

Crystal structure of 2-methoxy-6- $\{[1-(1\text{-adamantyl})\text{-ethyl}]\text{imino}\}$ methylphenol, $\text{C}_{20}\text{H}_{27}\text{NO}_2$

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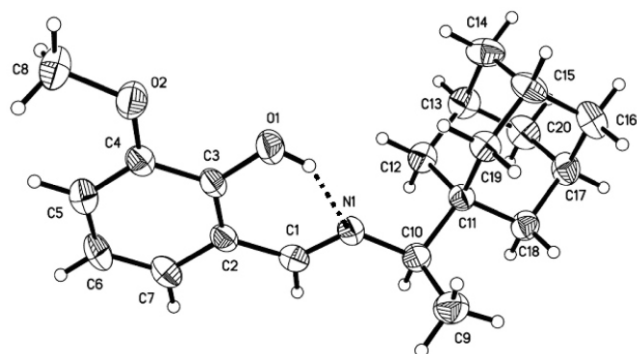


Table 1. Data collection and handling.

Crystal:	yellow plate, size $0.05 \times 0.30 \times 0.42$ mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	0.77 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	50.04°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	8446, 3020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1478
$N(\text{param})_{\text{refined}}$:	210
Programs:	SHELXS-97, SHELXL-97, SHELXTL [7]

Abstract

$\text{C}_{20}\text{H}_{27}\text{NO}_2$, monoclinic, $P12_1/c1$ (no. 14), $a = 10.164(1)$ Å, $b = 12.883(2)$ Å, $c = 14.068(2)$ Å, $\beta = 111.130(1)^{\circ}$, $V = 1718.2$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.046$, $wR_{\text{ref}}(F^2) = 0.137$, $T = 298$ K.

Source of material

A mixture of rimantadine (0.54 g, 3.0 mmol) and 2-hydroxy-3-methoxybenzaldehyde (0.46 g, 3.0 mmol) in anhydrous alcohol (30 ml) was stirred and refluxed for ca. 4 h. The solution was then concentrated and stood at room temperature. Yellow single crystals were obtained after 3 d with the solvent evaporation.

Discussion

The investigation of adamantane-ring containing Schiff bases has been attracting more and more attention in recent years because of their peculiar biological activity [1,2] as well as their use as anti-bacterial functional ligands [3,4].

The title compound crystallizes in the phenol-imine tautomeric form with bond length $d(\text{O1}—\text{C3}) = 1.348(3)$ Å. The bond length $d(\text{N1}—\text{C7}) = 1.270(3)$ Å is close to the value described for N=C bond of 1.263(5) Å [5]. The bond length $d(\text{O2}—\text{C4})$ is 1.367(3) Å. The torsion angle of C8–O2–C4–C3 is $8.0(4)^{\circ}$. A strong O–H $\cdots$ N intramolecular hydrogen bond involves the hydroxyl O1 atom and N1 atom [6]. A six-membered ring is formed by hydrogen bonds with the distances $d(\text{O}\cdots\text{H}) = 0.820$ Å and $d(\text{H}\cdots\text{N}) = 1.894$ Å as well as with the distance $d(\text{N}\cdots\text{O}) = 2.618$ Å and the angle $\angle\text{N–H}\cdots\text{O} = 146.59^{\circ}$.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.6620	0.6201	0.3143	0.096
H(1A)	4e	0.5798	0.8290	0.4209	0.065
H(5)	4e	0.2367	0.7252	0.0188	0.076
H(6)	4e	0.2239	0.8682	0.1131	0.083
H(7)	4e	0.3817	0.8896	0.2760	0.074
H(8A)	4e	0.2313	0.5411	−0.0147	0.121
H(8B)	4e	0.3447	0.4957	−0.0548	0.121
H(8C)	4e	0.3061	0.6139	−0.0689	0.121
H(9A)	4e	0.9561	0.7340	0.4838	0.109
H(9B)	4e	0.9904	0.7878	0.5899	0.109
H(9C)	4e	0.8989	0.8458	0.4900	0.109
H(10)	4e	0.7435	0.7865	0.5591	0.064
H(12A)	4e	0.6250	0.6545	0.6257	0.069
H(12B)	4e	0.6069	0.5812	0.5328	0.069
H(13)	4e	0.5951	0.4822	0.6705	0.079
H(14A)	4e	0.6923	0.3986	0.5620	0.090
H(14B)	4e	0.7652	0.3564	0.6736	0.090
H(15)	4e	0.9393	0.3957	0.6044	0.078
H(16A)	4e	1.0839	0.5031	0.7375	0.090
H(16B)	4e	1.0078	0.4203	0.7819	0.090
H(17)	4e	0.9866	0.5857	0.8472	0.076
H(18A)	4e	0.9972	0.6856	0.7101	0.070
H(18B)	4e	0.8641	0.7185	0.7346	0.070
H(19A)	4e	0.9682	0.5688	0.5603	0.064
H(19B)	4e	0.8176	0.5291	0.4921	0.064
H(20A)	4e	0.7407	0.5875	0.8045	0.087
H(20B)	4e	0.7956	0.4724	0.8225	0.087

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
N(1)	4e	0.6924(2)	0.7097(2)	0.4270(2)	0.058(2)	0.051(1)	0.048(1)	0.004(1)	0.012(1)	0.005(1)
O(1)	4e	0.6098(2)	0.6040(1)	0.2566(1)	0.058(1)	0.060(1)	0.061(1)	0.016(1)	0.006(1)	0.001(1)
O(2)	4e	0.4258(2)	0.5770(2)	0.0710(1)	0.061(1)	0.073(1)	0.060(1)	0.009(1)	0.007(1)	−0.007(1)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	0.5918(3)	0.7722(2)	0.3840(2)	0.062(2)	0.044(2)	0.058(2)	0.002(2)	0.024(2)	0.003(2)
C(2)	4e	0.4950(3)	0.7590(2)	0.2800(2)	0.049(2)	0.044(2)	0.052(2)	0.002(1)	0.019(1)	0.011(1)
C(3)	4e	0.5062(3)	0.6750(2)	0.2213(2)	0.041(2)	0.045(2)	0.053(2)	0.006(1)	0.016(1)	0.013(1)
C(4)	4e	0.4078(3)	0.6623(2)	0.1228(2)	0.048(2)	0.051(2)	0.055(2)	0.003(1)	0.020(2)	0.009(2)
C(5)	4e	0.3029(3)	0.7339(2)	0.0841(2)	0.056(2)	0.070(2)	0.057(2)	0.009(2)	0.012(2)	0.010(2)
C(6)	4e	0.2944(3)	0.8192(2)	0.1411(2)	0.060(2)	0.072(2)	0.070(2)	0.026(2)	0.018(2)	0.024(2)
C(7)	4e	0.3885(3)	0.8322(2)	0.2380(2)	0.067(2)	0.053(2)	0.068(2)	0.016(2)	0.027(2)	0.011(2)
C(8)	4e	0.3183(3)	0.5552(3)	−0.0246(2)	0.070(2)	0.101(3)	0.055(2)	−0.001(2)	0.005(2)	−0.006(2)
C(9)	4e	0.9205(3)	0.7794(2)	0.5230(2)	0.078(2)	0.067(2)	0.071(2)	−0.012(2)	0.024(2)	0.004(2)
C(10)	4e	0.7882(3)	0.7332(2)	0.5312(2)	0.056(2)	0.050(2)	0.051(2)	0.002(1)	0.016(2)	−0.003(1)
C(11)	4e	0.8105(3)	0.6364(2)	0.5989(2)	0.048(2)	0.043(2)	0.043(2)	0.003(1)	0.017(1)	−0.002(1)
C(12)	4e	0.6689(3)	0.5989(2)	0.6014(2)	0.054(2)	0.065(2)	0.058(2)	0.003(2)	0.024(2)	−0.005(2)
C(13)	4e	0.6872(3)	0.5044(2)	0.6704(2)	0.063(2)	0.077(2)	0.068(2)	−0.009(2)	0.037(2)	0.005(2)
C(14)	4e	0.7535(4)	0.4171(2)	0.6306(2)	0.108(3)	0.054(2)	0.074(2)	−0.009(2)	0.046(2)	0.001(2)
C(15)	4e	0.8965(3)	0.4521(2)	0.6300(2)	0.090(2)	0.050(2)	0.069(2)	0.018(2)	0.047(2)	0.004(2)
C(16)	4e	0.9930(3)	0.4805(2)	0.7378(2)	0.076(2)	0.079(2)	0.073(2)	0.025(2)	0.030(2)	0.019(2)
C(17)	4e	0.9251(3)	0.5676(2)	0.7777(2)	0.069(2)	0.074(2)	0.044(2)	0.005(2)	0.015(2)	0.004(2)
C(18)	4e	0.9059(3)	0.6624(2)	0.7091(2)	0.060(2)	0.060(2)	0.050(2)	0.002(2)	0.014(2)	−0.008(2)
C(19)	4e	0.8773(3)	0.5471(2)	0.5612(2)	0.060(2)	0.055(2)	0.053(2)	0.006(1)	0.029(1)	−0.002(1)
C(20)	4e	0.7833(3)	0.5324(2)	0.7784(2)	0.089(2)	0.081(2)	0.061(2)	0.010(2)	0.042(2)	0.007(2)

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