Hydrogen bonds in dimethyl (1R*,2R*)-2[(tert-butoxycarbonyl)amino]-1-hydroxy-2-phenylethylphosphonate – electron density studies

M. Małecka, L. Chęcińska, A. Rybarczyk-Pirek, D.G. Piotrowska, and C. Paulmann

1Dept. of Crystallography and Crystal Chemistry, University of Łódź, Pomorska 149/153, 90 236 Łódź, Poland
2Bioorganic Chemistry Laboratory, Faculty of Pharmacy, Medical University of Łódź, 90 151 Łódź, Muszyńskiego 1, Poland
3Mineralogisch-Petrographisches Institut, Universität Hamburg, Grindelallee 48, 20 146 Hamburg, Germany

The title compound dimethyl (1R*,2R*)-2[(tert-butoxycarbonyl)amino]-1-hydroxy-2-phenylethylphosphonate crystallizes in monoclinic $P2_1/c$ space group, $Z=4$. 161146 reflections were measured with synchrotron radiation ($\lambda=0.6\AA$) at a temperature 100K at beamline F1 (Hasylab/DESY). The XDS software was applied for integration of the MAR CCD detector data [1]. The structure was solved and refined with SHELXL [2] and the obtained model was used as the input for aspherical atom multipole formalism according to the method of Hansen and Coppens [3] using the XD2006 package program [4]. The preliminary calculations on hexadecapolar level of multipole refinement gave $R(F) = 2.05\%$.

The crystal structure of title compound (on the left) is stabilized by N-H…O, O-H…O and C-H…O hydrogen bonds (see Table). The combination of N-H…O and O-H…O hydrogen bonds forms dimers of molecules related by a centre of inversion. The value of the electron density of H-bonds and their Laplacian differentiates observed interactions to medium and weak hydrogen bonds.

<table>
<thead>
<tr>
<th>Bond</th>
<th>symmetry</th>
<th>D-H</th>
<th>H…A</th>
<th>D…A</th>
<th>$&lt;$D-H…A$&gt;$</th>
<th>$\rho_{\text{exp}}$</th>
<th>$V^2\rho_{\text{exp}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...(3)</td>
<td>-x, 1-y, -z</td>
<td>1.01</td>
<td>2.07</td>
<td>2.996(1)</td>
<td>152</td>
<td>0.08(1)</td>
<td>2.0(1)</td>
</tr>
<tr>
<td>O(6)-H(6A)...O(3)</td>
<td>-x, 1-y, -z</td>
<td>1.01</td>
<td>1.75</td>
<td>2.753(1)</td>
<td>170</td>
<td>0.15(1)</td>
<td>4.6(1)</td>
</tr>
<tr>
<td>C(6)-H(6)...O(2)</td>
<td>1-x, 1-y, -z</td>
<td>0.97</td>
<td>2.40</td>
<td>3.255(1)</td>
<td>147</td>
<td>0.07(1)</td>
<td>1.1(1)</td>
</tr>
<tr>
<td>C(51)-H(51A)...O(2)</td>
<td>x, 3/2-y, 1/2+z</td>
<td>1.06</td>
<td>2.34</td>
<td>3.334(1)</td>
<td>156</td>
<td>0.05(1)</td>
<td>1.1(1)</td>
</tr>
</tbody>
</table>

Acknowledgements

The research leading to these results has received funding from the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement No° 226716.

References