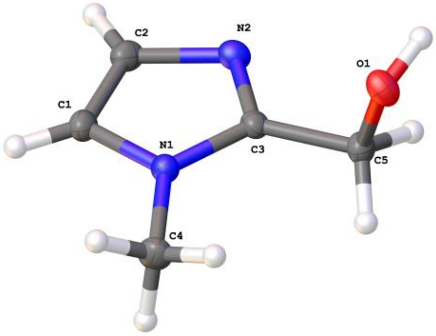
A brief introduction on carrying out a multipole refinement with invariom scattering factors and on preparing files suitable for a charge density refinements (in windows).

This document is meant to help beginners get started on multipole refinements. It is not meant to replace other documentation. The Invariomtool manual and Farrugia's charge density refinement tutorials are essential.

1. Collect good quality medium-high resolution data. Charge density quality data is not essential for invarioms, but it is helpful to compare an invariom refinement with a standard multipole refinement.
2. Scale the data with SADABS. Choose option [D] for a merged XD format hkl file.
3. Refine the structure completely with SHELX.

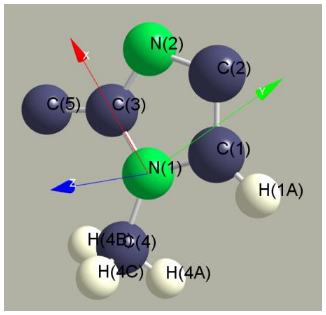


4. Run XDINI to create the XD (XD.mas and XD.inp) files suitable for refinement with XD2006. XDINI needs the shelx ins and hkl files to be named as shelx.ins and shelx.hkl. (My version of XDINI only works with isotropically refined thermal parameters. I am not sure if this is a bug or formatting errors). XDINI will creates an XD format hkl file from the SHELX hkl file, but this file usually contains unmerged data with negative intensities while XD prefers merged data with negative and zero intensities omitted. (Use the SADABS generated XD hkl file.) Note that windows, by default, associates the XD master file (.mas) with Microsoft access and then this file cannot be edited by right-clicking the file! In this case you have to change the file associations to be able to associate it with a text editor: (http://windows.microsoft.com/en-us/windows-vista/change-which-programs-windows-uses-by-default).

5. Perform a standard spherical atom refinement in XD2006. Create a sub-directory \spher for this refinement. See pages 9 – in Farrugia's tutorial on how to do this refinement: http://www.chem.gla.ac.uk/~louis/xdworkshop/workshop/documentation/tutorial3.pdf. Activate the Hirshfeld rigid bond test and change the number of cycles from -1 to 4. I like to refine with the default weights rather than the SHELX weights to see the refined GOOF. The results should be similar to a shelx refinement.

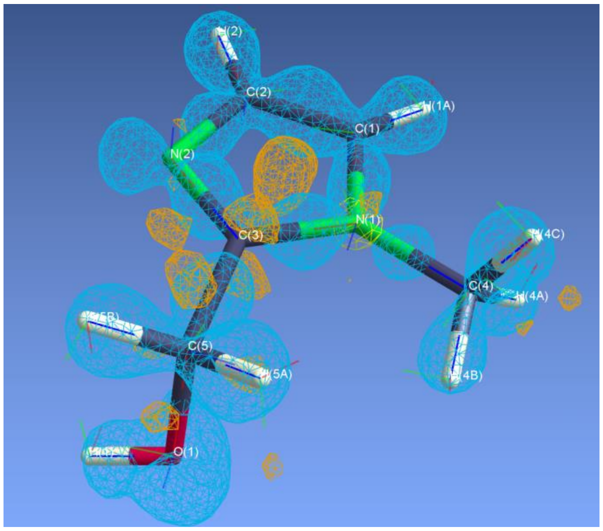
6. Download the latest version of the Invariom database with MoleCoolQT (www.molecoolqt.de). (Invarioms, Load latest Database from the Internet in MoleCoolQT). The invariom database will be located in AppData\Roaming\Christian_B._Huebschle. Copy this file and the latest version of invtool (http://ewald.ac.chemie.uni-goettingen.de/programs.html) to a folder \prepare_input. Follow Birger Dittrich and Christian Huebschle's instructions (manual) for running invtool. The XD2006 mas and inp files from a successful spherical atom refinement should be copied to this folder and the configuration file invtool.ini. Invtool created the files xd.mas.invariomtool and xd.res.invariomtool suitable for XD2006. The file Invariom.descent shows that two atoms do not appear to be in the database. O(1): O1c1h from: methanol
   N(1): 5-N#5c[#5n1c]#5c[#5c1h]1c IS NOT IN THE DATABASE YET!
   N(2): 5-N#5c[#5n1c]#5c[#5c1h] from: 2-methyl-1-phenyl-1H-imidazole
   C(3): 5-C#5n[#5c1c]#5n[#5c]1c IS NOT IN THE DATABASE YET!
   C(5): C1o@5c1h1h from: furan-3-ylmethanol
   C(2): 5-C#5n[#5c]#5c[#5n1h]1h from: imidazole
   C(4): C@5n1h1h1h from: 1-methyl-pyrrole
   C(1): 5-C#5n[#5c1c]#5c[#5n1h]1h from: n-methylimidazole
   H(5A): H1c[1o@5c1h] from: furan-3-ylmethanol
   H(5B): H1c[1o@5c1h] from: furan-3-ylmethanol
   H(1): H1o[1c] from: methanol
   H(4A): H1c[@5n1h1h] from: 1-methyl-pyrrole
   H(4B): H1c[@5n1h1h] from: 1-methyl-pyrrole
   H(4C): H1c[@5n1h1h] from: 1-methyl-pyrrole
   H(1A): H@5c from: cyclopentadienyl_anion
   H(2): H@5c from: cyclopentadienyl_anion

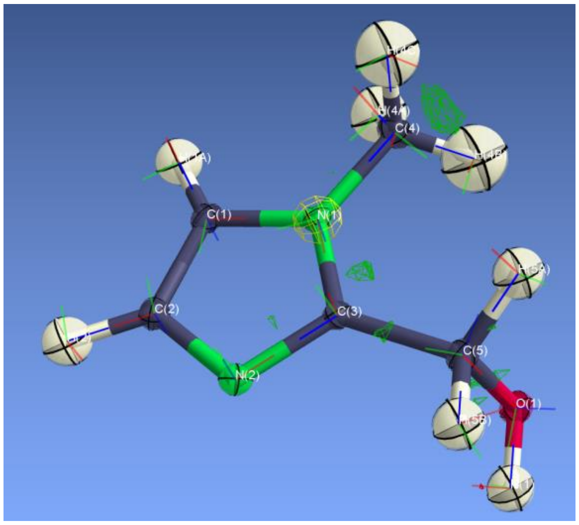
   However, since this molecule is an imidazole the database should be checked to see if the invariom assignments are correct and if not whether there are similar invarioms in the database. Atom, N(1): 5-N#5c[#5n1c]#5c[#5c1h]1c is very similar to 5-N#5c[#5n1h]#5c[#5c1h]1c in n-methylimidazole which is in the database. (Because I have charge density data I can choose to refine this atom's multipoles freely rather than use invarioms.) The calculated scattering factors for 5-N#5c[#5n1h]#5c[#5c1h]1c with Kappa values can by manually inserted into XD.inp. Note check that the local coordinate system of the atom in the database matches the symmetry in your molecule. In this case X is along the bond to C(3) and Y along the C(1) and has mirror symmetry along z. An excellent introduction to the local coordinate system is given in Farrugia's tutorial, starting at p 34.

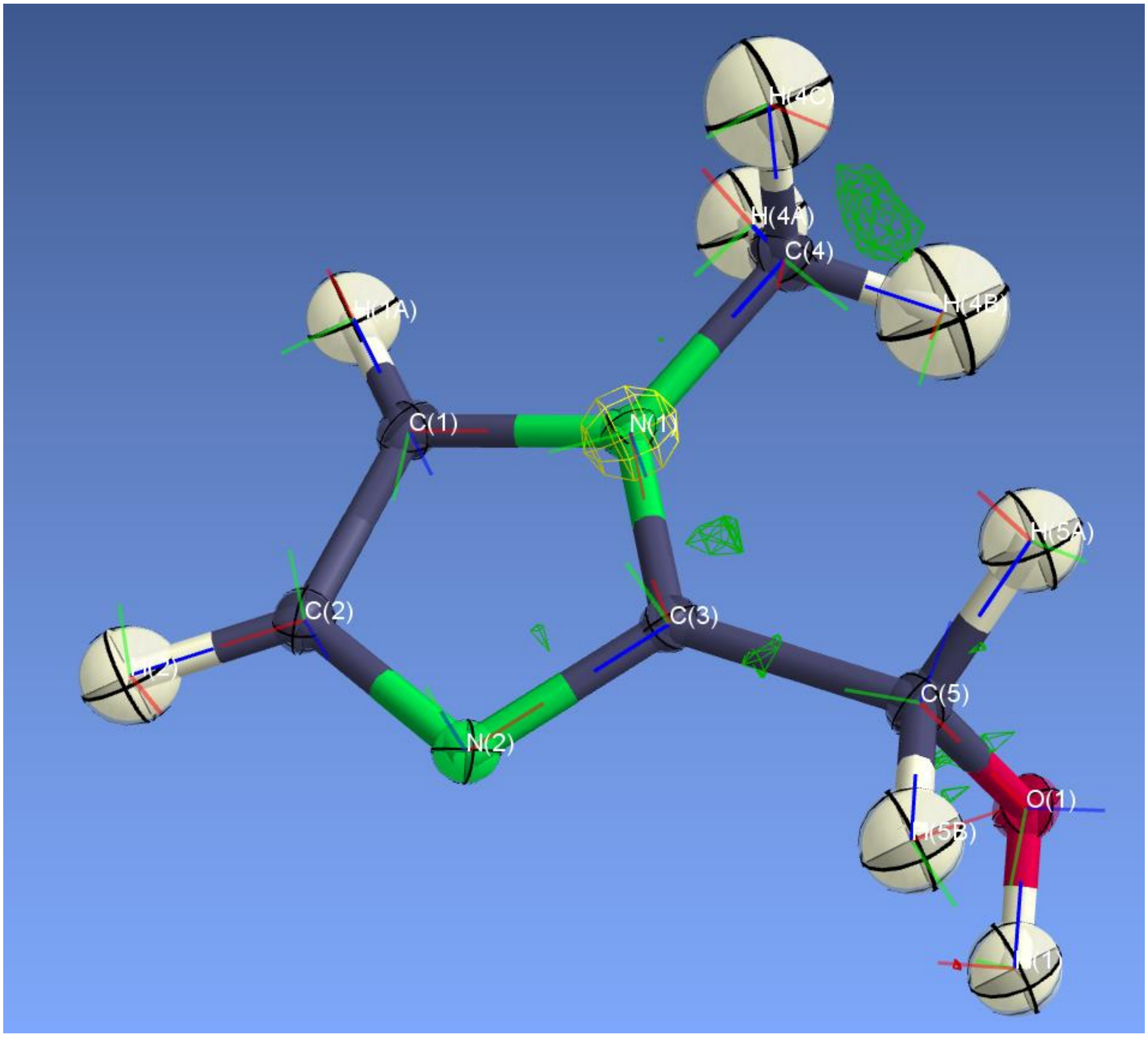


7. Copy the files xd.mas.invariomtool and xd.res.invariomtool to a folder /invariom and rename them xd.mas and xd.inp. Make sure that the following commands in the master file are selected (not commented out): DMSDA 1.1 1.8
   FOUR fmod1 4 2 0 0 fmod2 -1 2 0 0
   I get an R1 of 2% compared to 3-4% with spherical atom refinement. The file xd.mas.HasSameSymm can be used for a conventional charge density refinement.

   Have a look at the results to see if they are reasonable. Does the deformation density match the expected chemical features? Is there any residual density that was not modelled? How well do the H atoms refine? What do the DSMDA's look like?






H(4C)
H(4A)
C(4)
H(4B)
H(1A)
C(1)
N(1)
H(5A)
C(2)
C(3)
H(2)
C(5)
N(2)
O(1)
H(5B)
H(1)