Supporting information for

The Synthesis of macrocyclic peptidomimetics via Ugi/click-strategy

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Figure S1. Structure of compound 5o. Anisotropic displacement ellipsoids show 50% probability levels. Hydrogen atoms are drawn as circles with small radii.

Identification code: 5o
Empirical formula: C16 H19 N5 O2
Formula weight: 313.36
Temperature: 100(2) K
Wavelength: 0.96990 Å
Crystal system: Monoclinic
Space group: P2_1/c
Unit cell dimensions:
- a = 8.9287(18) Å  \( \square = 90^\circ \)
- b = 19.324(4) Å \( \square = 110.58(3)^\circ \)
- c = 9.833(2) Å  \( \square = 90^\circ \)
Volume: 1588.3(6) Å³
Z: 4
Density (calculated): 1.311 Mg/m³
Absorption coefficient: 0.189 mm⁻¹
F(000): 664
Crystal size: 0.20 x 0.03 x 0.02 mm³
Theta range for data collection: 3.345 to 38.446°
Index ranges: $-11 \leq h \leq 7$, $-22 \leq k \leq 22$, $-11 \leq l \leq 12$

Reflections collected: 10660

Independent reflections: 3285 [R(int) = 0.0886]

Completeness to theta = 35.587°: 98.1%

Absorption correction: Semi-empirical from equivalents

Max. and min. transmission: 0.987 and 0.960

Refinement method: Full-matrix least-squares on $F^2$

Data / restraints / parameters: 3285 / 0 / 211

Goodness-of-fit on $F^2$: 0.959

Final R indices [for 2152 reflns with $I>2\sigma(I)$]: $R_1 = 0.0825$, $wR_2 = 0.1707$

R indices (all data): $R_1 = 0.1099$, $wR_2 = 0.1881$

Extinction coefficient: 0.042(4)

Largest diff. peak and hole: 0.342 and -0.367 e.Å$^{-3}$

**Table S1.** Hydrogen bonds for 5o [Å and °].

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
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<tr>
<td>C(5)-H(5)...O(2)#1</td>
<td>0.95</td>
<td>2.42</td>
<td>3.150(2)</td>
<td>134</td>
</tr>
<tr>
<td>N(13)-H(13)...O(1)#2</td>
<td>0.91</td>
<td>1.94</td>
<td>2.846(2)</td>
<td>176</td>
</tr>
<tr>
<td>N(16)-H(16)...N(3)#1</td>
<td>0.91</td>
<td>2.09</td>
<td>2.982(3)</td>
<td>165</td>
</tr>
<tr>
<td>C(18)-H(18A)...O(1)#3</td>
<td>0.99</td>
<td>2.50</td>
<td>3.483(2)</td>
<td>170</td>
</tr>
<tr>
<td>C(18)-H(18B)...N(2)#1</td>
<td>0.99</td>
<td>2.52</td>
<td>3.362(3)</td>
<td>143</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 $x$, $-y+3/2$, $z-1/2$    #2 $x$, $-y+3/2$, $z+1/2$    #3 $x-1$, $y$, $z$
3c (unstable compound, easily decomposes)
two rotamers in ratio 5:1
3g (two rotamers in ratio 5:1)
3h
3j (two rotamers in ratio 4:1)
3k (several rotamers)
two rotamers in ratio 3:1
3m (two rotamers in ratio 5:1)
3p (two rotamers in ratio 4:1)
3u (One major rotomer and several minors in traces amounts)
4i (two conformer in ratio 3:1)
4j (two conformers in ratio 2:1)
4k (several conformers)
41 (several conformers)
4m (the mixture of monomer and dimer in ratio 1:1)
4o (several conformers)
4r (two conformers or diastereoisomer in ratio 2:1)
5o (the mixture of conformers)
5r (several conformers or diastereomers)