Gamal A. El-Hiti*, Bakr F. Abdel-Wahab, Mohammed Baashen, Amany S. Hegazy and Benson M. Kariuki

Crystal structure of (Z)-4-((E)-(4-chlorobenzylidene)hydrazono)-1-p-tolylpyrrolidine-3-carbonitrile, C$_{19}$H$_{17}$ClN$_4$

DOI 10.1515/ncrs-2016-0079
Received June 17, 2016; accepted August 10, 2016; available online August 24, 2016

Abstract
C$_{19}$H$_{17}$ClN$_4$; triclinic, P$ar{1}$ (no. 2), a = 6.9042(5) Å, b = 7.1990(5) Å, c = 18.2633(13) Å, $\alpha$ = 86.727(6)°, $\beta$ = 79.214(6)°, $\gamma$ = 69.876(7)°, V = 837.25(11) Å$^3$, Z = 2, $R_{gt}$($F$) = 0.0553, $wR_{ref}(F^2)$ = 0.1406, $T$ = 296(2) K.

CCDC no.: 1498676

The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal:</th>
<th>Colourless plate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength:</td>
<td>Mo Kα radiation (0.71073 Å)</td>
</tr>
<tr>
<td>$\mu$:</td>
<td>2.4 cm$^{-1}$</td>
</tr>
<tr>
<td>Diffractometer, scan mode:</td>
<td>SuperNova, $\omega$-scans</td>
</tr>
<tr>
<td>$2\theta_{max}$, completeness:</td>
<td>59.6°, &gt;83%</td>
</tr>
<tr>
<td>$N$(hkl)$<em>{measured}$, $N$(hkl)$</em>{unique}$, $R_{int}$:</td>
<td>8852, 3988, 0.028</td>
</tr>
<tr>
<td>Criterion for $I_{obs}$, $N$(hkl)$_{E}$:</td>
<td>$I_{obs}$ &gt; 2 $\sigma(I_{obs})$, 2319</td>
</tr>
<tr>
<td>$N$(param)$_{refined}$:</td>
<td>218</td>
</tr>
<tr>
<td>Programs:</td>
<td>CrysAlisPRO [12], SHELX [13], WinGX [14]</td>
</tr>
</tbody>
</table>

Source of material
(Z)-4-((E)-(4-chlorobenzylidene)hydrazono)-1-p-tolylpyrrolidine-3-carbonitrile was synthesized from reaction of equimolar quantities of 4-hydrazono-1-p-tolylpyrrolidine-3-carbonitrile and 4-chlorobenzaldehyde in ethanol in the presence of few drops of glacial acetic acid under reflux for 1 h. The solid produced was filtered, dried and recrystallized from dimethylformamide to give colourless crystals of the title compound (Mp 210–211 °C) [1].

Experimental details
All hydrogen atoms were placed in calculated positions and refined using a riding model. Methylen C–H bonds were fixed at 0.97 Å and methyl C–H bonds at 0.96 Å with 1.5$U_{eq}$(C). Methyl groups were allowed to spin about the C–C bond. Aromatic C–H distances were set to 0.93 Å and N–H set to 0.86 Å with $U_{iso}$ set to 1.2$U_{eq}$(N/C).

Discussion
The most efficient syntheses of pyrrolidines involve reactions of primary amines with diols in the presence of a metal complex catalyst [2, 3], of primary amines with dihaloalkanes in the presence of potassium carbonate under microwave conditions [4], of cyclization of amino alcohols in the presence of thionyl chloride [5] and of N-tosylhydrazones with vinyl iodides in the presence of a Pd-catalyst [6]. They can be...
used as inhibitors for thrombin, and as antiarrhythmic and antihypertensive drugs [7–11].

The asymmetric unit comprises one molecule. The molecule is almost planar as the angle between the chlorobenzene and tolylpyrrolidine-carbonitrile groups is 75.26(6)° and between the latter group and the toluene group is 73.3(8)°. In the crystal, the molecules are linked by N—H⋯N hydrogen bonds to form chains aligned to [010]. For the hydrogen bond, the N2⋯N4 distance is 2.953(2) Å and the N2—H2a⋯N4 angle is 161.0°. A short C1⋯C1 contact of 3.27 Å is observed in the structure.

Acknowledgements: The authors extend their appreciation to the College of Applied Medical Sciences Research Centre and the Deanship of Scientific Research at King Saud University for their funding and to Cardiff University for continued support.

References