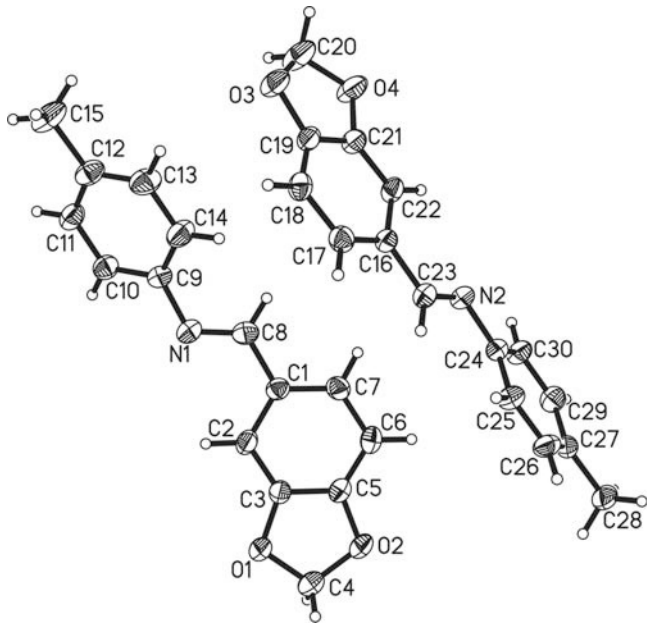
© by Oldenbourg Wissenschaftsverlag, München

Crystal structure of *N*-(benzo[d][1,3]dioxol-5-ylmethyl)-4-methylaniline, $C_{15}H_{13}NO_2$

Zhi-Gang Yin*, Zong-Lei Fei, Heng-Yu Qian and Chun-Xia Zhang

Key Laboratory of Surface and Interface Science of Henan, Zhengzhou University of Light Industry, School of Materials & Chemical Engineering, Zhengzhou 450002, P. R. China

Received February 2, 2010, accepted and available on-line February 21, 2010; CCDC no. 1267/2923



Abstract

$C_{15}H_{13}NO_2$, triclinic, $P\bar{1}$ (no. 2), a = 10.697(3) Å, b = 10.791(3) Å, c = 13.328(3) Å, α = 89.39(2)°, β = 67.05(2)°, γ = 62.45(3)°, V = 1228.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.094, T = 293 K.

Source of material

Benzo[d][1,3]dioxole-5-carbaldehyde (2 mmol, 0.30 g) was dissolved in anhydrous methanol (10 ml), the mixture was stirred for several minitutes at room temperature, *p*-toluidine (2 mmol, 0.214 g) in methanol (5 ml) was added dropwise and the mixture was stirred at room temperature for 2 h. The Schiff base was obtained and reduced with sodium borohydride (5 mmol, 0.185 g), The mixture was stirred for 5 h at RT, then evaporated to dryness, the product was recrystallised from methanol, colorless single crystals of the title compound were obtained after 1 d.

Discussion

The properties and molecular structure of complexes containing the amine group are of interest as their complexes with transition metal exhibit superior activity as catalysts in polymerizations [1,2].

The asymmetric unit of the title compound is built up from two independent but similar molecules. The title molecule crystallizes in the *E* conformation in each molecule, with the C9–N1–C8–C1 and C24–N2–C23–C16 torsion angles of 172.6(2) and –178.9(2)°, 1,3-dioxolane ring is almost co-planar with its attached benzene ring [dihedral angle = 1.3(1)° and 3.2(2)°], respectively. Each molecule is not planar, the dihedral angles between the two benzene ring are 42.4(1)° and 19.7(1)° in each molecule, respectively. The N1—C8 and N2—C23 bond distances of 1.273(2) and 1.271(2) Å are typical for the C=N double bond. Classic hydrogen bonds were not observed in the molecule.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.23 × 0.26 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.87 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω
$2\theta_{max}$:	52.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9034, 5023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1831
$N(param)_{refined}$:	325
Programs:	SHAELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(22A)	2*i*	–0.0959	0.6998	0.2964	0.057
H(25A)	2*i*	0.4354	0.3790	–0.0315	0.061
H(8A)	2*i*	0.1834	0.8477	0.3559	0.063
H(26A)	2*i*	0.6428	0.1493	–0.0875	0.067
H(18A)	2*i*	–0.0806	1.0790	0.1276	0.062
H(17A)	2*i*	0.1328	0.8464	0.0543	0.063
H(23A)	2*i*	0.2685	0.5932	0.0607	0.059
H(11A)	2*i*	–0.0470	1.4083	0.5509	0.071
H(30A)	2*i*	0.2486	0.2984	0.2674	0.065
H(6A)	2*i*	0.5625	0.3710	0.1978	0.067
H(2B)	2*i*	0.5710	0.7918	0.2706	0.062
H(7A)	2*i*	0.3289	0.5914	0.2848	0.064
H(28A)	2*i*	0.7026	–0.1168	0.0765	0.100
H(28B)	2*i*	0.8095	–0.0639	–0.0062	0.100
H(28C)	2*i*	0.7171	–0.1149	–0.0451	0.100
H(29A)	2*i*	0.4592	0.0701	0.2113	0.069
H(14A)	2*i*	0.0337	1.0507	0.3071	0.079
H(10A)	2*i*	0.1737	1.1871	0.4972	0.066
H(13A)	2*i*	–0.1826	1.2751	0.3575	0.082
H(15A)	2*i*	–0.2755	1.5783	0.5371	0.131
H(15B)	2*i*	–0.3721	1.5138	0.5200	0.131
H(15C)	2*i*	–0.2697	1.5534	0.4194	0.131
H(4A)	2*i*	1.0066	0.3590	0.1656	0.090
H(4B)	2*i*	1.0233	0.3798	0.0449	0.090
H(20C)	2*i*	–0.5385	1.1470	0.4162	0.100
H(20A)	2*i*	–0.4387	1.1529	0.475	0.100

* Correspondence author (e-mail: yinck2010@yahoo.com.cn)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(2)	2*i*	0.1841(2)	0.5018(2)	0.1682(2)	0.043(1)	0.039(1)	0.047(1)	–0.014(1)	–0.0148(9)	0.003(1)
O(2)	2*i*	0.8491(2)	0.3439(2)	0.1312(1)	0.053(1)	0.035(1)	0.085(1)	–0.0090(9)	–0.026(1)	–0.0027(9)
O(3)	2*i*	–0.3459(2)	1.1472(2)	0.3129(2)	0.060(1)	0.039(1)	0.078(1)	–0.0095(9)	–0.025(1)	0.011(1)
C(21)	2*i*	–0.2121(3)	0.9102(2)	0.3022(2)	0.047(2)	0.043(2)	0.048(2)	–0.019(1)	–0.016(1)	0.008(1)
N(1)	2*i*	0.2721(2)	0.9647(2)	0.3589(2)	0.050(1)	0.045(1)	0.049(1)	–0.015(1)	–0.018(1)	0.003(1)
C(24)	2*i*	0.3193(2)	0.3645(2)	0.1253(2)	0.040(1)	0.039(1)	0.046(2)	–0.018(1)	–0.016(1)	0.006(1)
C(22)	2*i*	–0.0907(2)	0.7741(2)	0.2624(2)	0.053(2)	0.036(1)	0.048(2)	–0.021(1)	–0.017(1)	0.008(1)
C(16)	2*i*	0.0431(2)	0.7505(2)	0.1679(2)	0.042(1)	0.039(1)	0.041(2)	–0.018(1)	–0.016(1)	0.004(1)
O(4)	2*i*	–0.3549(2)	0.9586(2)	0.3932(1)	0.055(1)	0.045(1)	0.074(1)	–0.0091(9)	–0.003(1)	0.0047(9)
C(25)	2*i*	0.4392(2)	0.3175(2)	0.0181(2)	0.046(1)	0.045(2)	0.047(2)	–0.015(1)	–0.018(1)	0.010(1)
C(8)	2*i*	0.2767(2)	0.8474(3)	0.3382(2)	0.045(2)	0.056(2)	0.046(2)	–0.021(1)	–0.017(1)	0.011(1)
C(26)	2*i*	0.5643(3)	0.1793(3)	–0.0151(2)	0.042(1)	0.057(2)	0.047(2)	–0.015(1)	–0.010(1)	–0.002(1)
O(1)	2*i*	0.8531(2)	0.5520(2)	0.1623(2)	0.046(1)	0.043(1)	0.132(2)	–0.0137(9)	–0.026(1)	–0.014(1)
C(19)	2*i*	–0.2081(3)	1.0220(2)	0.2540(2)	0.047(2)	0.037(1)	0.054(2)	–0.014(1)	–0.025(1)	0.003(1)
C(1)	2*i*	0.4259(3)	0.7108(2)	0.2870(2)	0.044(1)	0.045(2)	0.041(2)	–0.020(1)	–0.014(1)	0.008(1)
C(18)	2*i*	–0.0824(3)	1.0034(2)	0.1610(2)	0.062(2)	0.041(2)	0.058(2)	–0.028(1)	–0.028(1)	0.017(1)
C(17)	2*i*	0.0445(3)	0.8638(2)	0.1182(2)	0.054(2)	0.053(2)	0.050(2)	–0.030(1)	–0.019(1)	0.010(1)
C(3)	2*i*	0.7015(3)	0.5801(2)	0.2038(2)	0.047(2)	0.043(2)	0.060(2)	–0.021(1)	–0.026(1)	0.006(1)
C(5)	2*i*	0.6989(3)	0.4566(2)	0.1842(2)	0.052(2)	0.039(2)	0.051(2)	–0.018(1)	–0.022(1)	0.007(1)
C(23)	2*i*	0.1806(3)	0.6074(2)	0.1236(2)	0.048(2)	0.045(2)	0.047(2)	–0.020(1)	–0.016(1)	0.001(1)
C(11)	2*i*	–0.0304(3)	1.3374(2)	0.5002(2)	0.061(2)	0.045(2)	0.053(2)	–0.020(2)	–0.015(1)	0.003(1)
C(30)	2*i*	0.3288(3)	0.2695(2)	0.1957(2)	0.051(2)	0.044(2)	0.047(2)	–0.017(1)	–0.010(1)	0.004(1)
C(6)	2*i*	0.5635(3)	0.4546(2)	0.2122(2)	0.069(2)	0.041(2)	0.060(2)	–0.029(2)	–0.029(1)	0.012(1)
C(27)	2*i*	0.5765(2)	0.0847(2)	0.0556(2)	0.042(2)	0.040(1)	0.064(2)	–0.017(1)	–0.027(1)	0.006(1)
C(2)	2*i*	0.5678(3)	0.7094(2)	0.2562(2)	0.054(2)	0.036(2)	0.059(2)	–0.017(1)	–0.024(1)	0.004(1)
C(9)	2*i*	0.1283(3)	1.0951(2)	0.3958(2)	0.041(1)	0.046(2)	0.046(2)	–0.016(1)	–0.015(1)	0.006(1)
C(7)	2*i*	0.4245(3)	0.5873(2)	0.2647(2)	0.050(2)	0.052(2)	0.058(2)	–0.030(1)	–0.020(1)	0.018(1)
C(12)	2*i*	–0.1380(3)	1.3674(3)	0.4579(2)	0.054(2)	0.049(2)	0.049(2)	–0.014(1)	–0.015(1)	0.016(1)
C(28)	2*i*	0.7141(2)	–0.0666(2)	0.0167(2)	0.050(2)	0.046(2)	0.088(2)	–0.009(1)	–0.034(1)	–0.002(1)
C(29)	2*i*	0.4555(3)	0.1318(3)	0.1619(2)	0.062(2)	0.049(2)	0.058(2)	–0.025(1)	–0.027(1)	0.016(1)
C(14)	2*i*	0.0196(3)	1.1227(3)	0.3553(2)	0.064(2)	0.052(2)	0.067(2)	–0.013(2)	–0.034(2)	–0.004(1)
C(10)	2*i*	0.1019(3)	1.2038(2)	0.4688(2)	0.053(2)	0.053(2)	0.048(2)	–0.022(1)	–0.018(1)	0.004(1)
C(13)	2*i*	–0.1111(3)	1.2581(3)	0.3863(2)	0.060(2)	0.066(2)	0.070(2)	–0.016(2)	–0.040(2)	0.014(2)
C(15)	2*i*	–0.2766(3)	1.5170(2)	0.4862(2)	0.079(2)	0.052(2)	0.083(2)	–0.004(2)	–0.026(2)	0.017(2)
C(4)	2*i*	0.9488(3)	0.4011(2)	0.1220(2)	0.052(2)	0.045(2)	0.102(2)	–0.012(2)	–0.025(2)	0.001(2)
C(20)	2*i*	–0.4328(3)	1.1108(3)	0.4082(2)	0.072(2)	0.045(2)	0.080(2)	–0.007(2)	–0.012(2)	0.009(2)

References

1. Gao, H. Y.; Guo, W. J.; Bao, F.; Gui, G. Q.; Zhang, J. K.; Zhu, F. M.; Wu, Q.: Synthesis, Molecular Structure, and Solution-Dependent Behavior of Nickel Complexes Chelating Anilido-Imine Donors and Their Catalytic Activity toward Olefin Polymerization. Organometallics **23** (2004) 6273-6280.
2. Gui, G. Q.; Bao, F.; Gao, H. Y.; Zhu, F. M.; Wu, Q.: Synthesis, structure and olefin polymerization catalysis of nickel(II) complexes bearing N,O-chelate ligands. J. Coord. Chem. **59** (2006) 107-118.
3. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.