Supporting Information

Construction of 2,3,4,5-tetrahydro-1,2,4-triazines via [4+2] cycloaddition of α-halogeno hydrazones with imines

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1. $^1$H and $^{13}$C NMR spectra
3am

PGL-V-P19-1 1H

N
N
O

N
N

3am

PGL-V-P19-1 13C

N
N
O

N
N

3am

200 180 160 140 120 100 80 60 40 20 0 ppm
2. X-Ray crystal data of compound 3aa

![Image of compound 3aa]

**Figure 1.** X-ray single crystal structure of 3aa (with thermal ellipsoid shown at the 50% probability level)

<table>
<thead>
<tr>
<th>Identification code</th>
<th>3aa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C_{24}H_{23}N_{3}O</td>
</tr>
<tr>
<td>Formula weight</td>
<td>369.45</td>
</tr>
<tr>
<td>Temperature</td>
<td>113(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
<tr>
<td>Crystal system, space group</td>
<td>Orthorhombic, Pbca</td>
</tr>
</tbody>
</table>
| Unit cell dimensions| a = 13.3996(18) Å   alpha = 90 deg.  
                          b = 12.191(2) Å   beta = 90 deg.  
                          c = 22.958(3) Å   gamma = 90 deg. |
<p>| Volume              | 3750.3(9) Å³ |
| Z, Calculated density| 8, 1.309 Mg/m³ |
| Absorption coefficient| 0.081 mm⁻¹ |
| F(000)              | 1568 |
| Crystal size        | 0.20 x 0.18 x 0.12 mm |
| Theta range for data collection| 3.04 to 27.62 deg. |
| Limiting indices    | -17&lt;=h&lt;=17, -15&lt;=k&lt;=15, -29&lt;=l&lt;=29 |
| Reflections collected / unique | 45028 / 4327 [R(int) = 0.0237] |</p>
<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Completeness to theta</td>
<td>25.02</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.9903 and 0.9839</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on $F^2$</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>4327 / 0 / 254</td>
</tr>
<tr>
<td>Goodness-of-fit on $F^2$</td>
<td>1.069</td>
</tr>
<tr>
<td>Final R indices [$I&gt;2\sigma(I)$]</td>
<td>$R1 = 0.0343$, $wR2 = 0.0915$</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>$R1 = 0.0377$, $wR2 = 0.0934$</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>0.337 and -0.251 e.A$^{-3}$</td>
</tr>
</tbody>
</table>