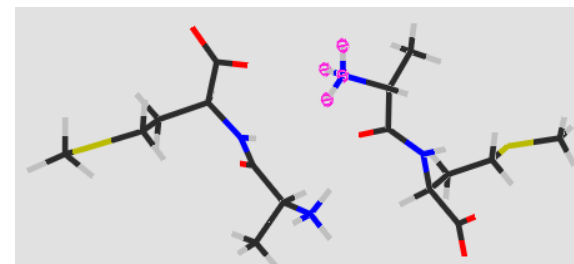
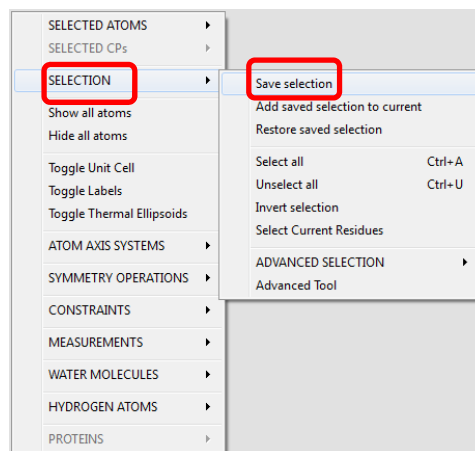
MoProViewer shows CPs & bond paths

Step 23 Compute electrostatic interaction energy of dimer

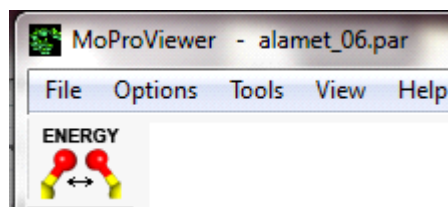


1) Select the carboxylate group

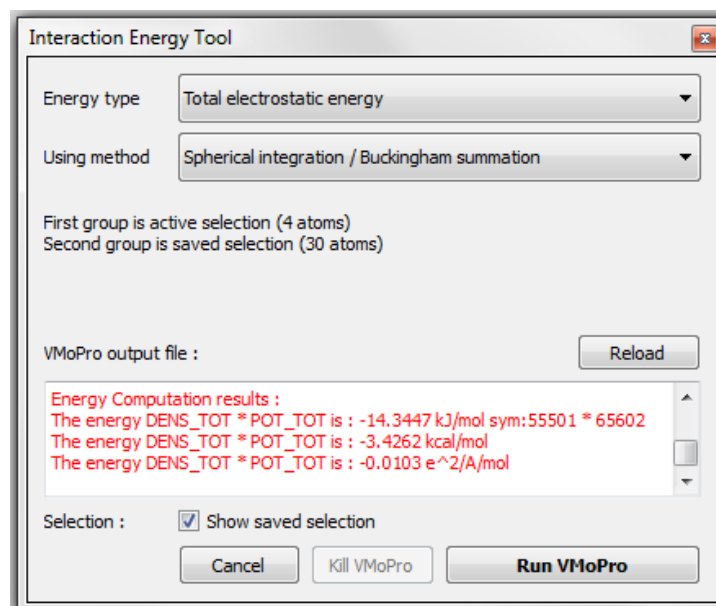
Right click, Selection / SAVE selection



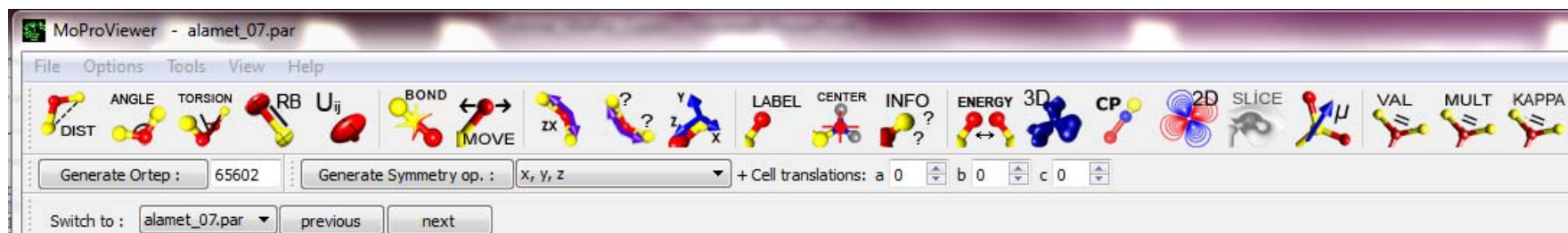
2) Select the NH3 group
on the other molecule



3) Click on **Energy** tool
of MoProViewer



Step 24 Discover the Tools of MoProViewer



Stereo-chemistry

Move atoms

Show local axes system for multipoles orientation

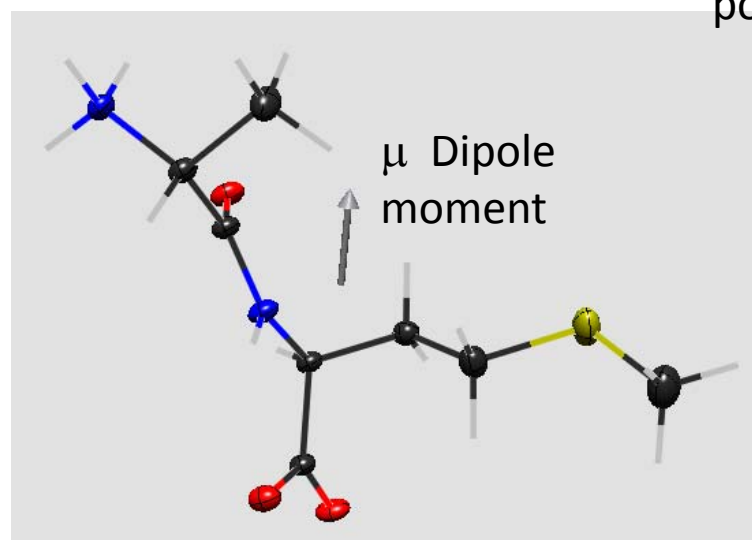
Compute 3D map

Compute 2D map

Critical points

Equivalence Constraints of atomic charge density

U_{ij}
Thermal ellipsoids



Step 25 Discover the MoPro Menus

