

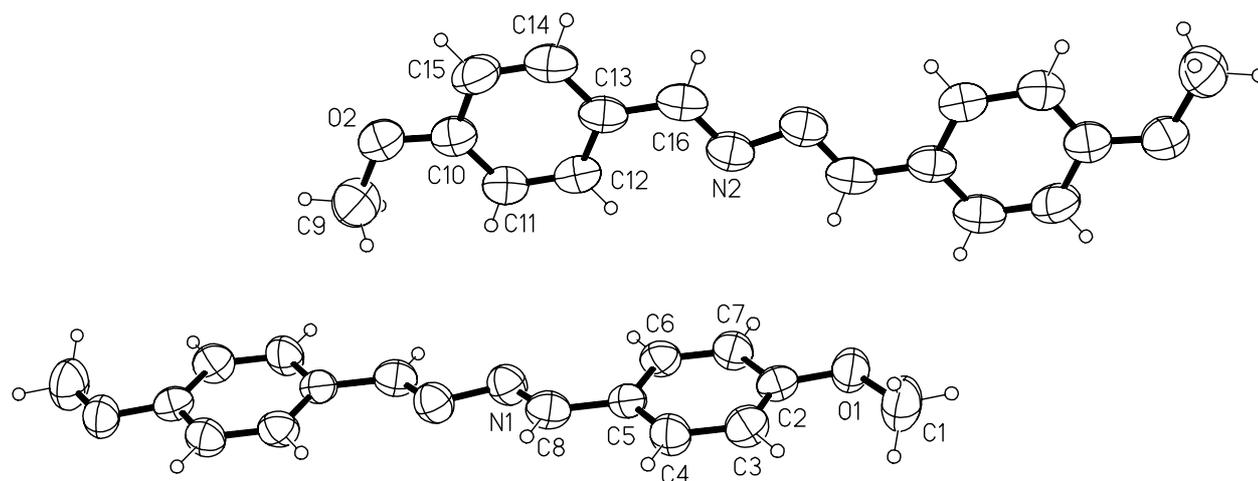
Crystal structure of *N,N'*-bis(4-methoxy-benzylidene)hydrazine, $C_{16}H_{16}N_2O_2$

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Abstract

$C_{16}H_{16}N_2O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.539(2)$ Å, $b = 9.230(2)$ Å, $c = 10.702(3)$ Å, $\alpha = 109.976(6)^\circ$, $\beta = 93.920(6)^\circ$, $\gamma = 111.081(6)^\circ$, $V = 721.9$ Å³, $Z = 2$, $R_{gt}(F) = 0.081$, $wR_{ref}(F^2) = 0.178$, $T = 298$ K.

Source of material

The title compound was prepared by the reaction of hydrazine (0.002 mol) and 4-methoxybenzaldehyde (0.002 mol) in ethanol (10 mL) was treated with concentrated HCl (0.1 mL). The desired product was obtained by filtration, drying and recrystallization from ethanol. Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethanol solution.

Discussion

Compounds with the structure of $-C=N-$ (azomethine group) are known as Schiff bases, which are usually synthesized from the condensation of primary amines and active carbonyl groups. Schiff bases are important class of compounds in medicinal and pharmaceutical field. They show biological applications including antibacterial [1–6], antifungal [3–6] and antitumor activity [7,8]. In the crystal structure of the title compound, the two benzene rings (C2→C7) as well as the other two benzene rings (C10→C15) are essentially coplanar. The bond angles of C16–C13–C14, C16–C13–C12, C6–C5–C8 and C6–C5–C4 close to 120° as well as the bond lengths of C13–C16 (1.461(6) Å) and C5–C8 (1.459(6) Å) confirm the sp^2 hybridization of the carbon atoms [9]. The C=N and N–N bond lengths ($d(C16=N2) = 1.279(5)$ Å, $d(C8=N1) = 1.264(5)$ Å, $d(N1-N1') = 1.416(6)$ Å, $d(N2-N2') = 1.417(7)$ Å) are within normal ranges for Schiff bases.

Table 1. Data collection and handling.

Crystal:	colorless block, size $0.05 \times 0.06 \times 0.33$ mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	0.83 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3788, 2499
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1677
$N(param)_{refined}$:	183
Programs:	SHELXS-97 [10], SHELXL-97 [11], SHELXTL [12]

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(1A)	2i	0.2283	0.2012	0.7926	0.127
H(1B)	2i	0.2691	0.3376	0.9404	0.127
H(1C)	2i	0.3069	0.1773	0.9170	0.127
H(3)	2i	0.3714	0.0172	0.7360	0.073
H(4)	2i	0.5189	-0.1212	0.6071	0.074
H(6)	2i	0.9147	0.3211	0.6954	0.066
H(7)	2i	0.7664	0.4580	0.8208	0.070
H(8)	2i	0.7613	-0.1243	0.5088	0.069
H(9A)	2i	1.2185	0.2388	0.2622	0.125
H(9B)	2i	1.2091	0.1022	0.1224	0.125
H(9C)	2i	1.0713	0.0567	0.2080	0.125
H(11)	2i	0.9980	0.2604	0.3687	0.069
H(12)	2i	0.8132	0.3686	0.4662	0.068
H(14)	2i	0.6451	0.3879	0.1183	0.087
H(15)	2i	0.8270	0.2752	0.0178	0.089
H(16)	2i	0.5292	0.4949	0.3160	0.072

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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
O(1)	2i	0.4755(4)	0.3463(4)	0.8657(3)	0.070(2)	0.059(2)	0.072(2)	0.028(2)	0.032(2)	0.024(2)
O(2)	2i	1.0374(5)	0.1960(4)	0.1143(3)	0.085(2)	0.085(3)	0.062(2)	0.039(2)	0.018(2)	0.034(2)
N(1)	2i	0.9728(5)	0.0599(4)	0.5424(4)	0.058(2)	0.052(3)	0.064(3)	0.021(2)	0.012(2)	0.020(2)
N(2)	2i	0.5624(5)	0.4669(5)	0.4831(4)	0.060(3)	0.061(3)	0.065(3)	0.021(2)	0.001(2)	0.028(2)
C(1)	2i	0.3060(6)	0.2584(7)	0.8801(6)	0.073(4)	0.083(4)	0.095(4)	0.033(3)	0.041(3)	0.028(3)
C(2)	2i	0.5525(5)	0.2503(6)	0.7896(4)	0.053(3)	0.056(3)	0.047(3)	0.018(2)	0.008(2)	0.024(2)
C(3)	2i	0.4804(6)	0.0786(6)	0.7273(5)	0.047(3)	0.052(3)	0.079(3)	0.010(2)	0.018(3)	0.029(3)
C(4)	2i	0.5696(6)	-0.0042(6)	0.6511(5)	0.062(3)	0.042(3)	0.079(4)	0.018(2)	0.026(3)	0.024(2)
C(5)	2i	0.7328(5)	0.0844(5)	0.6393(4)	0.053(3)	0.050(3)	0.051(3)	0.022(2)	0.014(2)	0.026(2)
C(6)	2i	0.8053(6)	0.2594(5)	0.7033(5)	0.052(3)	0.045(3)	0.066(3)	0.014(2)	0.015(2)	0.027(2)
C(7)	2i	0.7167(6)	0.3408(5)	0.7776(5)	0.062(3)	0.039(3)	0.066(3)	0.015(2)	0.017(3)	0.018(2)
C(8)	2i	0.8208(6)	-0.0081(6)	0.5555(5)	0.052(3)	0.050(3)	0.064(3)	0.013(2)	0.006(2)	0.025(2)
C(9)	2i	1.1426(6)	0.1442(7)	0.1822(5)	0.069(3)	0.102(5)	0.082(4)	0.038(3)	0.014(3)	0.038(3)
C(10)	2i	0.9331(6)	0.2578(6)	0.1834(5)	0.062(3)	0.054(3)	0.060(3)	0.014(2)	0.006(3)	0.023(3)
C(11)	2i	0.9271(6)	0.2857(5)	0.3176(5)	0.048(3)	0.055(3)	0.060(3)	0.012(2)	0.000(2)	0.025(2)
C(12)	2i	0.8163(6)	0.3509(5)	0.3757(5)	0.056(3)	0.049(3)	0.050(3)	0.007(2)	-0.001(2)	0.020(2)
C(13)	2i	0.7095(6)	0.3910(5)	0.3045(5)	0.052(3)	0.046(3)	0.066(3)	0.007(2)	0.000(3)	0.028(2)
C(14)	2i	0.7159(7)	0.3619(6)	0.1690(5)	0.079(4)	0.074(4)	0.069(4)	0.029(3)	0.002(3)	0.038(3)
C(15)	2i	0.8253(7)	0.2952(6)	0.1089(5)	0.088(4)	0.088(4)	0.057(3)	0.036(3)	0.017(3)	0.039(3)
C(16)	2i	0.5888(6)	0.4577(5)	0.3650(5)	0.056(3)	0.050(3)	0.065(3)	0.010(2)	-0.005(3)	0.027(3)

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References

- Abu-Hussen, A. A.: Synthesis and spectroscopic studies on ternary bis-Schiff base complexes having Oxygen and/or nitrogen donors. *J. Coord. Chem.* **59** (2006) 157-176.
- Sithambaram Karthikeyan, M.; Jagadeesh Parsad, D.; Poojary, B.; Subramanya, K.; Shivarama Holla, B.; Suchetha Kumari, N.: Synthesis of some novel pyrazolo[3,4-*d*]pyrimidine derivatives as potential antimicrobial agents. *Bioorg. Med. Chem.* **14** (2006) 7482-7489.
- Singh, K.; Sing Barwa, M.; Tyagi, P.: Synthesis, characterization and biological studies of Co(II), Ni(II), Cu(II), Zn(II) complexes with bidentate Schiff bases derived by heterocyclic ketone. *Eur. J. Med. Chem.* **41** (2006) 147-153.
- Panneerselvam, P.; Nair, R. R.; Vijayalakshmi, G.; Subramanian, E. H.; Sridhar, S. K.: Synthesis of Schiff bases of 4-(4-aminophenyl)-morpholine as potential antimicrobial agents. *Eur. J. Med. Chem.* **40** (2005) 225-229.
- Sridhar, S. K.; Saravan, M.; Ramesh, A.: Synthesis and antibacterial screening of hydrazones, Schiff and Mannich bases of isatin derivatives. *Eur. J. Med. Chem.* **36** (2001) 615-625.
- Pandeya, S. N.; Sriram, D.; Nath, G.; Declercq, E.: Synthesis and evaluation of Schiff and *N*-Mannich bases of isatin as potential antimicrobial agents. *Eur. J. Pharmacol.* **9** (1999) 25-31.
- Mladenova, R.; Ignatova, M.; Manolova, N.; Petrova, T.; Rashkov, I.: Preparation, characterization and biological activity of Schiff base compounds derived from 8-hydroxyquinoline-2-carboxaldehyde and Jeffamines ED. *Eur. Polym. J.* **38** (2002) 989-1000.
- Walsh, O. M.; Meegan, M. J.; Prendergast, R. M.; Nakib, T. A.: Synthesis of 3-acetoxyazetid-2-ones and 3-hydroxyazetid-2-ones with anti-fungal and antibacterial activity. *Eur. J. Med. Chem.* **31** (1996) 989-1000.
- Liu, S. L.; Dai, J. F.; Chen, Y.; Gao, J. B.; Liu, H. W.: Synthesis and crystal structure of 3-(2-hydroxy-phenyl)-5-phenyl-6-ethoxycarbonyl-cyclohex-2-enone. *Chin. J. Org. Chem.* **24** (2004) 1583-1586.
- Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
- Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
- Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 5.10. Bruker AXS, Madison, Wisconsin, USA 1998.