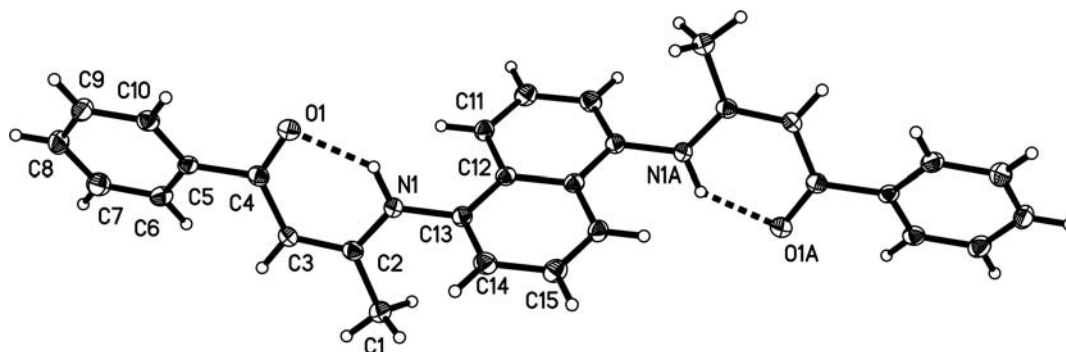


Crystal structure of bis(1-benzoylacetone)-*p*-naphthalenediimine, $C_{30}H_{26}N_2O_2$

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Abstract

$C_{30}H_{26}N_2O_2$, monoclinic, $P12_1/c1$ (no. 14), $a = 7.462(2)$ Å, $b = 16.556(6)$ Å, $c = 9.607(3)$ Å, $\beta = 104.205(4)^\circ$, $V = 1150.6$ Å³, $Z = 2$, $R_{gt}(F) = 0.043$, $wR_{ref}(F^2) = 0.103$, $T = 298$ K.

Source of material

Two drops of acetic acid was added to a solution of 1,5-naphthalenediamine (1.58 g, 10 mmol) and 1-benzoylacetone (3.24 g, 20 mmol) in ethanol (20 ml). The resulting mixture was refluxed for a period of 5 h. After cooling to room temperature, the reaction mixture was filtered and subsequently washed with ethanol to afford the title compound (3.51 g, yield 78.5 %). Crystals suitable for single crystal X-ray diffraction were obtained by slow evaporation of a solution in dichloromethane at room temperature.

Experimental details

H atoms attached to N atoms were initially located in a difference Fourier map and refined freely. Other H atoms were placed in idealized positions and treated as riding, with $d(C-H) = 0.96$ Å, $U_{iso}(H) = 1.5 U_{eq}(C)$ for methyl group and $d(C-H) = 0.93$ Å (CH), $U_{iso}(H) = 1.2 U_{eq}(C)$ for aromatic group.

Discussion

Schiff bases are of interest mainly due to their important role in the supramolecular chemistry as they usually act as multidentate ligands in the synthesis of coordination compounds with potential applications, such as catalysis, selective absorption, magnetics, and optics [1–5]. The title Schiff base derived from β -diketones can easily coordinate with metal ions in (N, O) fashion.

All bond lengths and angles in the title crystal structure are in normal ranges. The torsion angles are $N1-C2-C3-C4 = 2.0(2)^\circ$, $C12-C13-N1-C2 = 115.1(2)^\circ$ and $C3-C4-C5-C6 = -28.5(2)^\circ$.

The aromatic ring (C5/C6/C7/C8/C9/C10) and the naphthalene one form a dihedral angle of 86.1° . The intramolecular hydrogen bond consists of atom O1 and bond N1–H, with $d(H\cdots O1) = 1.864(1)$ Å, $d(N1-H\cdots O1) = 2.633(2)$ Å, and $\angle N1-H\cdots O1$ is 138.4° .

Table 1. Data collection and handling.

Crystal:	yellow block, size $0.20 \times 0.25 \times 0.30$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	51°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	5835, 2132
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1566
$N(\text{param})_{\text{refined}}$:	156
Programs:	SHELXS-97, SHELXL-97 [6]

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)	4e	0.4837	0.2737	0.3714	0.055
H(1B)	4e	0.4797	0.1837	0.4211	0.055
H(1C)	4e	0.6249	0.2440	0.5110	0.055
H(3)	4e	0.2412	0.1545	0.5260	0.037
H(6)	4e	−0.0379	0.1007	0.4621	0.041
H(7)	4e	−0.2597	0.0067	0.4774	0.050
H(8)	4e	−0.3921	0.0079	0.6709	0.052
H(9)	4e	−0.3095	0.1064	0.8454	0.048
H(10)	4e	−0.0932	0.2028	0.8279	0.040
H(11)	4e	0.1634	0.4380	0.4316	0.038
H(14)	4e	0.7251	0.3434	0.7043	0.042
H(15)	4e	0.9336	0.4460	0.6915	0.044
H(1)	4e	0.2777	0.3474	0.6378	0.051(6)

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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
C(1)	4e	0.5009(2)	0.2386(1)	0.4531(2)	0.036(1)	0.035(1)	0.0412(9)	−0.0003(8)	0.0139(8)	−0.0017(8)
C(2)	4e	0.3674(2)	0.2609(1)	0.5401(2)	0.0306(9)	0.028(1)	0.0282(8)	0.0048(7)	0.0065(7)	0.0032(7)
C(3)	4e	0.2378(2)	0.2066(1)	0.5614(2)	0.0338(9)	0.023(1)	0.0373(9)	0.0003(7)	0.0117(7)	−0.0008(7)
C(4)	4e	0.0988(2)	0.2252(1)	0.6345(2)	0.0295(9)	0.028(1)	0.0276(8)	0.0046(7)	0.0042(7)	0.0038(7)
C(5)	4e	−0.0406(2)	0.1618(1)	0.6438(2)	0.0226(8)	0.030(1)	0.0308(8)	0.0054(7)	0.0059(6)	0.0052(7)
C(6)	4e	−0.0927(2)	0.1023(1)	0.5391(2)	0.0279(9)	0.038(1)	0.0372(9)	0.0026(8)	0.0080(7)	−0.0027(8)
C(7)	4e	−0.2247(2)	0.0457(1)	0.5486(2)	0.033(1)	0.038(1)	0.053(1)	−0.0021(8)	0.0065(8)	−0.0064(9)
C(8)	4e	−0.3048(2)	0.0467(1)	0.6636(2)	0.030(1)	0.037(1)	0.063(1)	−0.0031(8)	0.0119(9)	0.009(1)
C(9)	4e	−0.2551(2)	0.1055(1)	0.7681(2)	0.035(1)	0.043(1)	0.045(1)	0.0030(9)	0.0162(8)	0.0095(9)
C(10)	4e	−0.1249(2)	0.1630(1)	0.7579(2)	0.0315(9)	0.034(1)	0.0339(9)	0.0059(8)	0.0076(7)	0.0039(8)
C(11)	4e	0.2482(2)	0.4788(1)	0.4292(2)	0.0271(9)	0.032(1)	0.0344(9)	−0.0045(7)	0.0066(7)	−0.0040(8)
C(12)	4e	0.4360(2)	0.4685(1)	0.5028(2)	0.0269(8)	0.029(1)	0.0243(7)	0.0007(7)	0.0085(6)	−0.0043(7)
C(13)	4e	0.5013(2)	0.3980(1)	0.5830(2)	0.0318(9)	0.029(1)	0.0305(8)	−0.0016(7)	0.0121(7)	−0.0021(7)
C(14)	4e	0.6840(2)	0.3901(1)	0.6522(2)	0.035(1)	0.034(1)	0.0355(9)	0.0054(8)	0.0059(7)	0.0054(8)
C(15)	4e	0.8094(2)	0.4522(1)	0.6450(2)	0.0266(9)	0.043(1)	0.0364(9)	0.0005(8)	0.0015(7)	0.0008(8)
N(1)	4e	0.3721(2)	0.33621(8)	0.5930(1)	0.0337(8)	0.0260(9)	0.0412(8)	−0.0004(6)	0.0180(6)	0.0011(7)
O(1)	4e	0.0875(2)	0.29252(7)	0.6920(1)	0.0393(7)	0.0321(8)	0.0458(7)	0.0014(5)	0.0170(5)	−0.0036(6)

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