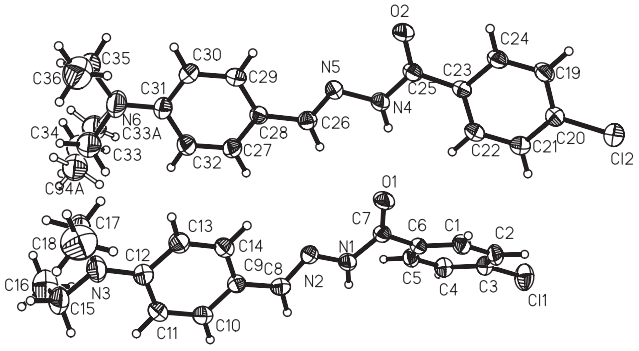
Open Access

Xue-Yue Jiang*, Lan-Lan Chen, Sen-Sen Hu, He-Yu Liu, Hua-Jie Xu and Chong-Fu Song

Crystal structure of (*E*)-4-chloro-*N*′-(4-(diethylamino)benzylidene)benzohydrazide, $C_{18}H_{20}ClN_3O$



DOI 10.1515/ncrs-2015-0187
Received August 19, 2015; accepted February 2, 2016; available online February 26, 2016

Abstract

$C_{18}H_{20}ClN_3O$, monoclinic, $P2_1/c$ (no. 14), $a = 10.0099(3)$ Å, $b = 16.2337(6)$ Å, $c = 21.0714(7)$ Å, $\beta = 91.324(3)°$, $V = 3423.1(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0381$, $wR_{ref}(F^2) = 0.1065$, $T = 291(2)$ K.

CCDC no.: 782825

Table 1: Data collection and handling.

Crystal:	yellow, block, size 0.36×0.40×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.31 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, ω scans
$2\theta_{max}$:	50.05°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	19165, 6033
$N(param)_{refined}$:	422
Programs:	SADABS [7], Bruker programs [8], SHELX [9]

***Corresponding author: Xue-Yue Jiang,** Department of chemical and materials engineering, Fuyang Normal College, Fuyang, Anhui 236041, China, e-mail: jiangxueyue@126.com
Lan-Lan Chen, Sen-Sen Hu, He-Yu Liu, Hua-Jie Xu and Chong-Fu Song: Department of chemical and materials engineering, Fuyang Normal College, Fuyang, Anhui 236041, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.9751	0.3861	0.4556	0.063
H(3)	4*e*	0.4718	0.3781	0.4835	0.064
H(4)	4*e*	1.0584	0.4005	0.6633	0.069
H(8)	4*e*	1.0355	0.4115	0.3593	0.064
H(13)	4*e*	0.6848	0.4779	0.1761	0.074
H(2)	4*e*	0.8415	0.1931	0.6565	0.076
H(24)	4*e*	0.1232	0.2480	0.5454	0.067
H(27)	4*e*	0.5620	0.4691	0.4243	0.063
H(14)	4*e*	0.7230	0.4274	0.2755	0.067
H(25)	4*e*	0.1539	0.1379	0.6121	0.072
H(33)	4*e*	0.6789	0.5679	0.3590	0.071
H(1A)	4*e*	0.7575	0.2306	0.5577	0.070
H(11)	4*e*	1.0761	0.5230	0.1608	0.076
H(10)	4*e*	1.1123	0.4688	0.2592	0.071
H(5)	4*e*	0.9778	0.4358	0.5631	0.062
H(22)	4*e*	0.5166	0.2856	0.5439	0.084
H(32)	4*e*	0.6867	0.6633	0.2803	0.081
H(29)	4*e*	0.2845	0.5490	0.3341	0.078
H(21)	4*e*	0.5474	0.1757	0.6114	0.094
H(30)	4*e*	0.2907	0.6422	0.2539	0.090
H(35A)	4*e*	0.3858	0.7961	0.1509	0.118
H(35B)	4*e*	0.2965	0.7453	0.1970	0.118
H(15A)	4*e*	1.0399	0.5512	0.0674	0.111
H(15B)	4*e*	0.9340	0.5823	0.0169	0.111
H(17A)	4*e*	0.6503	0.5520	0.1007	0.116
H(17B)	4*e*	0.7096	0.5963	0.0414	0.116
H(36A)	4*e*	0.2706	0.6958	0.0951	0.198
H(36B)	4*e*	0.4244	0.6782	0.0924	0.198
H(36C)	4*e*	0.3351	0.6274	0.1385	0.198
H(18A)	4*e*	0.6022	0.4773	0.0104	0.214
H(18B)	4*e*	0.7549	0.4699	−0.0034	0.214
H(18C)	4*e*	0.6958	0.4256	0.0558	0.214
H(16A)	4*e*	1.0537	0.6908	0.0569	0.179
H(16B)	4*e*	0.9035	0.6997	0.0753	0.179
H(16C)	4*e*	1.0094	0.6686	0.1257	0.179
H(33A)*	4*e*	0.5996	0.7437	0.1309	0.112
H(33B)*	4*e*	0.6792	0.6924	0.1824	0.112
H(34A)*	4*e*	0.7628	0.8249	0.1786	0.149
H(34B)*	4*e*	0.6214	0.8604	0.1938	0.149
H(34C)*	4*e*	0.7009	0.8091	0.2452	0.149
H(33C)*	4*e*	0.6487	0.7901	0.2494	0.112
H(33D)*	4*e*	0.5699	0.8390	0.1951	0.112
H(34D)*	4*e*	0.7657	0.7797	0.1529	0.149
H(34E)*	4*e*	0.7161	0.6924	0.1739	0.149
H(34F)*	4*e*	0.6382	0.7408	0.1203	0.149

*Disordered, occupancy factor: 0.5.

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(2)	4*e*	0.37897(7)	0.05824(4)	0.66415(4)	0.0813(5)	0.0801(5)	0.1187(6)	0.0101(4)	0.0197(4)	0.0373(5)
Cl(1)	4*e*	1.01685(8)	0.26786(5)	0.74373(3)	0.1152(6)	0.1046(5)	0.0649(4)	−0.0090(4)	−0.0154(4)	0.0243(4)
O(1)	4*e*	0.6823(1)	0.3496(1)	0.47325(7)	0.0383(9)	0.090(1)	0.068(1)	−0.0046(8)	0.0008(7)	0.0085(9)
O(2)	4*e*	0.1720(1)	0.3650(1)	0.47507(8)	0.0394(9)	0.108(1)	0.083(1)	0.0014(8)	0.0013(8)	0.030(1)
N(1)	4*e*	0.8921(2)	0.3832(1)	0.44432(8)	0.039(1)	0.065(1)	0.054(1)	−0.0017(8)	−0.0009(8)	0.008(1)
N(5)	4*e*	0.3737(2)	0.4508(1)	0.42424(8)	0.050(1)	0.061(1)	0.052(1)	−0.0013(9)	0.0048(9)	0.009(1)
N(4)	4*e*	0.3926(2)	0.3895(1)	0.46942(8)	0.042(1)	0.064(1)	0.055(1)	−0.0046(9)	−0.0007(8)	0.009(1)
N(2)	4*e*	0.8527(2)	0.4049(1)	0.38326(8)	0.048(1)	0.060(1)	0.047(1)	0.0033(9)	−0.0006(9)	0.005(1)
C(6)	4*e*	0.8590(2)	0.3367(1)	0.5499(1)	0.040(1)	0.047(1)	0.053(1)	−0.000(1)	0.006(1)	0.004(1)
C(9)	4*e*	0.9219(2)	0.4443(1)	0.2799(1)	0.053(1)	0.047(1)	0.052(1)	0.005(1)	0.007(1)	0.002(1)
C(4)	4*e*	0.9982(2)	0.3663(1)	0.6417(1)	0.061(1)	0.055(2)	0.057(2)	−0.006(1)	−0.002(1)	−0.004(1)
C(7)	4*e*	0.8019(2)	0.3577(1)	0.4862(1)	0.043(1)	0.048(1)	0.055(1)	−0.001(1)	0.003(1)	0.001(1)
C(23)	4*e*	0.3162(2)	0.2781(1)	0.5358(1)	0.040(1)	0.056(1)	0.049(1)	−0.001(1)	0.007(1)	−0.001(1)
C(8)	4*e*	0.9476(2)	0.4184(1)	0.3450(1)	0.049(1)	0.052(1)	0.059(2)	0.006(1)	0.004(1)	−0.005(1)
C(28)	4*e*	0.4811(2)	0.5466(1)	0.3561(1)	0.049(1)	0.047(1)	0.052(1)	0.001(1)	0.005(1)	0.001(1)
C(3)	4*e*	0.9561(2)	0.2940(1)	0.6688(1)	0.062(1)	0.060(2)	0.052(1)	0.001(1)	0.003(1)	0.009(1)
C(13)	4*e*	0.7714(2)	0.4781(1)	0.1930(1)	0.055(1)	0.072(2)	0.058(2)	−0.000(1)	−0.002(1)	0.003(1)
C(2)	4*e*	0.8678(2)	0.2423(1)	0.6379(1)	0.062(2)	0.058(1)	0.070(2)	−0.011(1)	0.006(1)	0.020(2)
N(3)	4*e*	0.8512(2)	0.5443(1)	0.0980(1)	0.073(2)	0.108(2)	0.065(1)	−0.015(1)	−0.003(1)	0.028(1)
C(24)	4*e*	0.2095(2)	0.2330(2)	0.5580(1)	0.041(1)	0.065(2)	0.062(2)	−0.005(1)	0.004(1)	0.001(1)
C(26)	4*e*	0.4810(2)	0.4848(1)	0.4055(1)	0.050(1)	0.053(1)	0.056(1)	0.000(1)	0.000(1)	0.000(1)
C(14)	4*e*	0.7947(2)	0.4471(1)	0.2527(1)	0.054(1)	0.057(1)	0.057(2)	−0.003(1)	0.007(1)	0.001(1)
C(19)	4*e*	0.2273(2)	0.1671(1)	0.5977(1)	0.049(1)	0.060(2)	0.071(2)	−0.009(1)	0.014(1)	0.003(1)
C(27)	4*e*	0.5999(2)	0.5830(1)	0.3380(1)	0.053(1)	0.062(2)	0.062(2)	−0.001(1)	−0.003(1)	0.008(1)
C(1)	4*e*	0.8188(2)	0.2646(1)	0.5787(1)	0.051(1)	0.057(1)	0.067(2)	−0.012(1)	−0.001(1)	0.006(1)
C(20)	4*e*	0.3541(2)	0.1445(1)	0.6160(1)	0.057(2)	0.058(1)	0.066(2)	0.004(1)	0.014(1)	0.009(1)
C(11)	4*e*	1.0037(2)	0.5044(1)	0.1837(1)	0.055(2)	0.075(2)	0.060(2)	0.001(1)	0.013(1)	0.007(1)
C(31)	4*e*	0.4901(2)	0.6645(2)	0.2571(1)	0.071(2)	0.062(2)	0.075(2)	0.002(1)	0.003(1)	0.020(2)
N(6)	4*e*	0.4928(2)	0.7221(2)	0.2095(1)	0.090(1)	0.127(2)	0.116(2)	−0.012(1)	−0.008(1)	0.064(2)
C(25)	4*e*	0.2869(2)	0.3480(1)	0.4912(1)	0.043(1)	0.067(2)	0.048(1)	−0.004(1)	0.003(1)	0.001(1)
C(10)	4*e*	1.0252(2)	0.4722(1)	0.2430(1)	0.052(1)	0.067(2)	0.060(2)	0.007(1)	0.004(1)	0.002(1)
C(5)	4*e*	0.9496(2)	0.3873(1)	0.5820(1)	0.056(1)	0.046(1)	0.053(1)	−0.005(1)	0.007(1)	0.004(1)
C(12)	4*e*	0.8747(2)	0.5100(1)	0.1566(1)	0.062(2)	0.061(2)	0.059(2)	0.002(1)	0.005(1)	0.004(1)
C(22)	4*e*	0.4430(2)	0.2557(2)	0.5570(1)	0.042(1)	0.081(2)	0.088(2)	−0.003(1)	0.012(1)	0.023(2)
C(32)	4*e*	0.6050(2)	0.6402(2)	0.2905(1)	0.058(2)	0.070(2)	0.075(2)	−0.009(1)	0.004(1)	0.017(2)
C(29)	4*e*	0.3662(2)	0.5714(2)	0.3231(1)	0.046(1)	0.076(2)	0.073(2)	0.003(1)	0.008(1)	0.012(2)
C(21)	4*e*	0.4617(2)	0.1897(2)	0.5971(1)	0.046(1)	0.090(2)	0.100(2)	0.007(1)	0.008(1)	0.034(2)
C(30)	4*e*	0.3698(2)	0.6278(2)	0.2751(1)	0.058(2)	0.089(2)	0.078(2)	0.014(1)	0.001(1)	0.028(2)
C(35)	4*e*	0.3738(3)	0.7424(2)	0.1702(1)	0.0956(9)	0.098(1)	0.100(1)	0.0078(9)	−0.0007(8)	0.0322(8)
C(15)	4*e*	0.9585(3)	0.5830(2)	0.0617(1)	0.100(2)	0.111(3)	0.066(2)	0.003(2)	−0.003(2)	0.026(2)
C(17)	4*e*	0.7167(3)	0.5477(2)	0.0680(1)	0.096(2)	0.117(3)	0.077(2)	−0.004(2)	−0.004(2)	0.029(2)
C(36)	4*e*	0.3488(3)	0.6805(2)	0.1196(2)	0.137(2)	0.128(2)	0.131(2)	−0.006(2)	−0.013(1)	0.009(1)
C(18)	4*e*	0.6901(4)	0.4737(2)	0.0293(2)	0.122(3)	0.149(3)	0.156(4)	−0.024(2)	−0.019(3)	−0.014(3)
C(16)	4*e*	0.9834(3)	0.6678(2)	0.0816(2)	0.134(3)	0.122(3)	0.102(3)	−0.021(2)	−0.009(2)	0.016(2)
C(33)*	4*e*	0.6183(4)	0.7383(4)	0.1761(3)	0.094(1)	0.094(1)	0.091(1)	−0.0005(9)	0.0016(9)	0.0042(9)
C(34)*	4*e*	0.6814(6)	0.8149(3)	0.2006(3)	0.096(2)	0.102(2)	0.100(2)	−0.009(1)	−0.006(2)	0.008(2)
C(33A)*	4*e*	0.6025(6)	0.7853(4)	0.2085(3)	0.094(1)	0.094(1)	0.091(1)	−0.0005(9)	0.0016(9)	0.0042(9)
C(34A)*	4*e*	0.6884(6)	0.7460(4)	0.1595(3)	0.096(2)	0.102(2)	0.100(2)	−0.009(1)	−0.006(2)	0.008(2)

*Disordered, occupancy factor: 0.5.

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents and solvents were used as obtained commercially and without further purification. 4-Diethylamino benzaldehyde (0.177 g, 1 mmol) and 4-chlorobenzohydrazide

(0.170 g, 1 mmol) were stirred in ethanol (20 mL). The mixture was refluxed for 3 h to give a clear yellow solution, then cooled to room temperature, and the resulting yellow compound was filtered, washed three with ethanol. Yellow block-shaped crystals of the title compound were obtained by slow evaporation of an ethanol solution in air after 10 days.

Experimental details

One ethyl group is disordered over two sites in a 0.5:0.5 ratio. All H-atoms were placed in calculated positions and treated as riding: C—H = 0.93 or 0.97 Å, N—H = 0.86 Å with $U_{iso}(H) = 1.2$ U_{eq}(parent C, N-atom) and 1.5 U_{eq}(C35).

Discussion

Schiff base compounds have been of great interest for many years. They play an important role in the development of coordination chemistry related to catalytic [1], pharmacological [2], antitumor activity [3] and as fluorescent sensors [4]. As an extension of work on the structural characterization of Schiff base compounds [5, 6], we here report the crystal structure of the Schiff base compound (*E*)-4-chloro-*N*′-(4-(diethylamino)benzylidene). The asymmetric unit of the title compound consists of two crystallographically independent molecules (Figure). In both independent molecules, the C = N bond adopt *E* configurations. The central moiety in both molcules is not exactly planar with torsion angles C7—N1—N2—C8 and C25—N4—N5—C26 of 172.0(4)° and 178.3(4)°, respectively. The dihedral angles between the benzene rings mean planes is 45.1(1)° and 169.0(1)°, respectively.

Acknowledgements: This project is supported by the Innovation Training Program for the College students (Grant No: AH201310371008, 201310371018), Educational Commission of Anhui Province of China (2014KJ016), and Anhui Provincial Key Laboratory for Degradation & Monitoring of Pollution of the Environment (No. 2012HJJC03) and the Natural Science Foundation of Fuyang Normal College (2014FSKJ06, 2015FSKJ02ZD).

References

1. Boghaei, D. M.; Bezaatpour, A.; Behzad, M.: Synthesis, characterization and catalytic activity of novel monomeric and polymeric vanadyl Schiff base complexes. J. Mol. Catal. **A245** (2006) 12–16.
2. Silva, Y. K. C.; Augusto, C. V.; Barbosa, M. L. C.; Melo, G. M. A.; Queiroz, A. C.; Dias, T. L. M. F.; Bispo-Junior, W.; Barreiro, E. J.; Lima, L. M.; Alexandre-Moreira, M. S.: Synthesis and pharmacological evaluation of pyrazine *N*-acylhydrazone derivatives designed as novel analgesic and anti-inflammatory drug candida. Bioorg. Med. Chem. **18** (2010) 5007–5015.
3. Yang, Z. Y.; Yang, R. D.; Li, F. S.; Yu, K. B.: Crystal structure and antitumor activity of some rare earth metal complexes with Schiff base. Polyhedron. **19** (2000) 2599–2604.
4. Zhao, Y. G.; Zhang, B. G.; Duan, C. Y.; Lin, Z. H.; Meng, Q. J.: A highly selective fluorescent sensor for fluoride through ESPT signaling transduction. New J. Chem. **30** (2006) 1207–1213.
5. Xu, H. J.; Du, N. N.; Jiang, X. Y.; Sheng, L. Q.; Tian, Y. P.: (*E*)-2-Hydroxynaphthalene-1-carbaldehyde semicarbazone. Acta Crystallogr. **E65** (2009) o1047.
6. Mao, F. L.; Dai, C. H.: (*E*)-N-(3-Hydroxybenzylidene)-3-nitrobenzohydrazide. Acta Crystallogr. **E67** (2011) o7.
7. Sheldrick, G. M.: SADABS. University of Göttingen, Germany 1996.
8. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA. 2007.
9. Sheldrick, G. M.: A short history of SHELXTL. Acta Cryst. **A64** (2008) 112–122.