

Tutorial

MoProSuite : Crystallographic software for charge density refinement

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

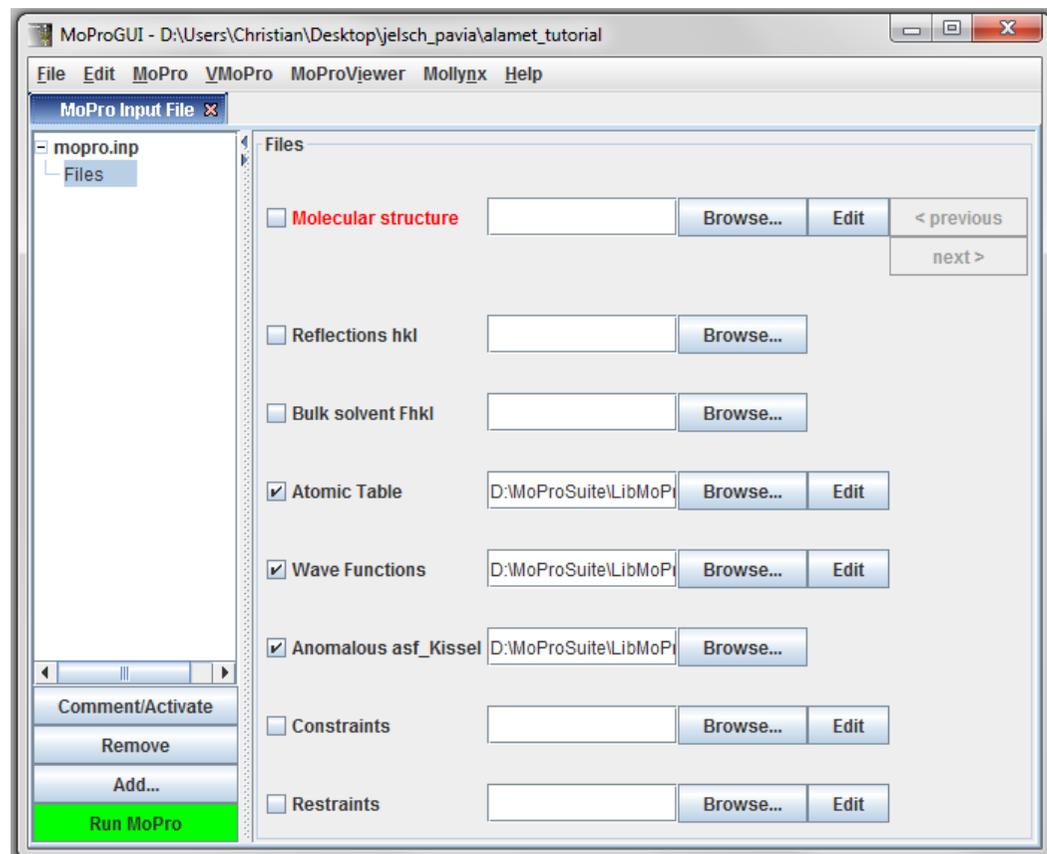
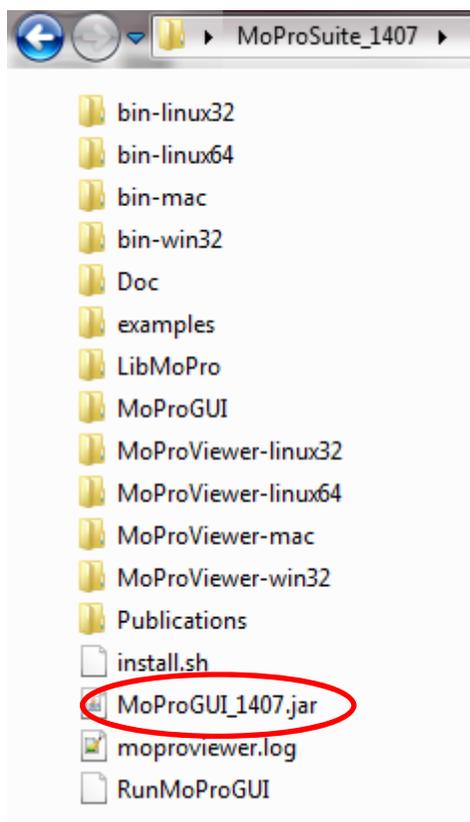
Christian Jelsch. CNRS. Université de Lorraine. France;

Available on windows, linux & mac.

July 2014

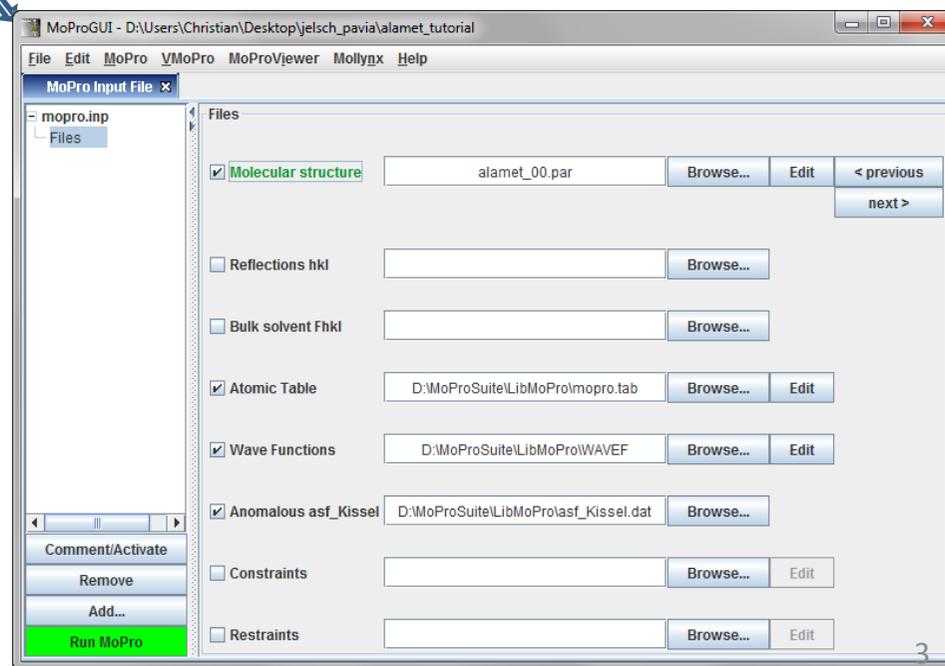
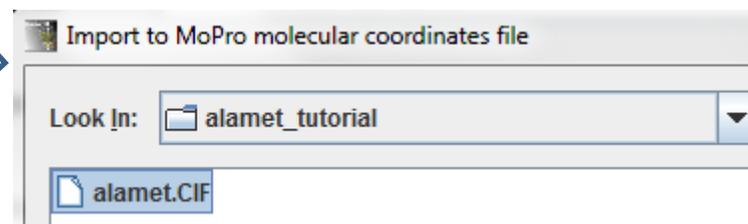
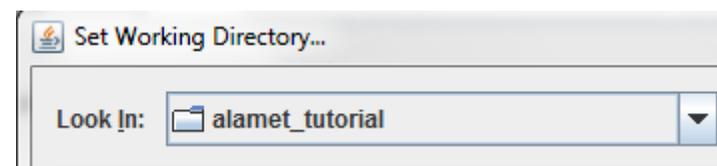
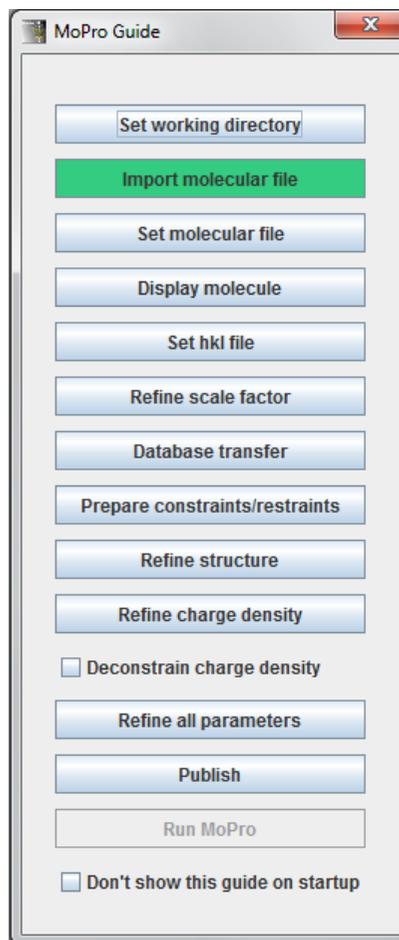
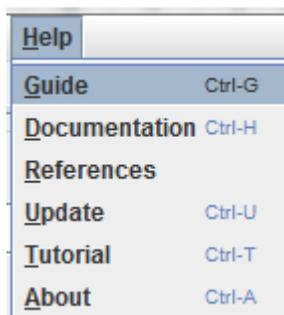
Step 1 Lauch MoProGUI

MoPro Graphical User Interface

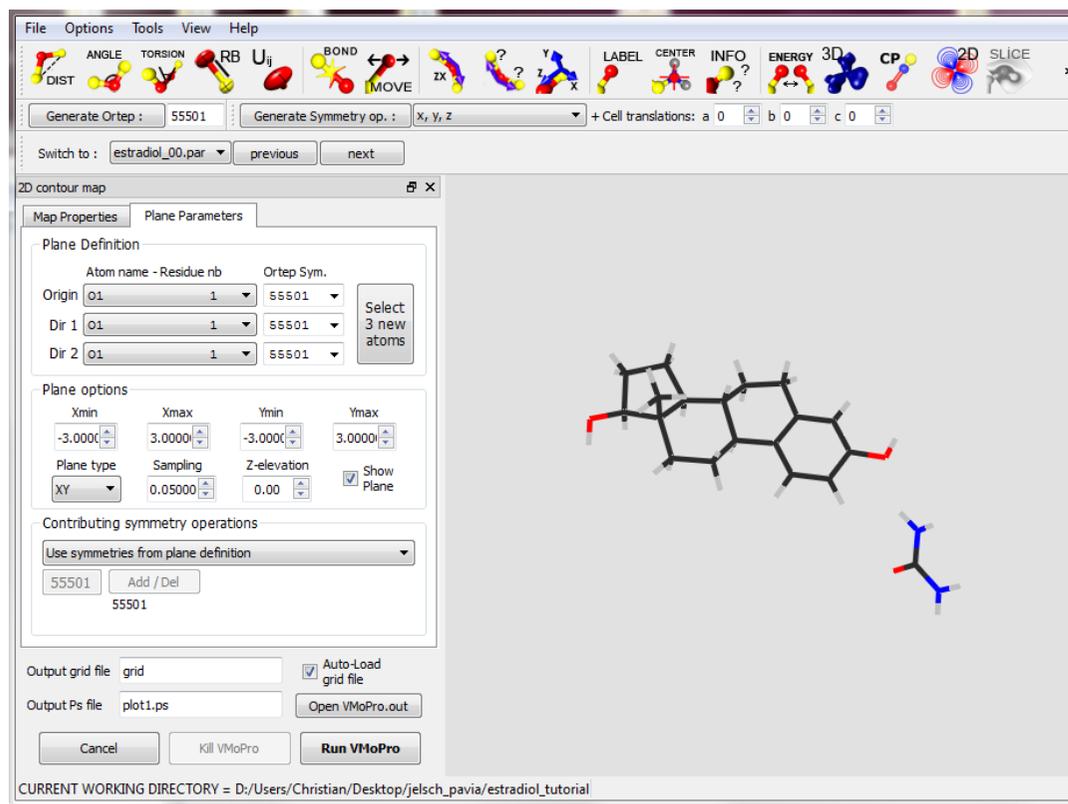
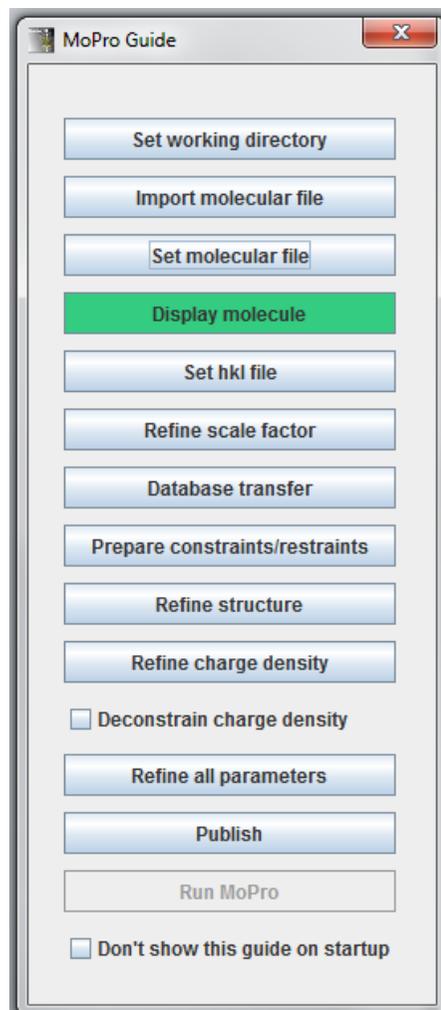


Requires JAVA, which can be downloaded at:
<https://www.java.com/fr/download/>

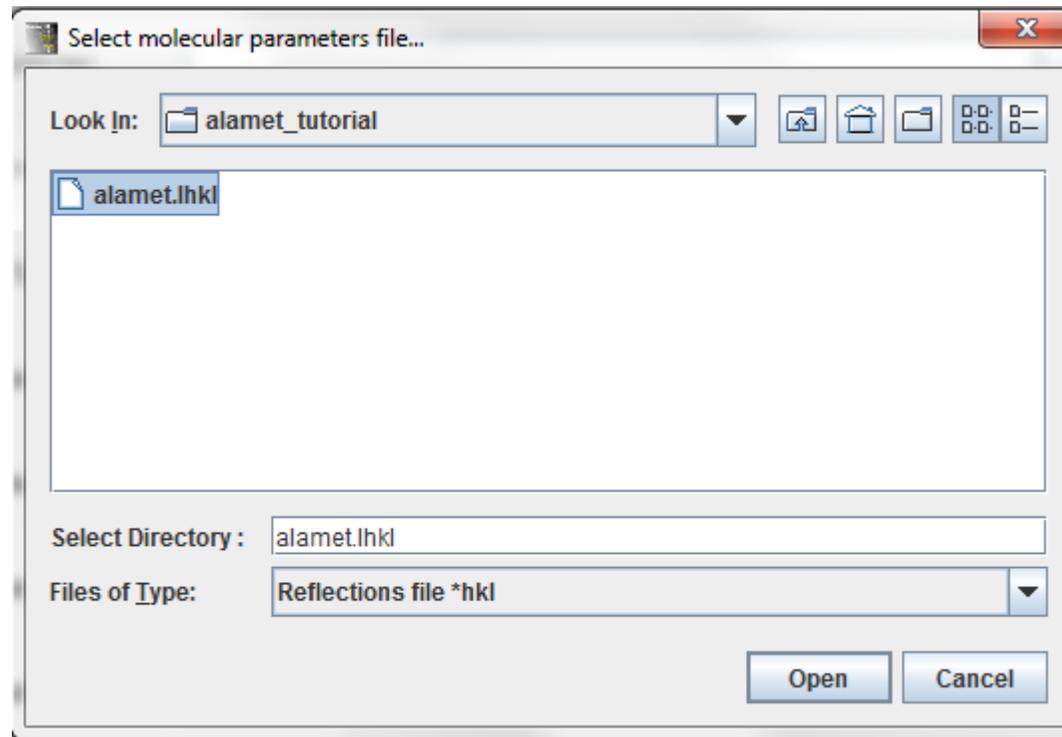
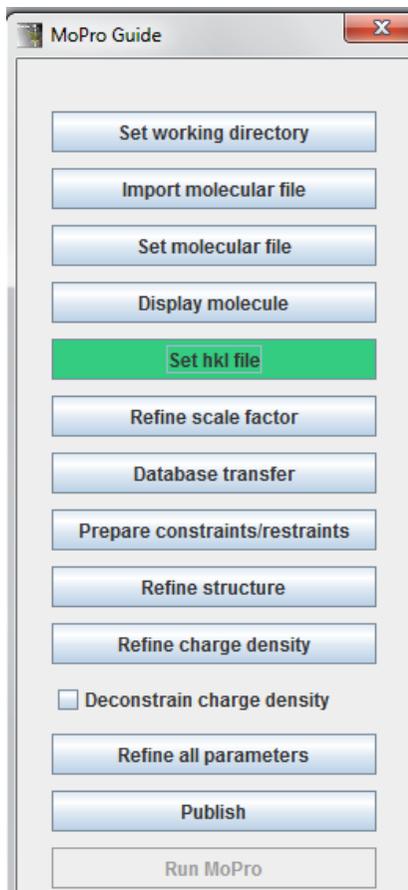
Step 2 initial steps : follow the GUIDE



Step 3 Display molecule with MoProViewer



Step 4 Select reflections file



h k l lobs sigma(lobs)

Step 5 Refine SCALE factor

MoPro Guide

- 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
- Refine all parameters
- Publish
- Run MoPro

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

MoProGUI - D:\Users\Christian\Desktop\jelsch_pavia\estradiol_tutorial

File Edit MoPro VMoPro MoProViewer Mollynx Help

MoPro Input File

Refinement block name
scale SET

RESOLUTION SELECTION (applies until a new selection is made)
 Resolution Range d(A) 0.25 to 300
 Reciprocal Resolution sin θ / λ (A⁻¹) 0.00 to 2.0

MANUAL SELECTION OF PARAMETERS TO REFINE
 Use file: Browse... Edit...

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

SELECTION OF CONCERNED ATOMS

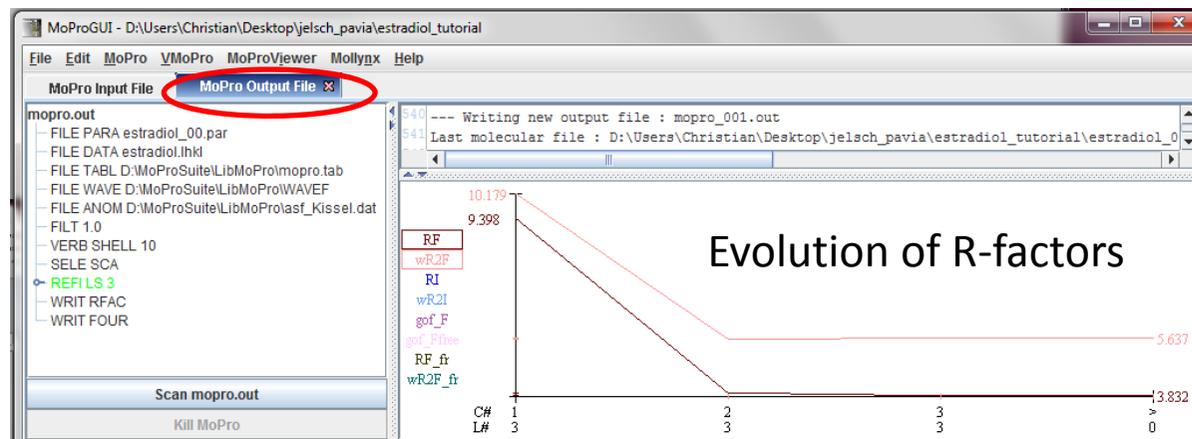
Water Isotropic only Anisotropic only
 only exclude
Hydrogen only exclude
Chemical type only exclude
Disorder only exclude
Limitation on thermal B-Factor Beq > 0.0 Å² Beq < 0.0 Å²
Manual Selection

Combination of selections + +

REFINEMENT OPTIONS

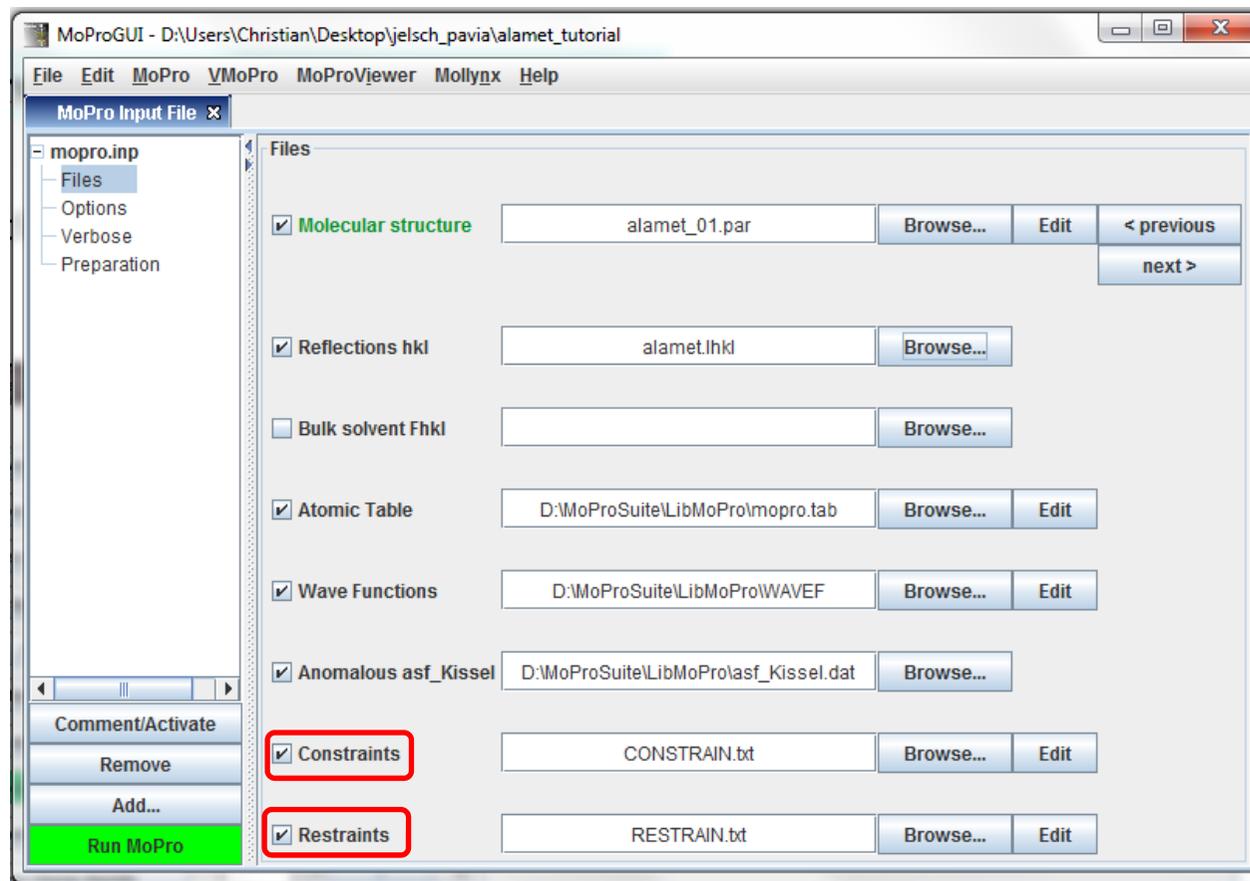
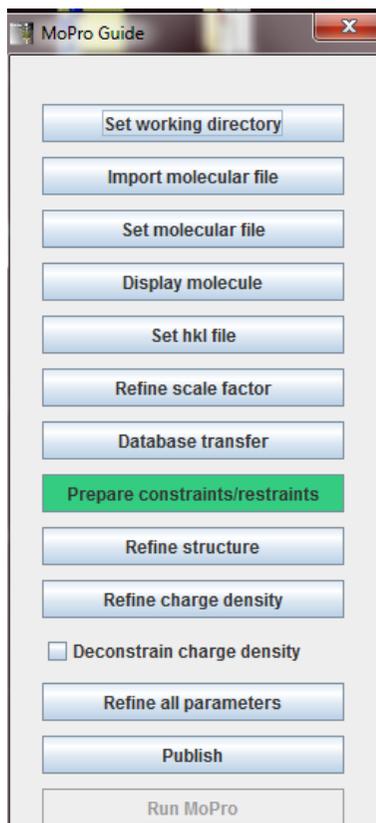
Full Matrix Inversion
 Conjugate gradients Sparse Matrix Distance cutoff(A) 5 Diagonal Matrix Block diagonal

Number of refinement cycles 2 Damping factor of parameters shifts 0.8
 convergence test max(parameter_shift/sigma) > 0.1
 Compute R-factors Statistics



Step 6 Preparation of constraints & restraints

e.g. H-X distances for Hydrogen atoms

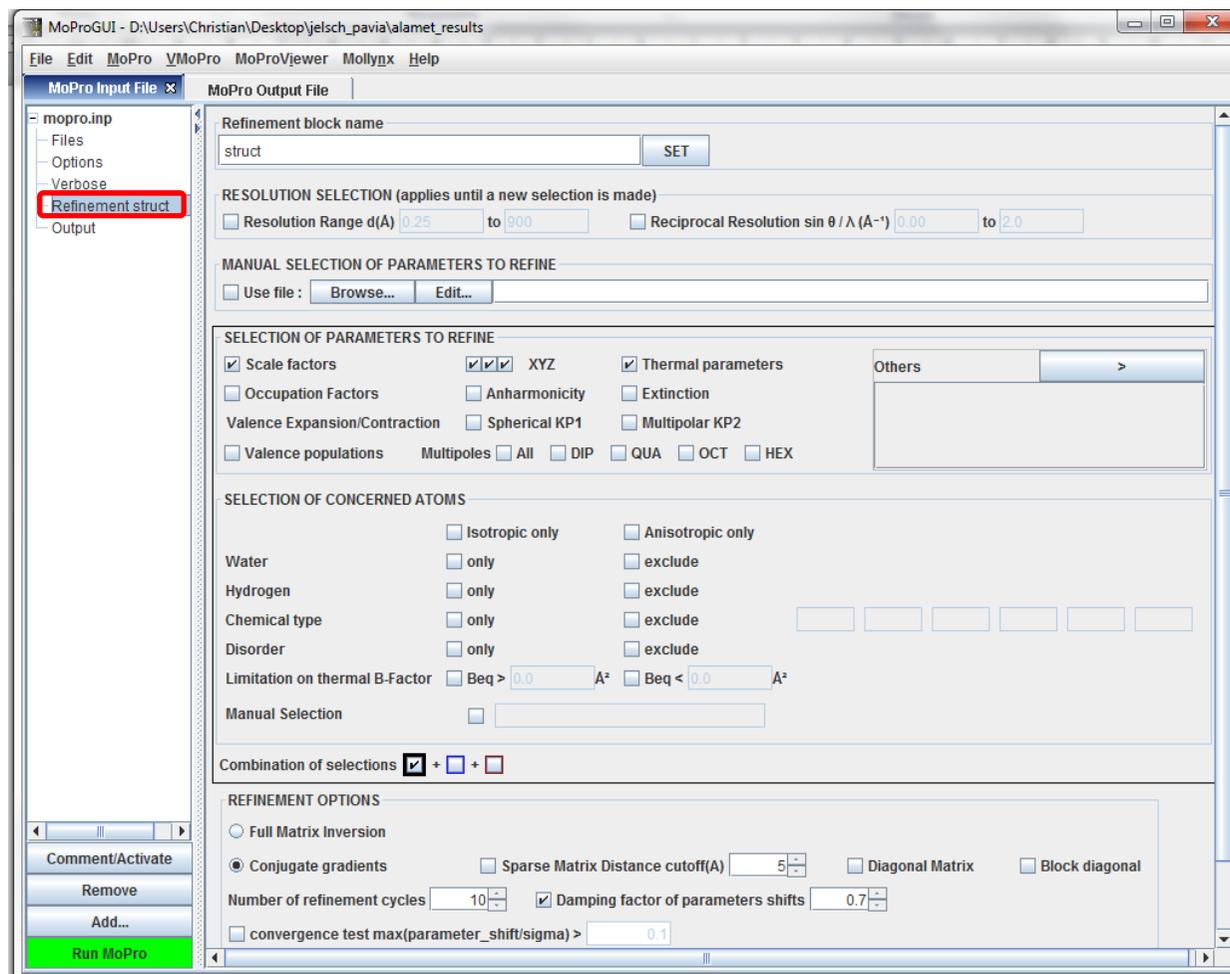
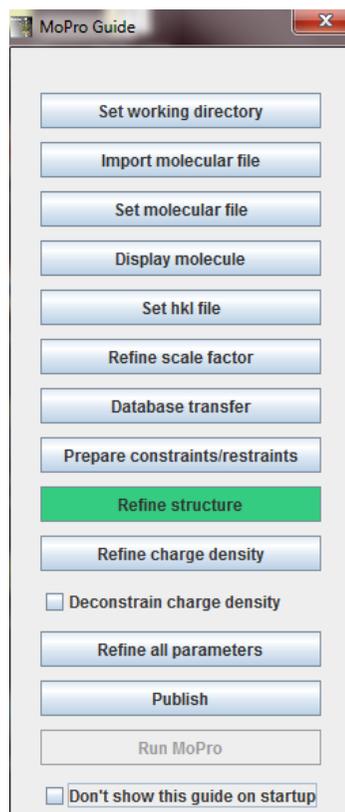


Edit and have a look at the generated
CONSTRAIN.txt & RESTRAIN.txt files

Step 7 Refine structure

SCA XYZ UIJ *are selected*

SCAle factor, Positions & Thermal motion parameters



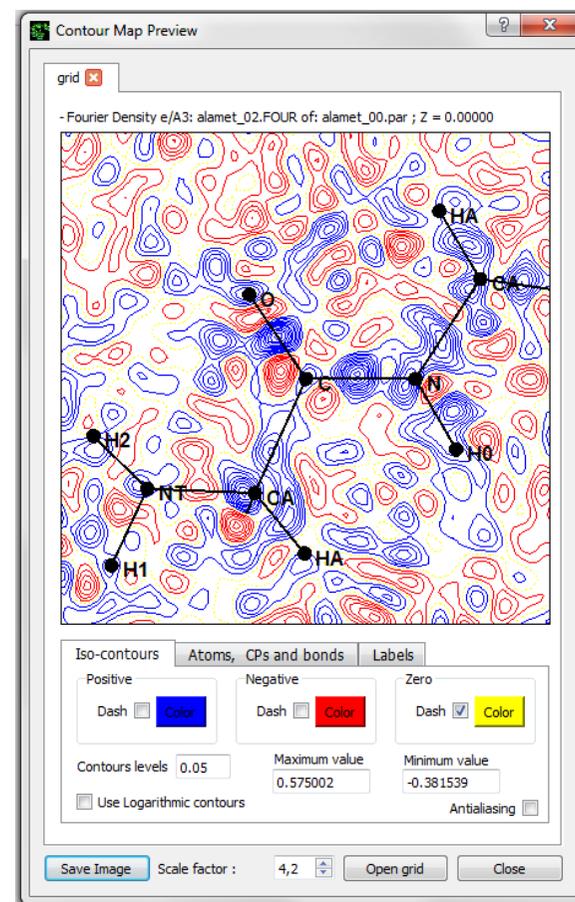
Step 8 Compute a Fourier Residual Electron Density 2D map

The screenshot shows the MoProViewer interface. The '2D contour map' panel is active, with the following settings:

- Map Properties: Property: **Fourier Synthesis** (circled in red), Of type: Deformation (full deformation)
- Critical Points: Type of critical points: Bond, Around: ALL
- Fourier Map: Use Fourier file: **amet_results/alamet_02.FOUR** (circled in red), Data are: **Totally merged** (circled in red)
- Resolution range: 0.0 < $\sin(\theta)/\lambda$ < 2.0 A-1
- Use coefficients: 1.0 Fobs, 1.0 Fcalc
- Output grid file: grid, Auto-Load grid file:
- Output Ps file: plot1.ps, Open VMoPro.out
- Buttons: Cancel, Kill VMoPro, **Run VMoPro** (circled in red)

The main window displays a 3D ball-and-stick model of a molecule with a green plane overlaid on it, indicating the plane for the 2D map calculation.

Click on
3 atoms
to define
a plane



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

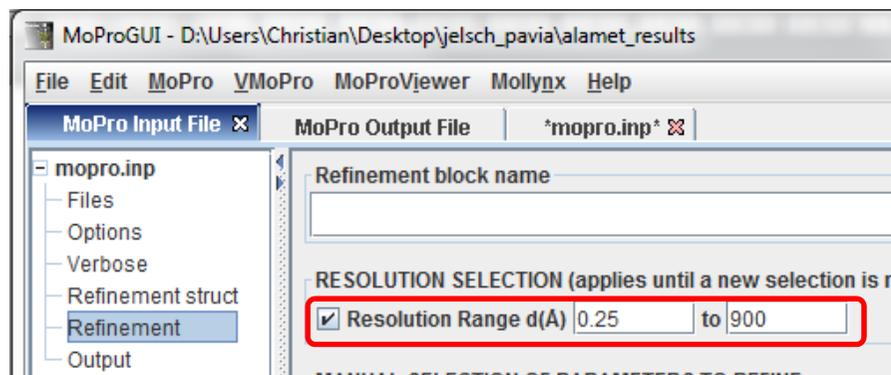
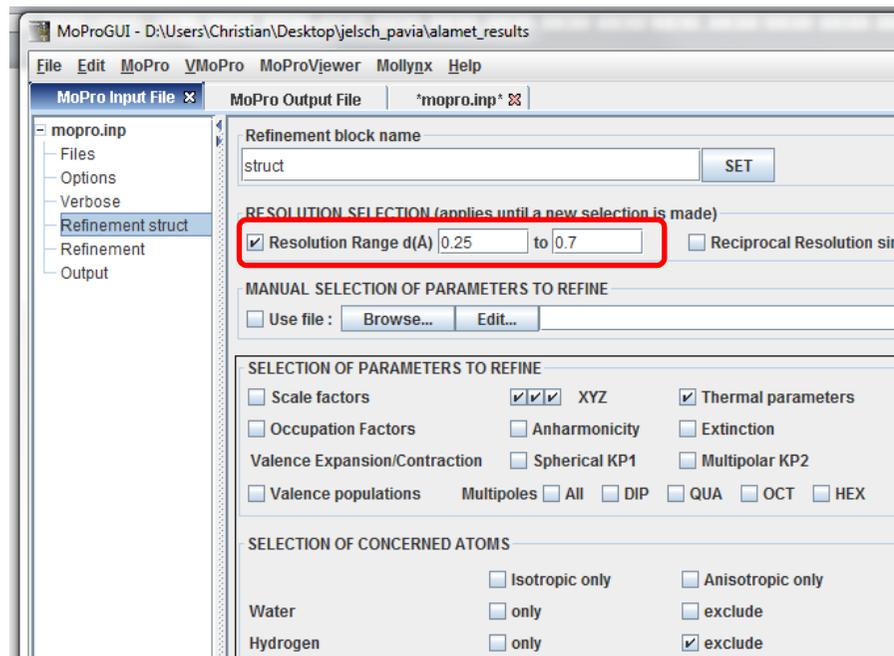
High resolution data $d < 0.7 \text{ \AA}$,
Hydrogen not refined

The MoPro commands

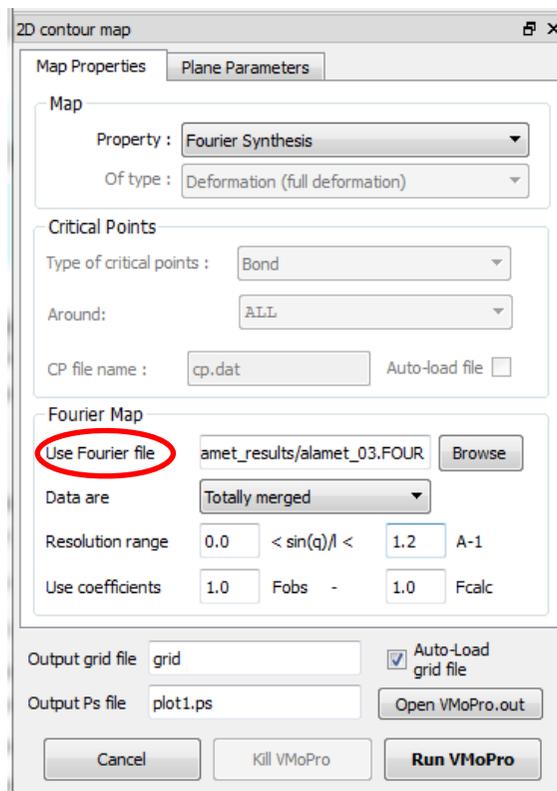
```
! <refinement> structural  
RESO 0.25 0.7  
SELE XYZ UIJ NOH  
REFI CG 10 DAMP 0.7  
WRIT RFAC
```

```
RESO 0.25 900  
WRIT FOUR
```

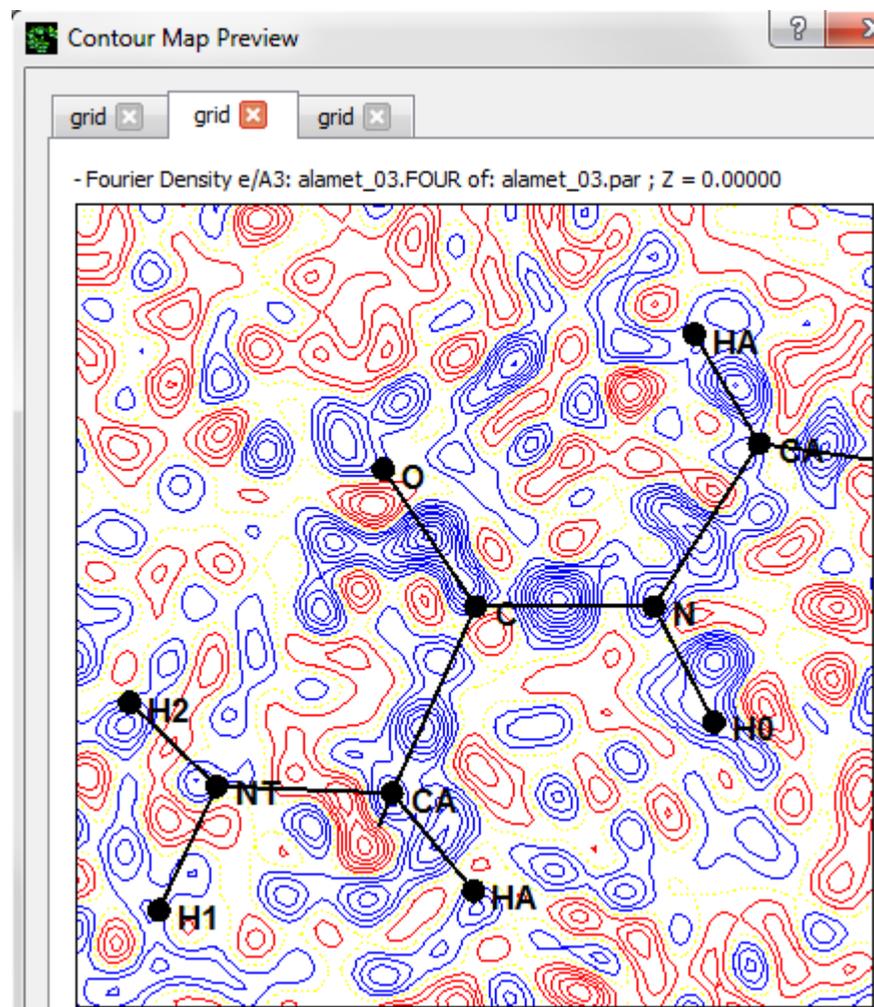
Write a Fourier reflections file
for all resolution



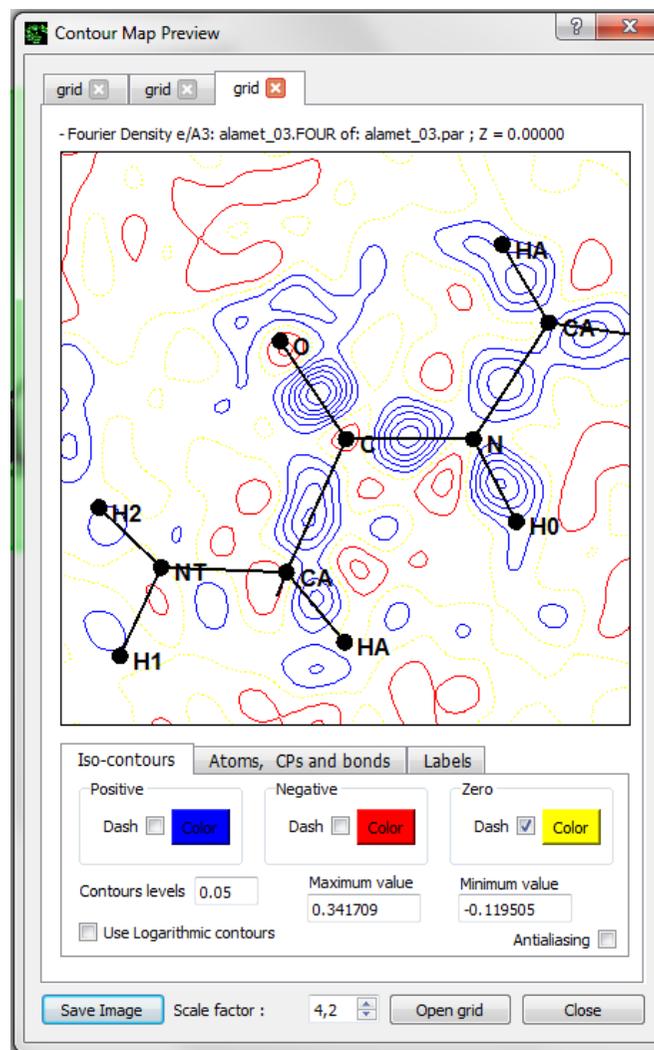
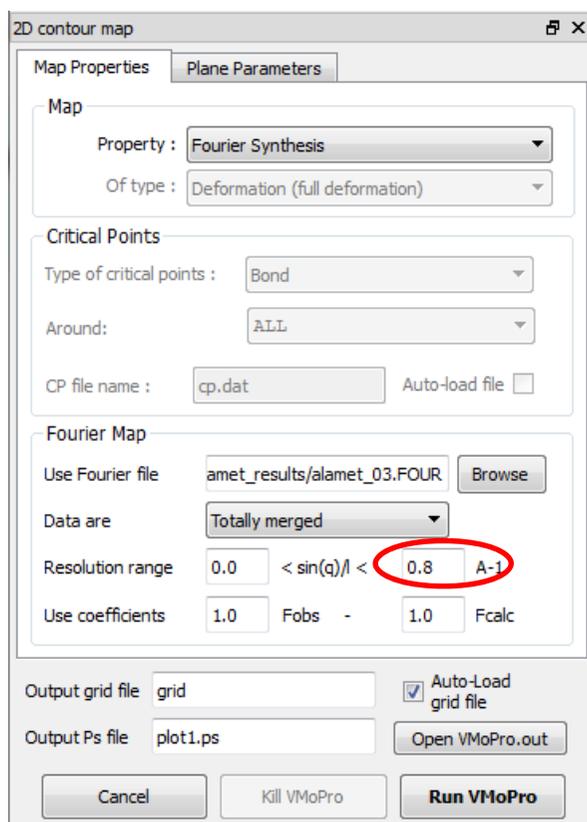
Step 10 Compute a Fourier Residual Electron Density after High-Order refinement



Stronger residual electron density visible on covalent bonds



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

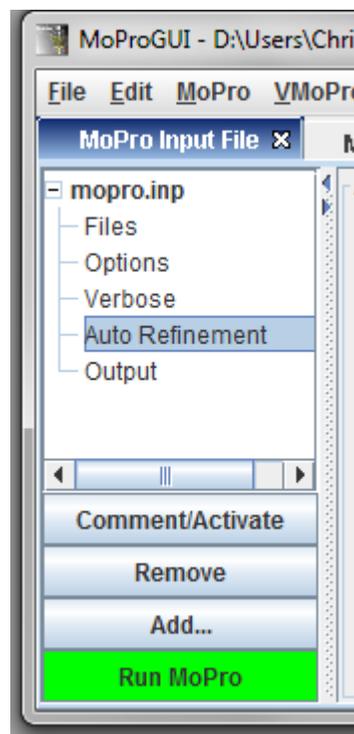
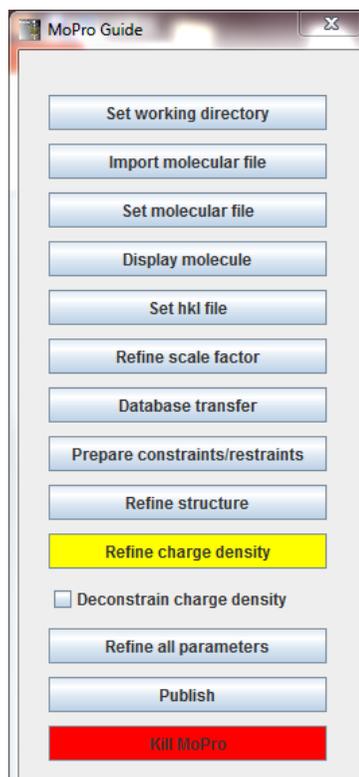


$$\sin \theta / \lambda < 0.8 \text{ \AA}^{-1}$$

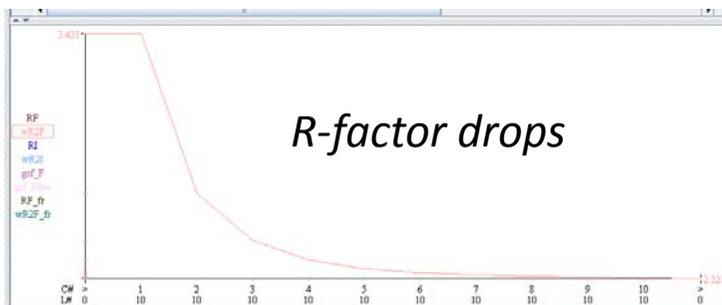
resolution
 $d > 0.6 \text{ \AA}$

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



R-factor drops

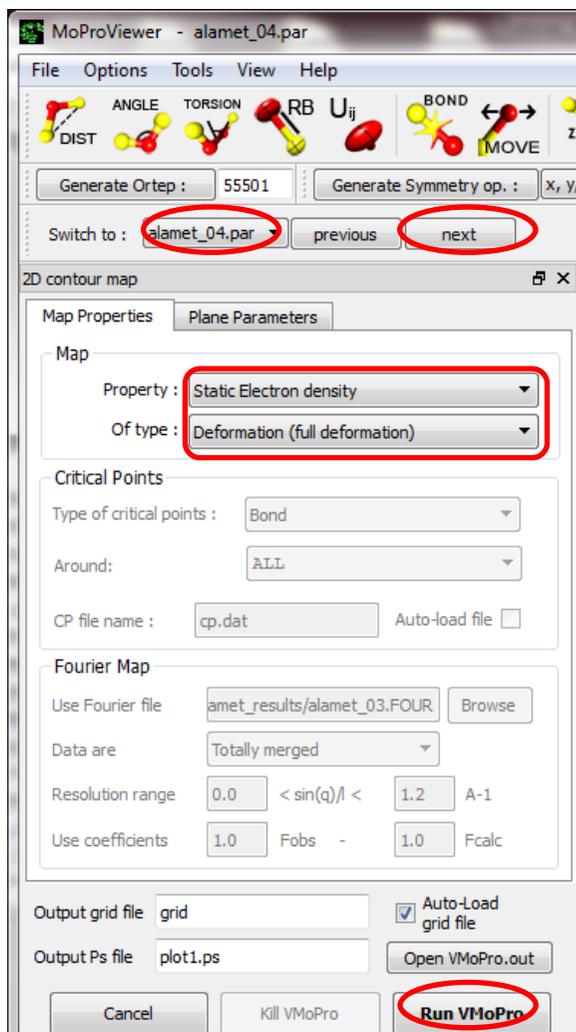
Have a look at resulting molecular .par file

ATOMS 30

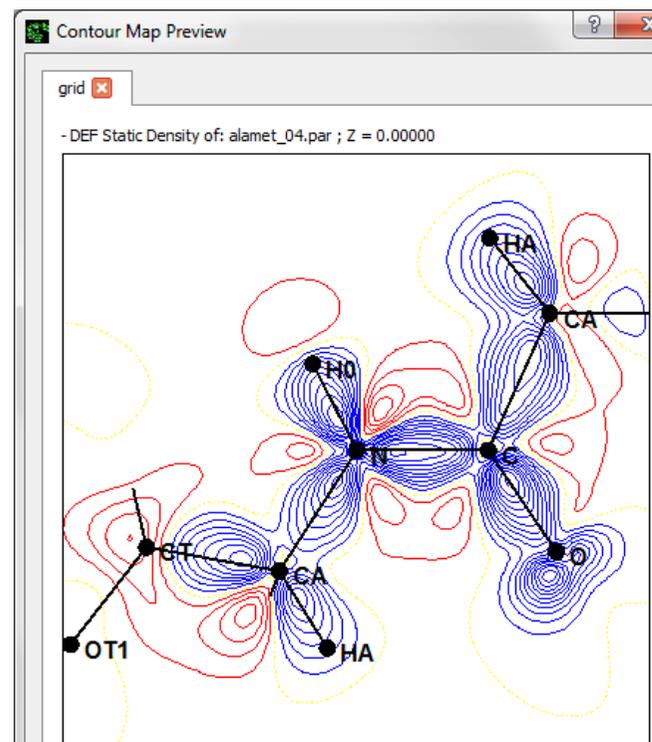
```

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.
0.173 0. 0. 0. 0. 0.128 0.
XYZ Pval =valence Plm = multiple populations
    
```

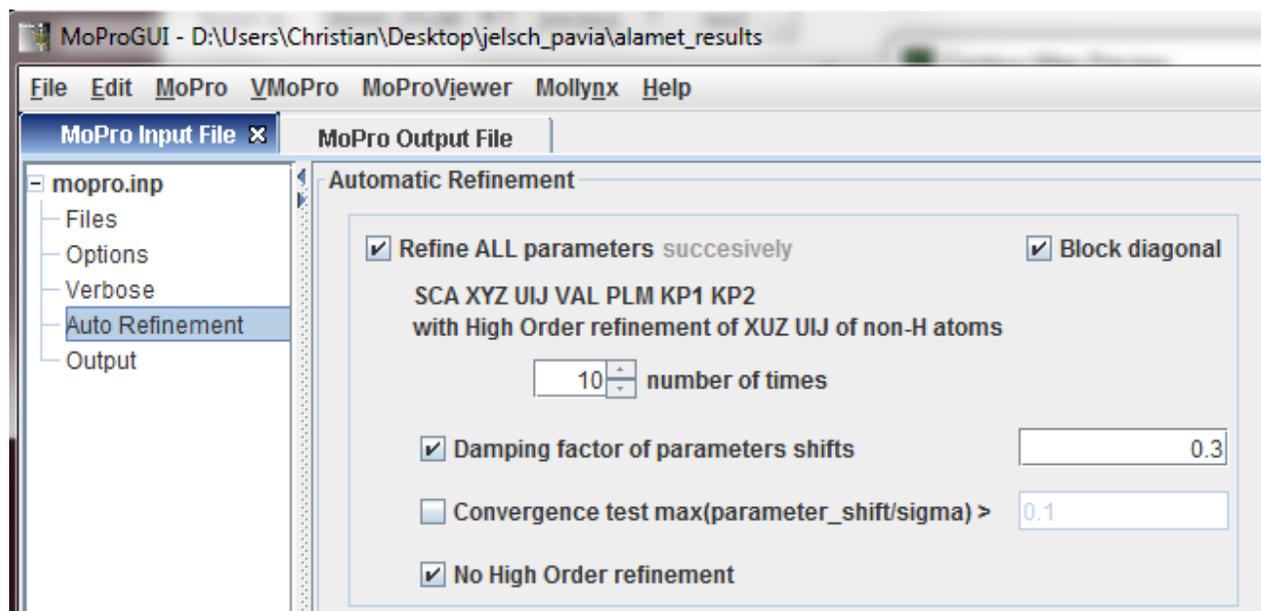
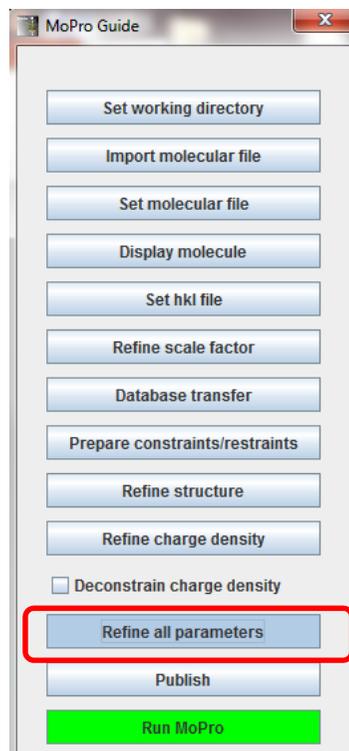
Step 13 Compute a Static Deformation Electron Density map



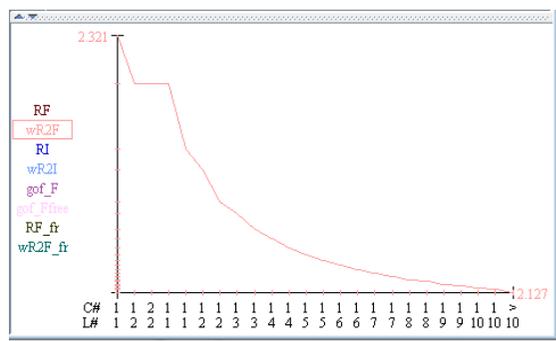
Choose appropriate molecular file



Step 14 Refinement of all parameters



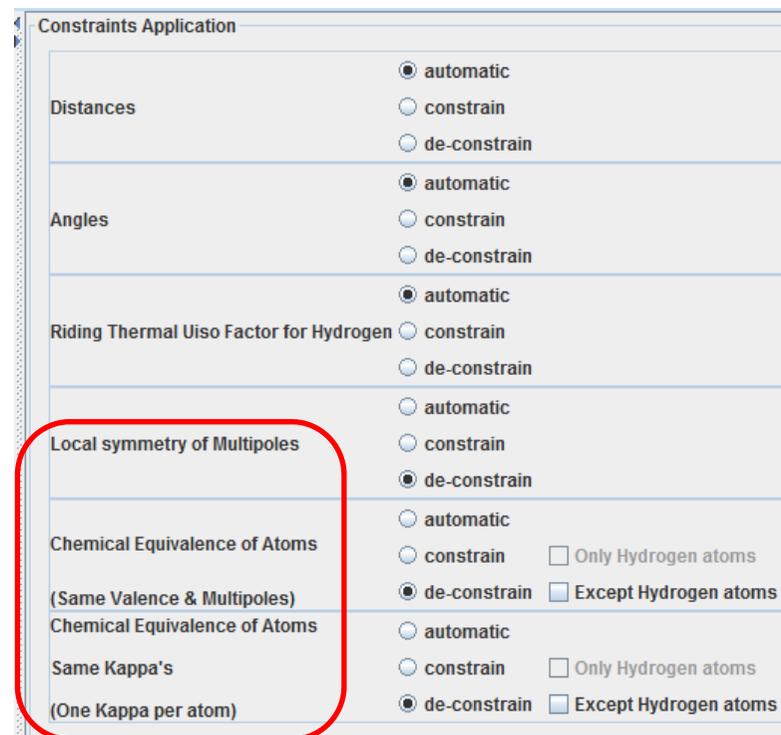
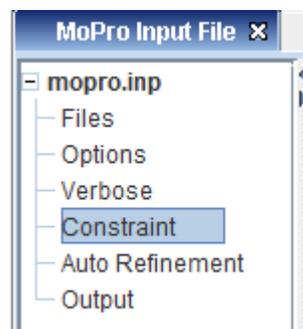
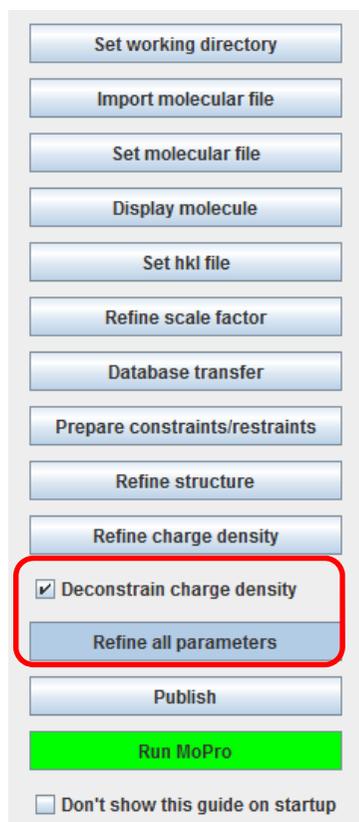
- Block diagonal & damp : refine all parameters together
- Else : refine them successively



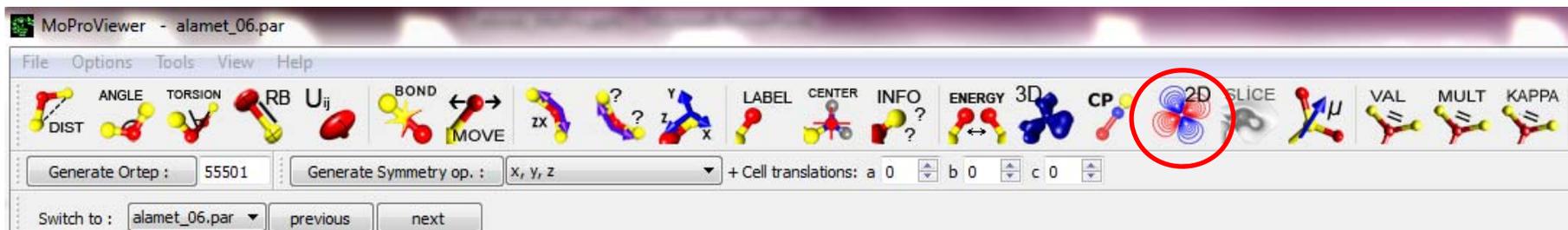
Step 15 Deconstrain charge density

Removes

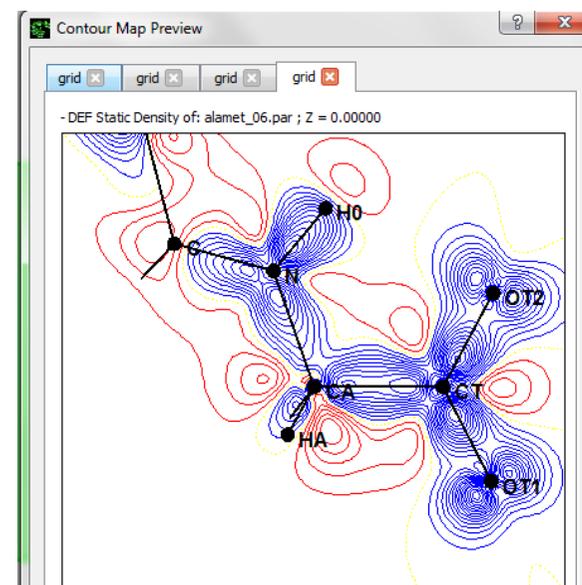
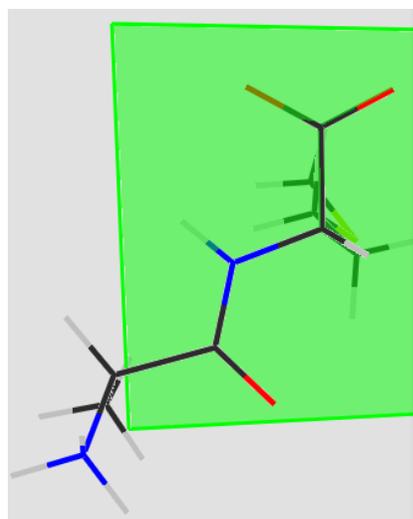
- Local symmetry of multipoles
- Atoms equivalencies : similar atoms have same charge density



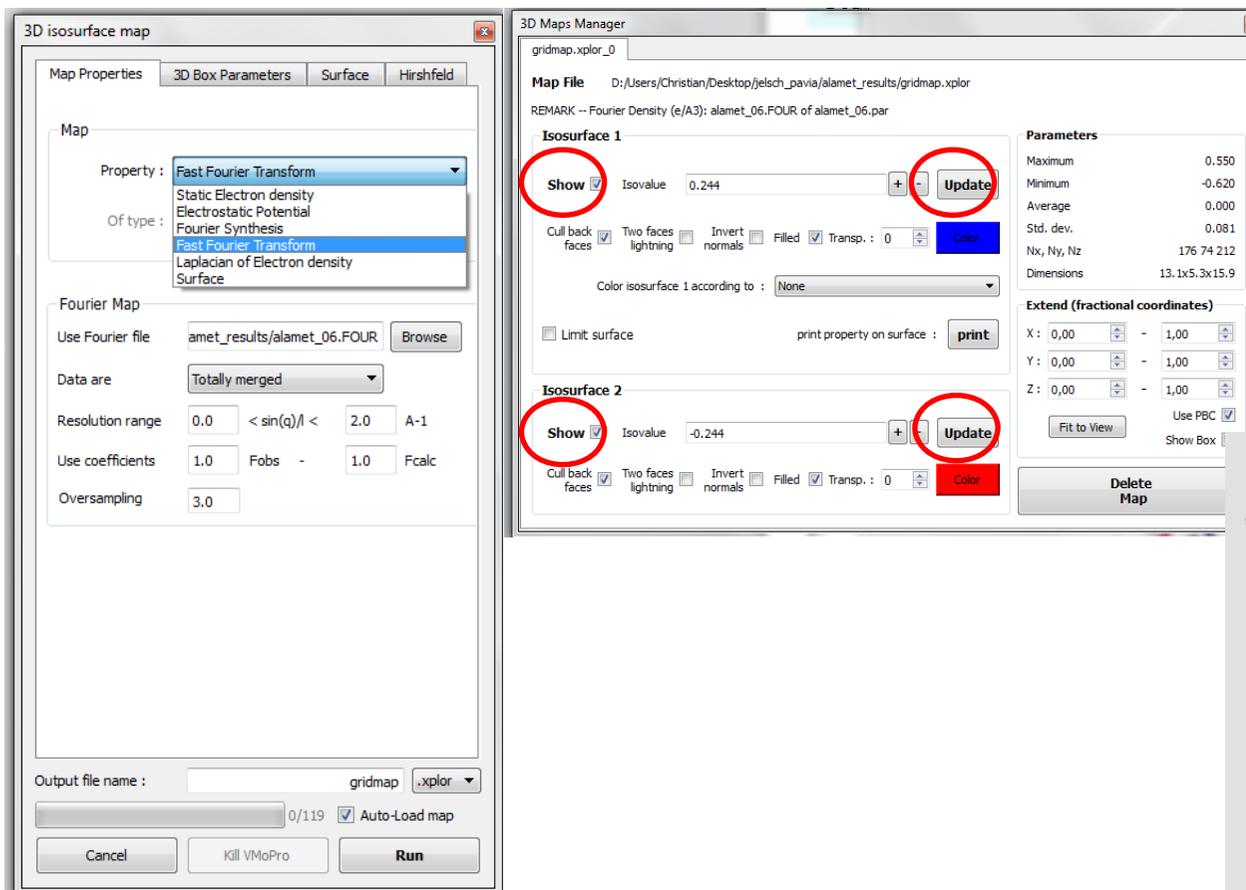
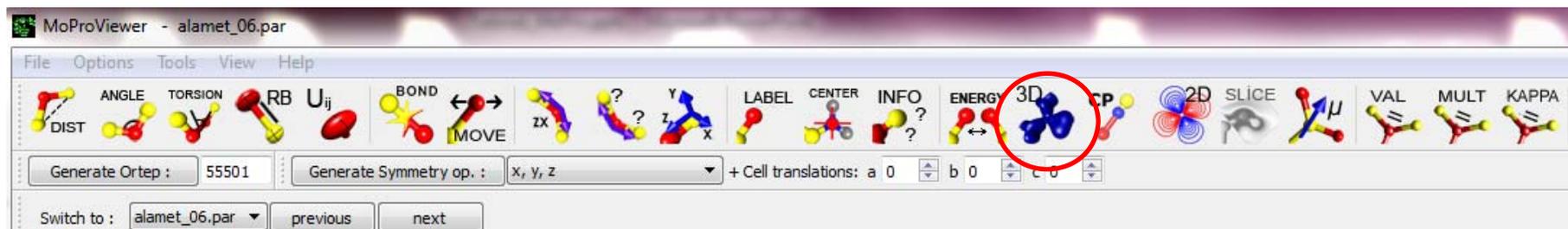
Step 17 Check Static Deformation Electron Density 2D maps



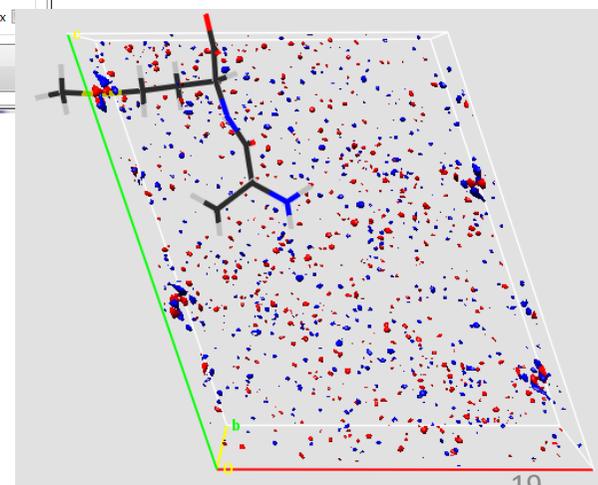
Choose
Triplets of connected atoms
to define planes



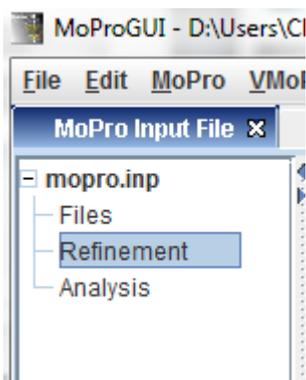
Step 18 Fast Fourier Transform 3D map



Residual Electron Density map in unit cell



Step 19 Stereochemical analysis



Refinement block name

RESOLUTION SELECTION (applies until a new selection is made)

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

MANUAL SELECTION OF PARAMETERS TO REFINE

Use file :

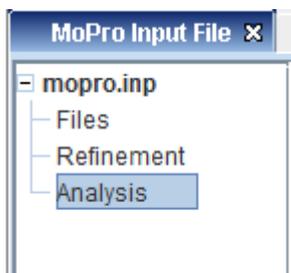
SELECTION OF PARAMETERS TO REFINE

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

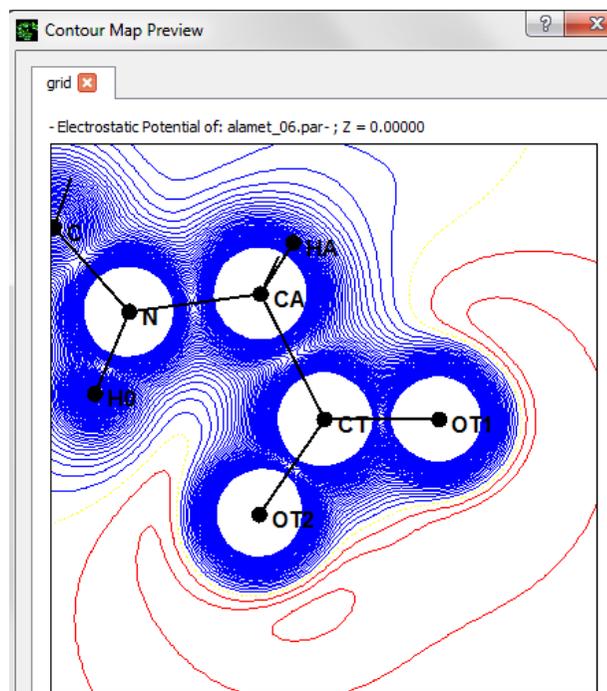
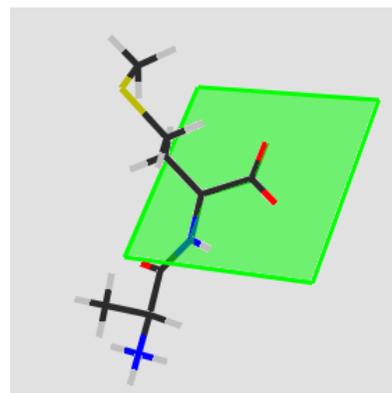
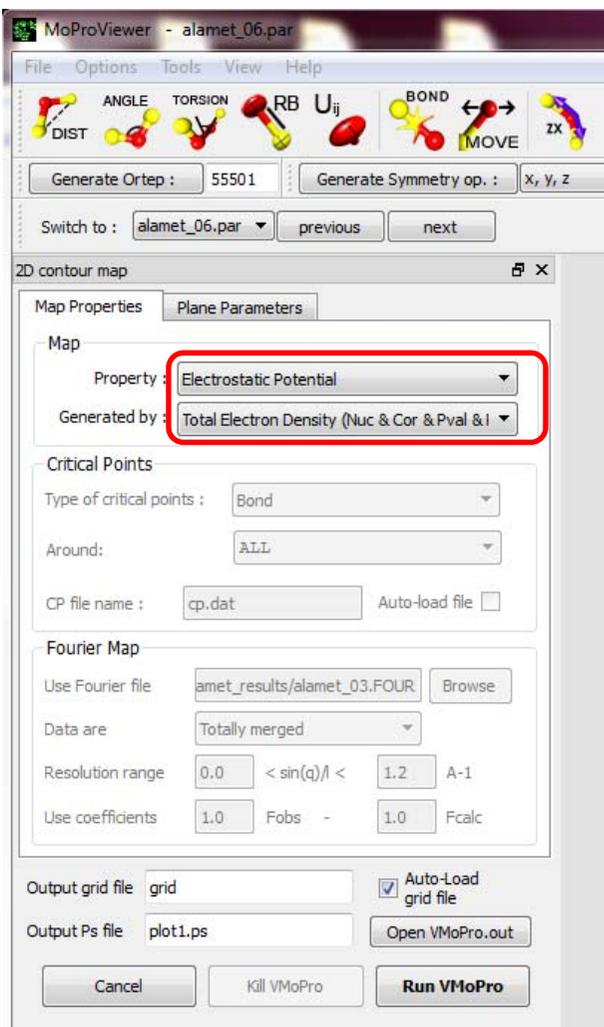
Molecular Connectivity

Refine
at first
structure
to obtain
sigmas
of distances



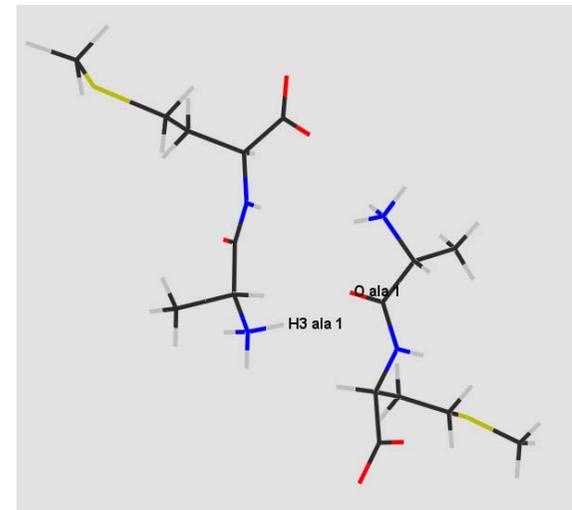
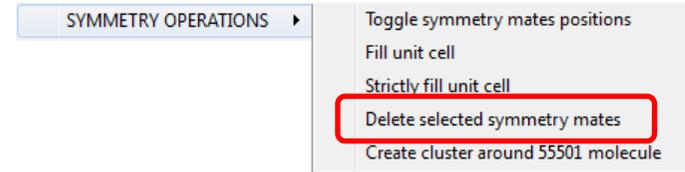
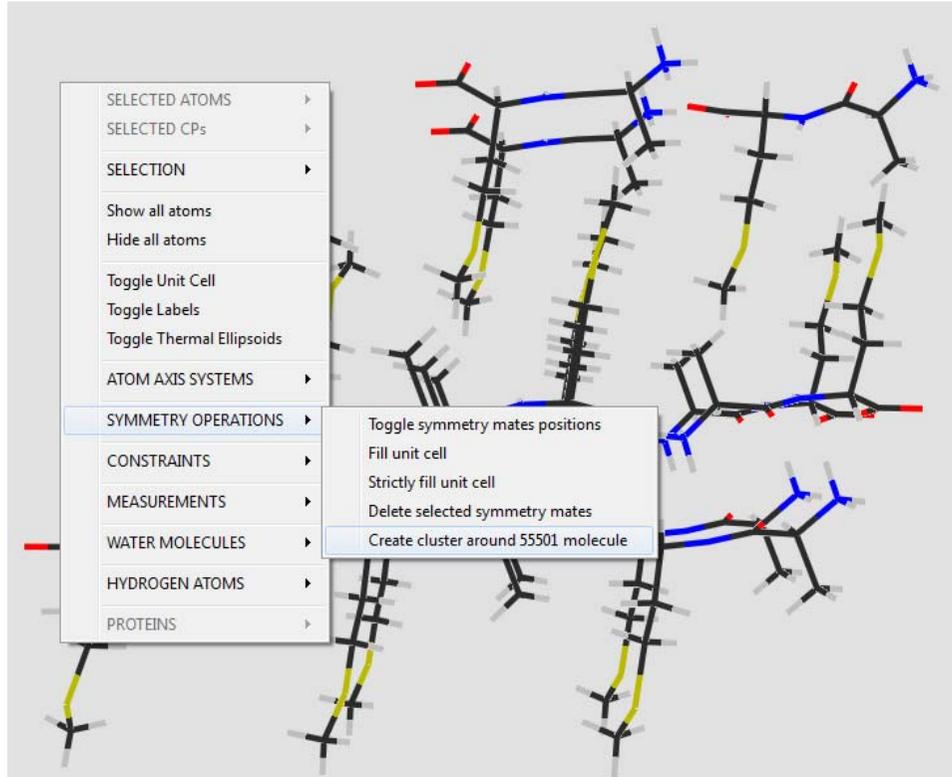
ATOM1				ATOM2			DIST	sigDIST	refined
NT	ala	1	--	CA	ala	1			xyz xyz
NT	ala	1	--	CA	ala	1	1.481091	0.001594	+++ +++
NT	ala	1	--	H2	ala	1	1.030136	0.001247	+++ +++
NT	ala	1	--	H3	ala	1	1.034194	0.002353	+++ +++
NT	ala	1	--	H1	ala	1	1.036034	0.001297	+++ +++
CA	ala	1	--	C	ala	1	1.525269	0.000535	+++ +++

Step 20 2D map of Electrostatic Potential



Step 21 Generate a dimer in MoProViewer

Click right on view



Keep a dimer like 55501 + 65602

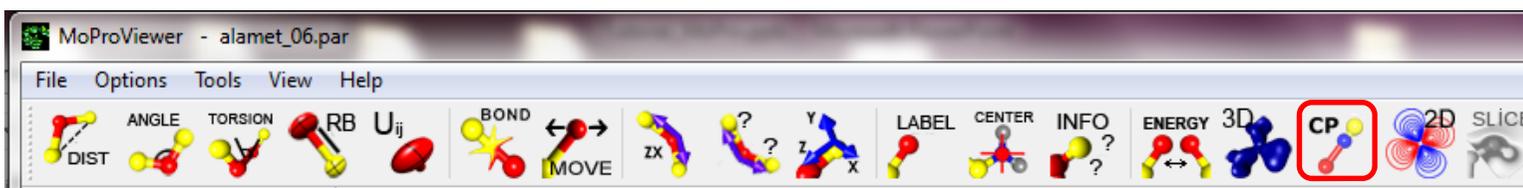
Ortep symmetry code

65602

Translation $1\mathbf{a} + 0\mathbf{b} + 1\mathbf{c}$ & symmetry #02

$6 \rightarrow 1$ $5 \rightarrow 0$ $4 \rightarrow -1$

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

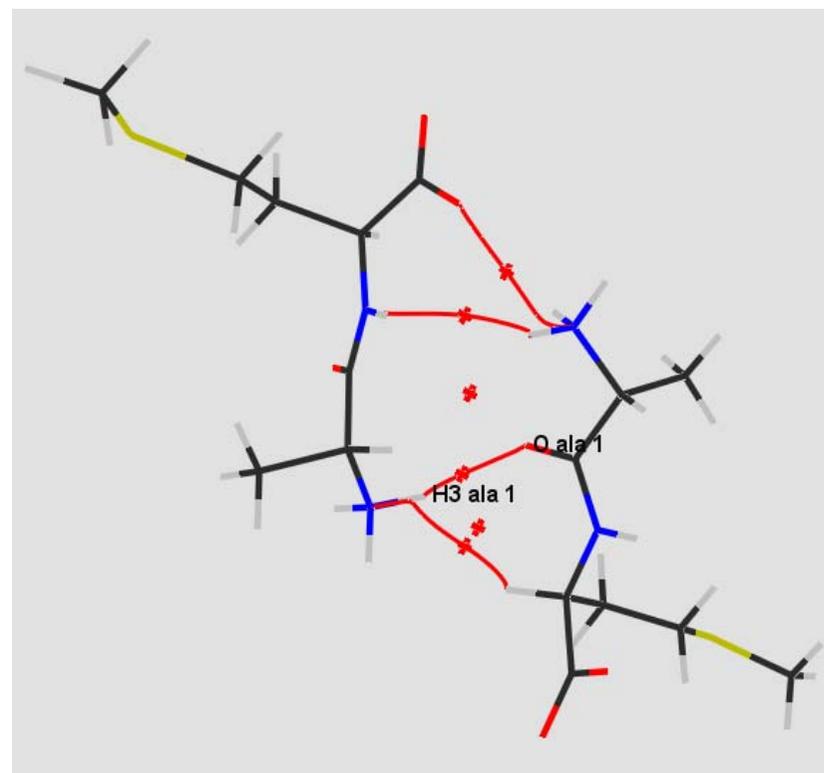
Search options

Number of iterations: 900

Output file name: cp.dat Auto-load

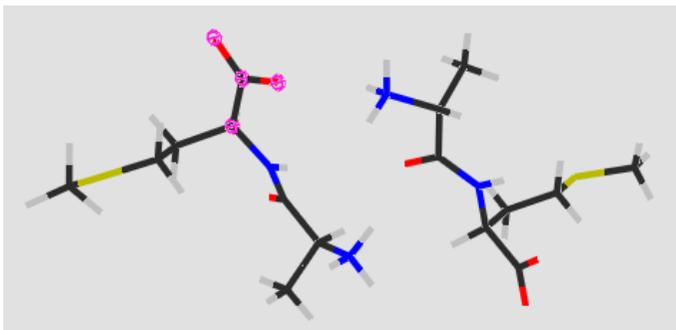
Cancel Kill VMoPro Run VMoPro

Search
around
atom
H3 only
(fast
calculation)



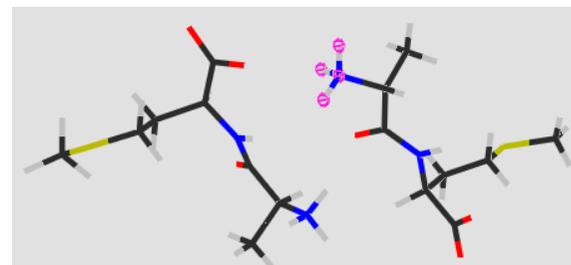
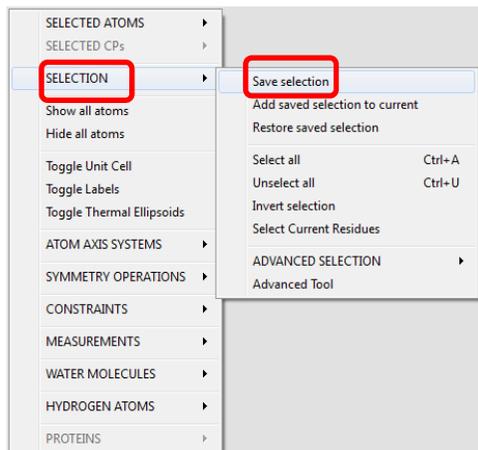
MoProViewer shows CPs & bond paths

Step 23 Compute electrostatic interaction energy of dimer



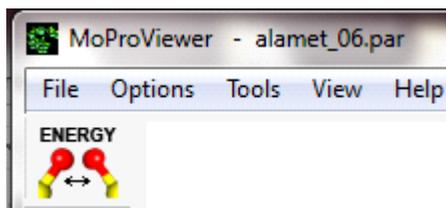
1) Select the carboxylate group

Right click, Selection / SAVE selection

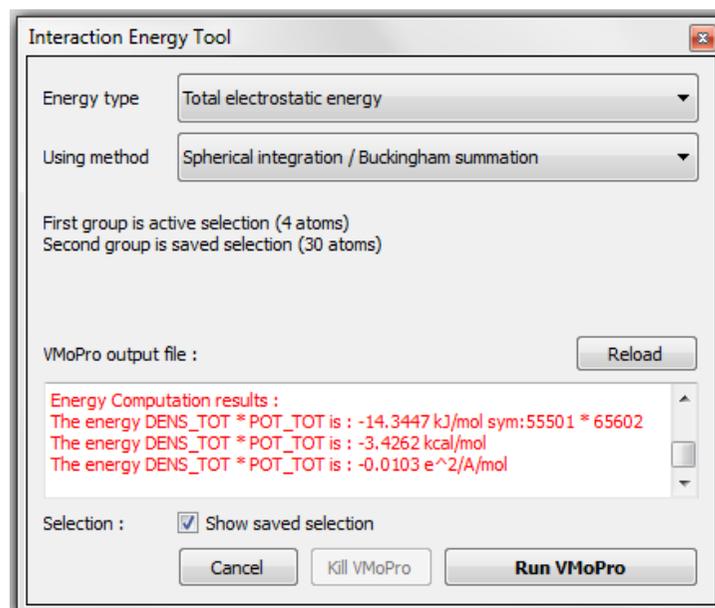


2) Select the NH3 group

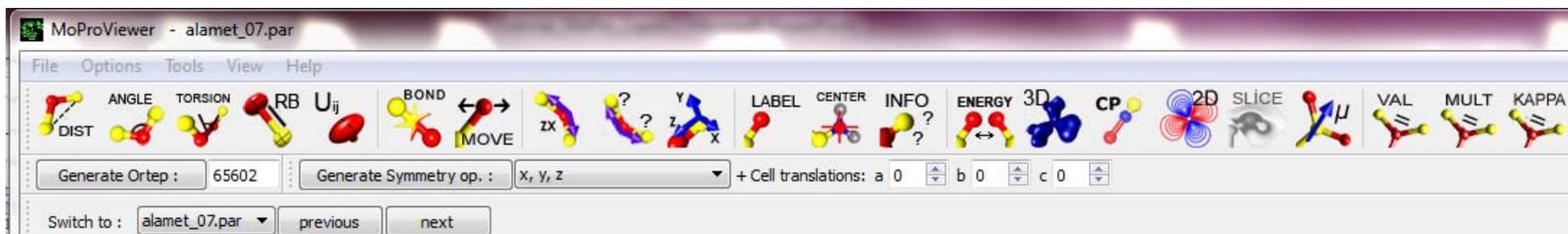
on the other molecule



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

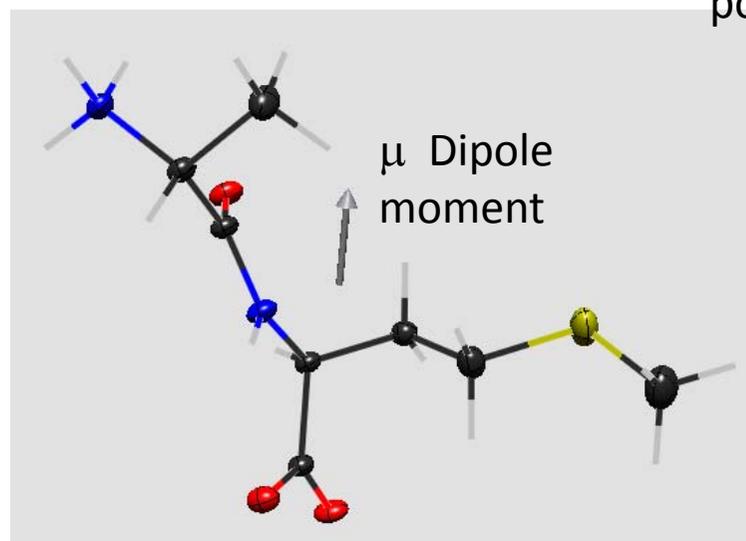
Compute 3D map

Compute 2D map

Critical points

Equivalence Constraints of atomic charge density

Uij Thermal ellipsoids



Step 25 Discover the MoPro Menus

The screenshot displays the MoProGUI software interface. The window title is "MoProGUI - D:\Users\Christian\Desktop\jelsch_pavia\alامت_tutorial". The menu bar includes "File", "Edit", "MoPro", "VMoPro", "MoProVjewer", "Mollynx", and "Help". The "MoPro Input File" tab is active, showing a tree view with "mopro.inp" expanded to show "Files", "Options", "Verbose", and "Preparation".

The "MoPro" menu is open, listing the following options: Files, Preparation, Verbose, Options, Database Transfer, Reset / Zero, **Modification** (highlighted), Constraints, Restraints, Refinement, Automatic Refinement, Solvent, Analysis, Output, Manual Commands, ADDR / GOTO / LOOP, INCLUDE, Stop, and Duplicate Block. The "Modification" menu item has a sub-menu with "Before selected item" and "After selected item".

The "Modification of Molecule" panel is visible, containing the following sections:

- Shake Molecular Structure:**
 - Shake
 - All parameters
 - XYZ
 - Uij
 - KAPpa
 - Kappa1
 - Kappa2
 - VALence
 - PLM multipoles
 - DIPoles
 - QUAdrupole
 - OCTupole
 - HEXadecapole
 - <shift>:
 - Atoms: HYDrogen only NO Hydrogen VIRTual only NO Virtual
- Set number of scale Factors:
- Neutralize Electrically. atoms selection
- Translate Sequence (Residue Number):
- Translate molecule by vector x y z Unit: Bohr Angstrom Fractional
- Apply Symmetry Operator. Ortep code
- Enantiomorph structure
- Atoms selection:
 - Isotropic \Rightarrow Anisotropic
 - Thermal Parameters: Anisotropic \Rightarrow Isotropic
 - Set Uij positive definite
- Set Multipole Level:

Chemical Types	Monopole	Dipole	Quadrupole	Octupole	Hexadecapole
<input type="text"/>	<input type="radio"/>				