Crystal structure of bis(acetylacetone)-*p*-naphthalenediimine, $C_{20}H_{22}N_2O_2$

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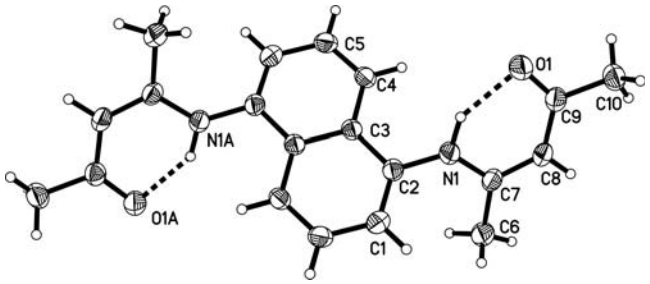
Received December 23, 2010, accepted and available on-line January 24, 2011; CCDC no. 1267/3342



Abstract

$C_{20}H_{22}N_2O_2$, monoclinic, $P12_1/c1$ (no. 14), a = 8.196(2) Å, b = 9.078(2) Å, c = 12.235(3) Å, β = 109.57(1)°, V = 857.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.053, $wR_{ref}(F^2)$ = 0.146, T = 298 K.

Source of material

To a solution of 1,5-naphthalenediamine (1.58 g, 10 mmol) and pentane-2,4-dione (4.01 g, 40 mmol) in ethanol (20 ml) was added two drops of acetic acid. The resulting mixture was refluxed for a period of 4 h. After cooling to room temperature, the reaction mixture was filtered and subsequently washed by ethanol to afford the title compound (2.42 g, yield 75.2 %). Crystals suitable for single crystal X-ray diffraction were obtained by slow evaporation of a solution in trichloromethane at room temperature in a yield of 46.5 %.

Experimental details

H atoms attached to N atoms were initially located in a difference Fourier map and restrained with U_{iso}(H) = 1.2 U_{eq}(N). Other H atoms were placed in idealized positions and treated as riding with d(C—H) = 0.96 Å (CH_3), U_{iso}(H) = 1.5 U_{eq}(C) and d(C—H) = 0.93 Å (CH), U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

Much attention has been drawn to the Schiff bases because of their potential applications in areas such as drugs, catalysis and supramolecular functional materials [1-4]. Schiff bases usually act as multi-dentate ligands in the construction of intriguing frameworks driven by coordination interactions [5-7].

All bond lengths and angles in the title crystal structure are in normal ranges. The bond length d(C8—C9) = 1.416(3) Å is shorter than C—C (d(C6—C7) = 1.493(3) Å, d(C9—C10) = 1.497(3) Å), but longer than C=C (d(C7=C8) = 1.371(3) Å). This is because of the electron delocalization effect of the conjugated structure. The intramolecular hydrogen bond N1–H···O1 connects the corresponding atoms (N1, C7, C8, C9, O1) to form a co-planar six-membered ring. The dihedral angle between N1/C7/C8/C9/O1 plane and naphthalene plane is 43.7°. There are two nitrogen atoms and two oxgen atoms in the title molecule which can coordinate the metal ions.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.24 × 0.24 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4346, 1599
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1345
$N(param)_{refined}$:	112
Programs:	SHELXS-97, SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.6556	0.3223	0.1435	0.067
H(4)	4*e*	0.2235	–0.0319	0.0626	0.061
H(5)	4*e*	0.1654	–0.2428	–0.0437	0.068
H(6A)	4*e*	0.4616	0.4958	0.1443	0.105
H(6B)	4*e*	0.2650	0.5387	0.1022	0.105
H(6C)	4*e*	0.3357	0.4333	0.0269	0.105
H(8)	4*e*	0.1572	0.4371	0.2379	0.065
H(10A)	4*e*	–0.0464	0.1778	0.3482	0.099
H(10B)	4*e*	–0.0327	0.3466	0.3258	0.099
H(10C)	4*e*	0.0982	0.2729	0.4365	0.099
H(1A)	4*e*	0.3333	0.1296	0.1947	0.064

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}
C(1)	4*e*	0.6292(3)	0.2361(2)	0.1000(2)	0.061(1)	0.045(1)	0.062(1)	–0.0022(8)	0.0211(9)	–0.0045(9)
C(2)	4*e*	0.4849(2)	0.1575(2)	0.0949(2)	0.055(1)	0.0419(9)	0.047(1)	0.0075(7)	0.0218(8)	0.0057(7)
C(3)	4*e*	0.4441(2)	0.0239(2)	0.0304(1)	0.0475(9)	0.0388(9)	0.0440(9)	0.0050(7)	0.0194(7)	0.0086(7)
C(4)	4*e*	0.2972(2)	–0.0619(2)	0.0234(2)	0.052(1)	0.048(1)	0.060(1)	0.0040(8)	0.0288(9)	0.0077(8)
C(5)	4*e*	0.2628(2)	–0.1878(2)	–0.0401(2)	0.052(1)	0.049(1)	0.073(1)	–0.0052(8)	0.026(1)	0.0031(9)

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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(6)	4*e*	0.3461(3)	0.4614(2)	0.1046(2)	0.094(2)	0.046(1)	0.083(2)	0.006(1)	0.048(1)	0.004(1)
C(7)	4*e*	0.3087(2)	0.3316(2)	0.1673(2)	0.060(1)	0.0415(9)	0.047(1)	0.0018(8)	0.0189(8)	–0.0052(7)
C(8)	4*e*	0.2011(3)	0.3442(2)	0.2318(2)	0.065(1)	0.044(1)	0.058(1)	0.0043(8)	0.027(1)	–0.0088(8)
C(9)	4*e*	0.1518(2)	0.2262(2)	0.2898(2)	0.054(1)	0.056(1)	0.050(1)	0.0002(8)	0.0195(9)	–0.0072(8)
C(10)	4*e*	0.0320(3)	0.2588(3)	0.3560(2)	0.062(1)	0.076(1)	0.069(1)	–0.005(1)	0.033(1)	–0.011(1)
N(1)	4*e*	0.3757(2)	0.1991(2)	0.1575(1)	0.071(1)	0.0431(8)	0.058(1)	0.0063(7)	0.0356(8)	0.0039(7)
O(1)	4*e*	0.2008(2)	0.0972(2)	0.2869(1)	0.089(1)	0.0547(9)	0.084(1)	0.0081(7)	0.0517(9)	0.0070(7)

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