Supplementary Material

Tert-Butylation of naphthalene-2,6-diol and 6-methoxynaphthalen-2-ol

Andrey A. Berezin*, Davide Marinelli

School of Chemistry, Cardiff University Main Building, Park Pl, Cardiff CF10 3AT, United Kingdom
E-mail: Berezina@cardiff.ac.uk

Dedicated to Professor Oleg A. Rakitin on the occasion of his 65th birthday

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X-ray measurements:
Single crystals of 2 and 3 were grown by slow evaporation of CHCl₃ solutions, while a single crystal of 5 was obtained by evaporation of a hexane solution. Crystallographic studies were undertaken on single crystal mounted in paratone, mounted on the goniometer head with a nylon loop and studied on an Agilent SuperNova Dual three-circle diffractometer using Cu-Kα (λ = 1.540598 Å) or Mo-Kα (λ = 0.7093187 Å) radiation and a CCD detector. Measurements were typically made at 150(1) K with temperatures maintained using an Oxford Cryostream unless otherwise stated. Data were collected, integrated and corrected for absorption using a numerical absorption correction based on Gaussian integration over a multifaceted crystal model within CrysAlisPro. The structures were solved by direct methods and refined against F2 within SHELXL-2013. A summary of crystallographic data are available as ESI and the structures deposited with the Cambridge Structural Database (CCDC deposition numbers 1558768 (2), 1558766(3), and 1558767 (5)). These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data for compound 2

Table S1. Crystal data and structure refinement for 2.

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  b = 11.0764(3) Å   β = 104.115(4)°.
  c = 24.8986(10) Å  γ = 90°.
Volume           3276.8(2) Å³
Z                 4
Density (calculated)  1.177 Mg/m³
Absorption coefficient  0.622 mm⁻¹
F(000)             1264
Crystal size      0.260 x 0.182 x 0.068 mm³
Theta range for data collection   3.661 to 73.864°.
Index ranges     -15<=h<=14, -8<=k<=13, -30<=l<=30
Reflections collected  9291
Independent reflections  4729 [R(int) = 0.0329]
Completeness to theta = 67.684°  100.0 %
Absorption correction Semi-empirical from equivalents
Max. and min. transmission  1.00000 and 0.52751
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters  4729 / 0 / 206
Goodness-of-fit on F²         1.039
Final R indices [I>2sigma(I)]   R1 = 0.0897, wR2 = 0.2341
R indices (all data)         R1 = 0.1000, wR2 = 0.2576
Extinction coefficient  n/a
Largest diff. peak and hole  0.738 and -0.338 e.Å⁻³

Table S2. Atomic coordinates ( x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³)for 2. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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**Table S3.** Bond lengths [Å] and angles [°] for 2.

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C(1)-C(5)-C(6) 121.1(2)
C(7)-C(6)-C(8) 110.3(3)
C(7)-C(6)-C(9) 107.8(3)
C(8)-C(6)-C(9) 107.4(3)
C(7)-C(6)-C(5) 109.3(2)
C(8)-C(6)-C(5) 110.5(3)
C(9)-C(6)-C(5) 111.5(2)
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C(10)-O(2)-H(2A)  109.5

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1  #2 -x+1,-y,-z+1

**Table S4.** Anisotropic displacement parameters (Å$^2$ x 10$^3$) for 2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[a^{2}U_{11} + ... + 2ah a^* b^* U_{12}]$

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Crystal data for compound 3

Table S5. Crystal data and structure refinement for 3.

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\[ \text{c} = 25.2730(14) \ \text{Å} \quad \gamma = 90^\circ. \]

Volume  
\[ 1152.92(12) \ \text{Å}^3 \]

\[ Z = 4 \]

Density (calculated)  
\[ 1.246 \ \text{Mg/m}^3 \]

Absorption coefficient  
\[ 0.082 \ \text{mm}^{-1} \]

\[ F(000) = 464 \]

Crystal size  
\[ 0.775 \times 0.646 \times 0.135 \ \text{mm}^3 \]

Theta range for data collection  
\[ 2.972 \text{ to } 29.635^\circ. \]

Index ranges  
\[ -6 \leq h \leq 8, -9 \leq k \leq 7, -33 \leq l \leq 34 \]

Reflections collected  
5223

Independent reflections  
2724 [R(int) = 0.0187]

Completeness to theta = 25.242\(^\circ\)  
100.0 %

Absorption correction  
Gaussian

Max. and min. transmission  
1.000 and 0.340

Refinement method  
Full-matrix least-squares on \( F^2 \)

Data / restraints / parameters  
2724 / 0 / 145

Goodness-of-fit on \( F^2 \)  
1.037

Final R indices [I>2\sigma(I)]  
R1 = 0.0474, wR2 = 0.1102

R indices (all data)  
R1 = 0.0707, wR2 = 0.1240

Extinction coefficient  
n/a

Largest diff. peak and hole  
0.222 and -0.223 e.Å\(^{-3}\)

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(1)</td>
<td>11338(2)</td>
<td>4645(2)</td>
<td>1190(1)</td>
<td>33(1)</td>
</tr>
<tr>
<td>O(2)</td>
<td>4243(2)</td>
<td>2605(2)</td>
<td>3480(1)</td>
<td>39(1)</td>
</tr>
<tr>
<td>C(5)</td>
<td>6799(2)</td>
<td>3215(2)</td>
<td>2225(1)</td>
<td>20(1)</td>
</tr>
<tr>
<td>C(12)</td>
<td>9232(2)</td>
<td>1617(3)</td>
<td>528(1)</td>
<td>37(1)</td>
</tr>
<tr>
<td>C(4)</td>
<td>6514(2)</td>
<td>2866(2)</td>
<td>1673(1)</td>
<td>21(1)</td>
</tr>
<tr>
<td>C(3)</td>
<td>7961(2)</td>
<td>3323(2)</td>
<td>1318(1)</td>
<td>22(1)</td>
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<tr>
<td>C(11)</td>
<td>7572(2)</td>
<td>2953(2)</td>
<td>721(1)</td>
<td>26(1)</td>
</tr>
<tr>
<td>C(10)</td>
<td>8679(2)</td>
<td>4083(2)</td>
<td>2426(1)</td>
<td>22(1)</td>
</tr>
<tr>
<td>C(2)</td>
<td>9848(2)</td>
<td>4192(2)</td>
<td>1535(1)</td>
<td>23(1)</td>
</tr>
</tbody>
</table>

**Table S6.** Atomic coordinates (x 10\(^4\)) and equivalent isotropic displacement parameters (Å\(^2\)x 10\(^3\)) for 3. U(eq) is defined as one third of the trace of the orthogonalized U\(_{ij}\) tensor.
Table S7. Bond lengths [Å] and angles [°] for 3.

<table>
<thead>
<tr>
<th>Bond</th>
<th>Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(1)-C(2)</td>
<td>1.3767(15)</td>
</tr>
<tr>
<td>O(1)-H(1A)</td>
<td>0.8200</td>
</tr>
<tr>
<td>O(2)-C(7)</td>
<td>1.3712(16)</td>
</tr>
<tr>
<td>O(2)-H(10)</td>
<td>0.8200</td>
</tr>
<tr>
<td>C(5)-C(4)</td>
<td>1.4168(18)</td>
</tr>
<tr>
<td>C(5)-C(6)</td>
<td>1.4177(18)</td>
</tr>
<tr>
<td>C(5)-C(10)</td>
<td>1.4184(19)</td>
</tr>
<tr>
<td>C(12)-C(11)</td>
<td>1.534(2)</td>
</tr>
<tr>
<td>C(12)-H(12A)</td>
<td>0.9600</td>
</tr>
<tr>
<td>C(12)-H(12B)</td>
<td>0.9600</td>
</tr>
<tr>
<td>C(12)-H(12C)</td>
<td>0.9600</td>
</tr>
<tr>
<td>C(4)-C(3)</td>
<td>1.3747(18)</td>
</tr>
<tr>
<td>C(4)-H(4)</td>
<td>0.9300</td>
</tr>
<tr>
<td>C(3)-C(2)</td>
<td>1.4350(19)</td>
</tr>
<tr>
<td>C(3)-C(11)</td>
<td>1.5331(19)</td>
</tr>
<tr>
<td>C(11)-C(14)</td>
<td>1.5313(19)</td>
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<tr>
<td>C(11)-C(13)</td>
<td>1.537(2)</td>
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<tr>
<td>C(10)-C(1)</td>
<td>1.4106(18)</td>
</tr>
<tr>
<td>C(10)-C(9)</td>
<td>1.4175(19)</td>
</tr>
<tr>
<td>C(2)-C(1)</td>
<td>1.3647(19)</td>
</tr>
<tr>
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<td>1.365(2)</td>
</tr>
<tr>
<td>C(9)-H(9)</td>
<td>0.9300</td>
</tr>
<tr>
<td>C(1)-H(1)</td>
<td>0.9300</td>
</tr>
<tr>
<td>C(6)-C(7)</td>
<td>1.3662(19)</td>
</tr>
<tr>
<td>C(6)-H(6)</td>
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</tr>
<tr>
<td>C(7)-C(8)</td>
<td>1.410(2)</td>
</tr>
<tr>
<td>C(14)-H(14A)</td>
<td>0.9600</td>
</tr>
<tr>
<td>C(14)-H(14B)</td>
<td>0.9600</td>
</tr>
<tr>
<td>C(14)-H(14C)</td>
<td>0.9600</td>
</tr>
</tbody>
</table>
C(8)-H(8) 0.9300  
C(13)-H(13A) 0.9600  
C(13)-H(13B) 0.9600  
C(13)-H(13C) 0.9600  
C(2)-O(1)-H(1A) 109.5  
C(7)-O(2)-H(10) 109.5  
C(4)-C(5)-C(6) 122.12(12)  
C(4)-C(5)-C(10) 118.48(12)  
C(6)-C(5)-C(10) 119.40(12)  
C(11)-C(12)-H(12A) 109.5  
C(11)-C(12)-H(12B) 109.5  
H(12A)-C(12)-H(12B) 109.5  
C(11)-C(12)-H(12C) 109.5  
H(12A)-C(12)-H(12C) 109.5  
H(12B)-C(12)-H(12C) 109.5  
C(3)-C(4)-C(5) 123.69(12)  
C(3)-C(4)-H(4) 118.2  
C(5)-C(4)-H(4) 118.2  
C(4)-C(3)-C(2) 116.33(12)  
C(4)-C(3)-C(11) 121.98(12)  
C(2)-C(3)-C(11) 121.69(12)  
C(14)-C(11)-C(3) 111.59(11)  
C(14)-C(11)-C(12) 106.97(13)  
C(3)-C(11)-C(12) 110.57(11)  
C(14)-C(11)-C(13) 107.85(12)  
C(3)-C(11)-C(13) 109.42(12)  
C(12)-C(11)-C(13) 110.38(12)  
C(1)-C(10)-C(9) 123.01(12)  
C(1)-C(10)-C(5) 118.22(12)  
C(9)-C(10)-C(5) 118.78(13)  
C(1)-C(2)-O(1) 120.71(12)  
C(1)-C(2)-C(3) 121.61(12)  
O(1)-C(2)-C(3) 117.67(12)  
C(8)-C(9)-C(10) 120.63(13)  
C(8)-C(9)-H(9) 119.7  
C(10)-C(9)-H(9) 119.7  
C(2)-C(1)-C(10) 121.67(12)  
C(2)-C(1)-H(1) 119.2  
C(10)-C(1)-H(1) 119.2
Symmetry transformations used to generate equivalent atoms:

Table S8. Anisotropic displacement parameters (Å² x 10³) for 3. The anisotropic displacement factor exponent takes the form: -2π²[ h²a²U₁₁ + ... + 2 h k a* b* U₁₂ ]

<table>
<thead>
<tr>
<th></th>
<th>U₁₁</th>
<th>U₂₂</th>
<th>U₃₃</th>
<th>U₁₂</th>
<th>U₁₃</th>
<th>U₁₂</th>
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<td>37(1)</td>
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<td>8(1)</td>
<td>-8(1)</td>
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<tr>
<td>O(2)</td>
<td>48(1)</td>
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<td>30(1)</td>
<td>-4(1)</td>
<td>13(1)</td>
<td>-17(1)</td>
</tr>
<tr>
<td>C(5)</td>
<td>20(1)</td>
<td>14(1)</td>
<td>27(1)</td>
<td>1(1)</td>
<td>-1(1)</td>
<td>2(1)</td>
</tr>
<tr>
<td>C(12)</td>
<td>30(1)</td>
<td>43(1)</td>
<td>38(1)</td>
<td>-12(1)</td>
<td>3(1)</td>
<td>7(1)</td>
</tr>
<tr>
<td>C(4)</td>
<td>17(1)</td>
<td>19(1)</td>
<td>27(1)</td>
<td>1(1)</td>
<td>-2(1)</td>
<td>0(1)</td>
</tr>
<tr>
<td>C(3)</td>
<td>20(1)</td>
<td>17(1)</td>
<td>27(1)</td>
<td>1(1)</td>
<td>1(1)</td>
<td>4(1)</td>
</tr>
<tr>
<td>C(11)</td>
<td>22(1)</td>
<td>29(1)</td>
<td>26(1)</td>
<td>-1(1)</td>
<td>3(1)</td>
<td>3(1)</td>
</tr>
<tr>
<td>C(10)</td>
<td>21(1)</td>
<td>14(1)</td>
<td>28(1)</td>
<td>0(1)</td>
<td>-3(1)</td>
<td>3(1)</td>
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</table>
Crystal data for compound 5

Table S9. Crystal data and structure refinement for 5.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tbody>
<tr>
<td>Identification code</td>
<td>1558767 (5)</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C19 H26 O2</td>
</tr>
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<td>Formula weight</td>
<td>286.40</td>
</tr>
<tr>
<td>Temperature</td>
<td>150(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>1.54184 Å</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>Space group</td>
<td>P b c n</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 21.420(2) Å, α = 90°</td>
</tr>
</tbody>
</table>

Z = -97, db1656b, P b c n, R = 0.10, RES = 0, 64 X
$b = 6.4137(6)$ Å $\beta = 90^\circ$.
$c = 24.380(2)$ Å $\gamma = 90^\circ$.

Volume
3349.3(6) Å³

Z
8

Density (calculated) 1.136 Mg/m³

Absorption coefficient 0.557 mm⁻¹

F(000) 1248

Crystal size 0.940 x 0.157 x 0.084 mm³

Theta range for data collection 3.626 to 70.134°.

Index ranges $-18 \leq h \leq 25$, $-7 \leq k \leq 7$, $-29 \leq l \leq 23$

Reflections collected 8334

Independent reflections 3165 [R(int) = 0.0777]

Completeness to theta = 67.684° 99.9 %

Absorption correction Semi-empirical from equivalents

Max. and min. transmission 1.00000 and 0.28053

Refinement method Full-matrix least-squares on F²

Data / restraints / parameters 3165 / 0 / 198

Goodness-of-fit on F² 1.096

Final R indices [I>2sigma(I)] $R1 = 0.0968$, $wR2 = 0.2641$

R indices (all data) $R1 = 0.1214$, $wR2 = 0.2853$

Extinction coefficient n/a

Largest diff. peak and hole 0.282 and -0.383 e.Å⁻³

### Table S10

Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 5. U(eq) is defined as one third of the trace of the orthogonalized Uᵢⱼ tensor.

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U(eq)</th>
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</thead>
<tbody>
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<td>9630(6)</td>
<td>1287(1)</td>
<td>38(1)</td>
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<tr>
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<td>912(2)</td>
<td>8899(6)</td>
<td>766(2)</td>
<td>37(1)</td>
</tr>
<tr>
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<td>1279(2)</td>
<td>9975(6)</td>
<td>363(1)</td>
<td>37(1)</td>
</tr>
<tr>
<td>C(4)</td>
<td>1594(2)</td>
<td>11745(6)</td>
<td>539(1)</td>
<td>36(1)</td>
</tr>
<tr>
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<td>1083(1)</td>
<td>34(1)</td>
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<td>1260(1)</td>
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<td>15066(6)</td>
<td>1787(1)</td>
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<td>14034(6)</td>
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<td>1099(2)</td>
<td>12264(5)</td>
<td>1995(1)</td>
<td>36(1)</td>
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</table>
**Table S11.** Bond lengths [Å] and angles [°] for 5.

<table>
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<th>Bond</th>
<th>Length/Angle</th>
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<td>C(2)-O(2)</td>
<td>1.368(5)</td>
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<tr>
<td>C(2)-C(3)</td>
<td>1.434(5)</td>
</tr>
<tr>
<td>C(3)-C(4)</td>
<td>1.389(5)</td>
</tr>
<tr>
<td>C(3)-C(12)</td>
<td>1.527(5)</td>
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<tr>
<td>C(4)-C(5)</td>
<td>1.419(5)</td>
</tr>
<tr>
<td>C(4)-H(4)</td>
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</tr>
<tr>
<td>C(5)-C(10)</td>
<td>1.402(5)</td>
</tr>
<tr>
<td>C(5)-C(6)</td>
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<tr>
<td>C(6)-C(7)</td>
<td>1.376(5)</td>
</tr>
<tr>
<td>C(6)-H(6)</td>
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</tr>
<tr>
<td>C(7)-O(1)</td>
<td>1.373(4)</td>
</tr>
<tr>
<td>C(7)-C(8)</td>
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<tr>
<td>C(8)-C(9)</td>
<td>1.371(5)</td>
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<tr>
<td>C(8)-C(16)</td>
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</tr>
<tr>
<td>C(9)-C(10)</td>
<td>1.409(5)</td>
</tr>
<tr>
<td>C(9)-H(9)</td>
<td>0.9500</td>
</tr>
<tr>
<td>C(11)-O(2)</td>
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<tr>
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</tr>
<tr>
<td>C(11)-H(11B)</td>
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</table>
C(11)-H(11C) 0.9800
C(12)-C(15) 1.525(7)
C(12)-C(14) 1.532(6)
C(12)-C(13) 1.552(7)
C(13)-H(13A) 0.9800
C(13)-H(13B) 0.9800
C(13)-H(13C) 0.9800
C(14)-H(14A) 0.9800
C(14)-H(14B) 0.9800
C(14)-H(14C) 0.9800
C(15)-H(15A) 0.9800
C(15)-H(15B) 0.9800
C(15)-H(15C) 0.9800
C(16)-C(19) 1.532(6)
C(16)-C(17) 1.533(7)
C(16)-C(18) 1.536(6)
C(17)-H(17A) 0.9800
C(17)-H(17B) 0.9800
C(17)-H(17C) 0.9800
C(18)-H(18A) 0.9800
C(18)-H(18B) 0.9800
C(18)-H(18C) 0.9800
C(19)-H(19A) 0.9800
C(19)-H(19B) 0.9800
C(19)-H(19C) 0.9800
O(1)-H(1A) 0.8400

C(2)-C(1)-C(10) 121.4(3)
C(2)-C(1)-H(1) 119.3
C(10)-C(1)-H(1) 119.3
C(1)-C(2)-O(2) 123.2(3)
C(1)-C(2)-C(3) 121.5(3)
O(2)-C(2)-C(3) 115.3(3)
C(4)-C(3)-C(2) 116.7(3)
C(4)-C(3)-C(12) 121.6(3)
C(2)-C(3)-C(12) 121.7(4)
C(3)-C(4)-C(5) 122.7(3)
C(3)-C(4)-H(4) 118.6
C(5)-C(4)-H(4) 118.6
C(10)-C(5)-C(6) 118.4(3)
C(10)-C(5)-C(4) 119.0(3)
C(6)-C(5)-C(4) 122.6(3)
C(7)-C(6)-C(5) 120.6(3)
C(7)-C(6)-H(6) 119.7
C(5)-C(6)-H(6) 119.7
O(1)-C(7)-C(6) 121.2(3)
O(1)-C(7)-C(8) 116.9(3)
C(6)-C(7)-C(8) 121.9(3)
C(9)-C(8)-C(7) 116.1(3)
C(9)-C(8)-C(16) 122.8(3)
C(7)-C(8)-C(16) 121.1(3)
C(8)-C(9)-C(10) 123.5(3)
C(8)-C(9)-H(9) 118.2
C(10)-C(9)-H(9) 118.2
C(5)-C(10)-C(9) 119.4(3)
C(5)-C(10)-C(1) 118.7(3)
C(9)-C(10)-C(1) 121.9(3)

O(2)-C(11)-H(11A) 109.5
O(2)-C(11)-H(11B) 109.5
H(11A)-C(11)-H(11B) 109.5
O(2)-C(11)-H(11C) 109.5
H(11A)-C(11)-H(11C) 109.5
H(11B)-C(11)-H(11C) 109.5
C(15)-C(12)-C(3) 112.1(4)
C(15)-C(12)-C(14) 106.6(4)
C(3)-C(12)-C(14) 109.8(3)
C(15)-C(12)-C(13) 109.0(4)
C(3)-C(12)-C(13) 109.9(4)
C(14)-C(12)-C(13) 109.3(4)
C(12)-C(13)-H(13A) 109.5
C(12)-C(13)-H(13B) 109.5
H(13A)-C(13)-H(13B) 109.5
C(12)-C(13)-H(13C) 109.5
H(13A)-C(13)-H(13C) 109.5
H(13B)-C(13)-H(13C) 109.5
C(12)-C(14)-H(14A) 109.5
C(12)-C(14)-H(14B) 109.5
H(14A)-C(14)-H(14B) 109.5
C(12)-C(14)-H(14C) 109.5
H(14A)-C(14)-H(14C) 109.5
H(14B)-C(14)-H(14C) 109.5
C(12)-C(15)-H(15A) 109.5
C(12)-C(15)-H(15B) 109.5
H(15A)-C(15)-H(15B) 109.5
C(12)-C(15)-H(15C) 109.5
H(15A)-C(15)-H(15C) 109.5
H(15B)-C(15)-H(15C) 109.5
C(19)-C(16)-C(17) 111.0(4)
C(19)-C(16)-C(18) 107.3(4)
C(17)-C(16)-C(18) 107.7(3)
C(19)-C(16)-C(8) 110.4(3)
C(17)-C(16)-C(8) 109.7(3)
C(18)-C(16)-C(8) 110.7(3)
C(16)-C(17)-H(17A) 109.5
C(16)-C(17)-H(17B) 109.5
H(17A)-C(17)-H(17B) 109.5
C(16)-C(17)-H(17C) 109.5
H(17A)-C(17)-H(17C) 109.5
H(17B)-C(17)-H(17C) 109.5
C(16)-C(18)-H(18A) 109.5
C(16)-C(18)-H(18B) 109.5
H(18A)-C(18)-H(18B) 109.5
C(16)-C(18)-H(18C) 109.5
H(18A)-C(18)-H(18C) 109.5
H(18B)-C(18)-H(18C) 109.5
C(16)-C(19)-H(19A) 109.5
C(16)-C(19)-H(19B) 109.5
H(19A)-C(19)-H(19B) 109.5
C(16)-C(19)-H(19C) 109.5
H(19A)-C(19)-H(19C) 109.5
H(19B)-C(19)-H(19C) 109.5
C(7)-O(1)-H(1A) 109.5
C(2)-O(2)-C(11) 117.6(3)

Symmetry transformations used to generate equivalent atoms:

Table S12. Anisotropic displacement parameters (Å² x 10³) for 5. The anisotropic displacement factor exponent takes the form: -2π² [ h²a*²U¹¹ + ... + 2hk a* b* U¹² ]
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