Crystal structure of 2-((1-(1-adamantyl)ethyl)iminomethyl)phenol, $C_{19}H_{25}NO$

X. Shi, Z.-X. Lü and G.-L. Zhao*

Zhejiang Normal University, Institute of Physical Chemistry, Zhejiang Key Laboratory for Reactive Chemistry on Solid Surfaces, Jinhua, Zhejiang 321004, P. R. China

Received February 23, 2006, accepted and available on-line March 29, 2006; CCDC no. 1267/1739

Abstract

$C_{19}H_{25}NO$, monoclinic, $P12_11$ (no. 4), $a = 6.4436(8)$ Å, $b = 16.438(2)$ Å, $c = 15.340(2)$ Å, $\beta = 98.536(2)^\circ$, $V = 1606.9$ Å³, $Z = 4$, $R_{gt}(F) = 0.079$, $wR_{ref}(F^2) = 0.172$, $T = 293$ K.

Source of material

A mixture of rimantadine (0.18 g, 1.0 mmol) and 2-hydroxybenzaldehyde (0.13 g, 1.0 mmol) in absolute methanol (30 ml) was stirred and refluxed for 4 h. The solution was cooled to room temperature. Yellow single crystals suitable for X-ray diffraction were obtained by slow evaporation after about 5 d.

Discussion

For many years, there has been considerable interest in the study of Schiff base compounds due to their biological activity [1,2]. Recently, we have already reported some organic complexes and metal-organic coordination complexes constructed by Schiff bases [3–5]. As an extension of our work in the structural characterization of Schiff base compounds, we synthesized the title compound and analyzed its crystal structure.

The title crystal structure contains two independent $C_{19}H_{25}NO$ units. The compound crystallizes in the phenol-imine tautomeric form with O1—C1 and O2—C20 bond lengths of 1.350(5) Å and

1.354(5) Å, respectively. The C7=N1 bond length of 1.263(5) Å and the C26=N2 bond length of 1.262(5) Å correspond to the value for a double bond [6]. The dihedral angles between two neighboring benzene rings are 62.9°. Furthermore, there are strong O—H...N intramolecular hydrogen bond involving hydroxy O1 atom and N1 atom as well as hydroxy O2 atom and N2 atom. Three C atoms, one N atom, one O atom and one H atom generate a six-membered ring through coordination and hydrogen bonds, which stabilize the three-dimensional network.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.08 × 0.12 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.71 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	50.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8590, 5617
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4467
$N(param)_{refined}$:	382
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2a	0.3166	0.3988	1.3589	0.098
H(2A)	2a	0.2748	0.2330	1.2123	0.099
H(3A)	2a	0.5546	0.1481	1.1971	0.115
H(4A)	2a	0.8761	0.1660	1.2817	0.103
H(5A)	2a	0.9233	0.2712	1.3789	0.080
H(7A)	2a	0.7967	0.3869	1.4545	0.059
H(8A)	2a	0.7039	0.4879	1.5326	0.065
H(9A)	2a	0.3999	0.4417	1.5823	0.117
H(9B)	2a	0.2654	0.5057	1.5228	0.117
H(9C)	2a	0.4295	0.5348	1.6025	0.117
H(11A)	2a	0.3465	0.5619	1.3453	0.051
H(11B)	2a	0.2663	0.6070	1.4242	0.051
H(12A)	2a	0.2712	0.6984	1.3065	0.058
H(13A)	2a	0.3402	0.7550	1.4487	0.066
H(13B)	2a	0.4645	0.8032	1.3846	0.066
H(14A)	2a	0.6890	0.7739	1.5163	0.059
H(15A)	2a	0.9652	0.7145	1.4540	0.074
H(15B)	2a	0.8483	0.7784	1.3878	0.074
H(16A)	2a	0.8967	0.6563	1.3113	0.063
H(17A)	2a	0.5271	0.6541	1.5540	0.061
H(17B)	2a	0.7676	0.6379	1.5549	0.061
H(18A)	2a	0.5910	0.7308	1.2586	0.068
H(18B)	2a	0.5458	0.6373	1.2444	0.068
H(19A)	2a	0.8926	0.5651	1.4287	0.062
H(19B)	2a	0.7305	0.5367	1.3477	0.062
H(2)	2a	0.7092	0.6732	1.0415	0.092
H(21A)	2a	0.3957	0.5142	1.0776	0.079
H(22A)	2a	0.5291	0.4062	1.1600	0.083

* Correspondence author (e-mail: sky53@zjnu.cn)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(23A)	2a	0.8778	0.4023	1.2208	0.081
H(24A)	2a	1.0900	0.5096	1.2001	0.069
H(26A)	2a	1.1555	0.6422	1.1354	0.057
H(27A)	2a	1.2382	0.7558	1.0877	0.072
H(28A)	2a	1.1221	0.7257	0.9379	0.155
H(28B)	2a	1.2278	0.8115	0.9516	0.155
H(28C)	2a	0.9827	0.8042	0.9320	0.155
H(30A)	2a	1.3483	0.9006	1.1067	0.072
H(30B)	2a	1.1938	0.9307	1.0241	0.072
H(31A)	2a	1.2691	1.0388	1.1233	0.074
H(32A)	2a	0.9225	1.0782	1.1324	0.078

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(32B)	2a	0.9322	1.0383	1.0400	0.078
H(33A)	2a	0.6477	0.9840	1.1003	0.071
H(34A)	2a	0.7102	0.9074	1.2301	0.078
H(34B)	2a	0.7840	0.9976	1.2493	0.078
H(35A)	2a	1.0464	0.9075	1.3161	0.077
H(36A)	2a	1.1668	1.0334	1.2634	0.092
H(36B)	2a	1.3329	0.9648	1.2551	0.092
H(37A)	2a	0.7268	0.8450	1.0848	0.065
H(37B)	2a	0.8101	0.8956	1.0104	0.065
H(38A)	2a	0.9749	0.7989	1.2161	0.071
H(38B)	2a	1.2135	0.8215	1.2243	0.071

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	2a	0.2747(5)	0.3559(2)	1.3132(2)	0.067(2)	0.082(2)	0.089(2)	-0.008(2)	-0.014(2)	0.006(2)
N(1)	2a	0.5253(5)	0.4334(2)	1.4315(2)	0.056(2)	0.043(2)	0.054(2)	0.005(2)	0.006(2)	0.008(2)
C(1)	2a	0.4364(7)	0.3054(3)	1.3060(3)	0.068(3)	0.052(3)	0.062(3)	-0.006(2)	0.005(2)	0.020(2)
C(2)	2a	0.4053(9)	0.2415(3)	1.2461(3)	0.101(4)	0.072(3)	0.068(3)	-0.033(3)	-0.007(3)	-0.007(3)
C(3)	2a	0.573(1)	0.1905(3)	1.2377(4)	0.142(6)	0.065(3)	0.083(4)	-0.015(4)	0.031(4)	-0.020(3)
C(4)	2a	0.766(1)	0.2011(3)	1.2875(4)	0.098(4)	0.067(3)	0.097(4)	-0.004(3)	0.027(3)	-0.015(3)
C(5)	2a	0.7922(7)	0.2635(3)	1.3453(3)	0.069(3)	0.061(3)	0.073(3)	-0.005(2)	0.020(2)	-0.006(2)
C(6)	2a	0.6307(6)	0.3166(2)	1.3566(3)	0.060(2)	0.041(2)	0.048(2)	-0.006(2)	0.018(2)	0.011(2)
C(7)	2a	0.6654(6)	0.3823(2)	1.4203(3)	0.054(2)	0.043(2)	0.050(2)	0.001(2)	0.007(2)	0.015(2)
C(8)	2a	0.5680(6)	0.4990(2)	1.4967(3)	0.057(2)	0.051(2)	0.053(3)	0.011(2)	-0.002(2)	0.005(2)
C(9)	2a	0.4003(8)	0.4949(3)	1.5566(3)	0.098(4)	0.073(3)	0.069(3)	0.018(3)	0.036(3)	0.018(3)
C(10)	2a	0.5843(5)	0.5821(2)	1.4494(2)	0.039(2)	0.041(2)	0.036(2)	0.005(2)	0.005(2)	-0.004(2)
C(11)	2a	0.3802(6)	0.6039(2)	1.3895(2)	0.043(2)	0.042(2)	0.043(2)	-0.005(2)	0.006(2)	-0.005(2)
C(12)	2a	0.4031(6)	0.6852(2)	1.3444(3)	0.045(2)	0.049(2)	0.049(2)	-0.003(2)	-0.001(2)	0.005(2)
C(13)	2a	0.4526(6)	0.7512(2)	1.4133(3)	0.050(2)	0.043(2)	0.073(3)	0.004(2)	0.016(2)	-0.004(2)
C(14)	2a	0.6574(6)	0.7312(2)	1.4718(2)	0.056(2)	0.045(2)	0.048(2)	-0.004(2)	0.008(2)	-0.019(2)
C(15)	2a	0.8338(6)	0.7266(3)	1.4166(3)	0.050(2)	0.062(3)	0.072(3)	-0.011(2)	0.008(2)	-0.012(2)
C(16)	2a	0.7835(6)	0.6597(2)	1.3473(3)	0.047(2)	0.056(3)	0.060(3)	-0.010(2)	0.028(2)	-0.013(2)
C(17)	2a	0.6369(6)	0.6504(2)	1.5172(2)	0.049(2)	0.065(3)	0.037(2)	0.008(2)	0.001(2)	-0.007(2)
C(18)	2a	0.5781(7)	0.6795(2)	1.2886(3)	0.082(3)	0.047(2)	0.043(2)	-0.012(2)	0.012(2)	-0.002(2)
C(19)	2a	0.7613(6)	0.5788(2)	1.3921(3)	0.044(2)	0.055(2)	0.056(3)	0.002(2)	0.008(2)	-0.017(2)
O(2)	2a	0.5787(5)	0.6433(2)	1.0397(2)	0.064(2)	0.066(2)	0.095(3)	0.005(2)	-0.007(2)	0.007(2)
N(2)	2a	0.9485(5)	0.7102(2)	1.0721(2)	0.061(2)	0.039(2)	0.061(2)	0.000(2)	0.013(2)	0.000(2)
C(20)	2a	0.6641(6)	0.5795(2)	1.0882(3)	0.067(3)	0.037(2)	0.046(2)	0.003(2)	0.013(2)	-0.007(2)
C(21)	2a	0.5367(7)	0.5139(3)	1.1021(3)	0.065(3)	0.061(3)	0.074(3)	-0.011(2)	0.019(2)	-0.023(3)
C(22)	2a	0.6165(9)	0.4495(3)	1.1510(3)	0.104(4)	0.046(3)	0.069(3)	-0.027(3)	0.049(3)	-0.016(2)
C(23)	2a	0.8240(9)	0.4470(3)	1.1877(3)	0.111(4)	0.042(2)	0.055(3)	0.000(3)	0.026(3)	0.006(2)
C(24)	2a	0.9496(7)	0.5110(2)	1.1749(3)	0.074(3)	0.055(3)	0.045(2)	0.004(2)	0.012(2)	-0.006(2)
C(25)	2a	0.8752(6)	0.5780(2)	1.1255(3)	0.058(2)	0.036(2)	0.040(2)	-0.006(2)	0.014(2)	-0.012(2)
C(26)	2a	1.0138(6)	0.6462(2)	1.1127(3)	0.050(2)	0.048(2)	0.048(2)	0.001(2)	0.012(2)	-0.008(2)
C(27)	2a	1.1000(7)	0.7739(2)	1.0589(3)	0.068(3)	0.047(2)	0.070(3)	-0.005(2)	0.031(2)	-0.001(2)
C(28)	2a	1.109(1)	0.7793(3)	0.9612(3)	0.180(6)	0.058(3)	0.089(4)	-0.024(3)	0.071(4)	-0.012(3)
C(29)	2a	1.0484(5)	0.8539(2)	1.1036(3)	0.044(2)	0.041(2)	0.051(2)	-0.002(2)	0.011(2)	0.006(2)
C(30)	2a	1.2068(6)	0.9198(2)	1.0868(3)	0.055(2)	0.050(2)	0.080(3)	-0.006(2)	0.024(2)	-0.003(2)
C(31)	2a	1.1677(6)	0.9974(3)	1.1354(3)	0.050(2)	0.053(2)	0.086(4)	-0.018(2)	0.019(2)	-0.006(2)
C(32)	2a	0.9474(7)	1.0278(2)	1.1028(3)	0.085(3)	0.041(2)	0.071(3)	0.000(2)	0.020(3)	0.005(2)
C(33)	2a	0.7899(6)	0.9644(2)	1.1214(3)	0.043(2)	0.054(2)	0.082(3)	0.006(2)	0.013(2)	0.007(2)
C(34)	2a	0.8119(7)	0.9481(3)	1.2186(3)	0.070(3)	0.055(3)	0.078(3)	-0.008(2)	0.038(3)	-0.010(2)
C(35)	2a	1.0319(7)	0.9181(2)	1.2526(3)	0.088(3)	0.056(3)	0.045(3)	0.003(2)	0.004(2)	0.000(2)
C(36)	2a	1.1914(7)	0.9831(3)	1.2335(3)	0.066(3)	0.076(3)	0.083(4)	0.000(2)	-0.006(3)	-0.024(3)
C(37)	2a	0.8285(6)	0.8856(2)	1.0734(3)	0.053(2)	0.050(2)	0.057(3)	-0.003(2)	0.002(2)	0.005(2)
C(38)	2a	1.0725(7)	0.8406(2)	1.2034(3)	0.066(3)	0.053(2)	0.057(3)	0.001(2)	0.006(2)	0.014(2)

Acknowledgment. This work was financially supported by the foundation of Zhejiang Key Laboratory for Reactive Chemistry on Solid Surfaces.

References

1. Golcu, A.; Tumer, M.; Demirelli, H.; Wheatley, R. A.: Cd(II) and Cu(II) complexes of polydentate Schiff base ligands: synthesis, characterization, properties and biological activity. *Inorg. Chim. Acta* 358 (2005) 1785-1797.
2. Kumar, K. N.; Ramesh, R.: Synthesis, characterization, redox property and biological activity of Ru(II) carbonyl complexes containing O,N-donor ligands and heterocyclic bases. *Spectrochim. Acta A60* (2004) 2913-2918.
3. Zhao, G.-L.; Feng, Y.-L.; Liu, X.-H.: Synthesis, Characterization, Crystal Structure and Kinetics of Thermal Decomposition of Zn(II) Complex. *Chin. J. Inorg. Chem.* 21 (2005) 598-602.

4. Zhao, G.-L.; Zhang, P.-H.; Feng, Y.-L.: Studies on the Synthesis, Characterization and Antibacterial Activity of Rare Earth Complexes with Schiff Base Derived from *o*-Vanillin and Adamantaneamine. *Chin. J. Inorg. Chem.* **21** (2005) 421-424.
5. Feng, Y.-L.; Zhao, G.-L.; Wen, Y.-H.: 4-[[1-(1-Adamantyl)ethyl]imino-methyl]phenol. *Acta Crystallogr. E* **61** (2005) o1771-o1773.
6. Fernández-G, J. M.; del Rio-Portilla, F.; Quiroz-Garcia, B.; Toscano, R. A.; Salcedo, R.: The structures of some *ortho*-hydroxy Schiff base ligands. *J. Mol. Struct.* **561** (2001) 197-207.
7. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
8. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.