



## Synthesis and Structural Characterization of (*E*)-*N*-(4-(Diethylamino)benzylidene)naphthalen-1-amine

LI JIAN

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, P.R. China

Corresponding author: Fax: +86 536 8877561; Tel: +86 536 8877561; E-mail: ljwfu@163.com

(Received: 8 December 2010;

Accepted: 15 September 2011)

AJC-10408

The compound (*E*)-*N*-(4-(diethylamino)benzylidene)naphthalen-1-amine (C<sub>21</sub>H<sub>22</sub>N<sub>2</sub>, M<sub>r</sub> = 302.41) was synthesized and characterized by elemental analysis, IR spectra, <sup>1</sup>H NMR spectra and single crystal X-ray diffraction. The crystal belongs to monoclinic, space group P2<sub>1</sub>/c, with a = 24.517(2), b = 7.5266(8), c = 20.1621(19) Å, β = 109.9680(10)°, V = 3496.8(6) Å<sup>3</sup>, Z = 8, D<sub>c</sub> = 1.149 g/cm<sup>3</sup>, λ = 0.71073 Å, μ(MoK<sub>α</sub>) = 0.067 mm<sup>-1</sup>, F(000) = 1296. The final refinement gave R = 0.0620, wR(F<sup>2</sup>) = 0.1374 for 6,145 observed reflections with I > 2σ(I). X-Ray diffraction analysis reveals that the asymmetric unit of the title compound contains two independent molecules which adopt an *E*-configuration about the C=N double bond. The dihedral angles between the naphthalene ring and the benzene ring are 71.7 (2)° and 57.4 (2)°, respectively, in the two independent molecules.

**Key Words:** (*E*)-*N*-(4-(Diethylamino)benzylidene)naphthalen-1-amine, Synthesis, Structure characterization.

### INTRODUCTION

The Schiff bases are used as substrates in the preparation of a number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions<sup>1</sup>. Schiff bases are known to have biological activities such as antimicrobial<sup>2-4</sup>, antitumor<sup>5</sup> and herbicidal properties<sup>6</sup>. They have also been widely used as versatile ligands involved in various metal chelation reactions to form metal complexes<sup>7-9</sup>, which are very interesting in many fields, such as catalysis and enzymatic reactions<sup>10-11</sup> and magnetism<sup>12</sup>. Recently, a few Schiff base compounds with antibacterial activity have been investigated<sup>14,13-15</sup>. However, to the best of our knowledge, the Schiff base (*E*)-*N*-(4-(diethylamino)benzylidene)naphthalen-1-amine is not reported so far. Investigation on the structure of the Schiff base compounds may be helpful to design and synthesise new metal complexes. In this paper, the title compound was synthesized and its molecular structure was investigated by elemental analysis, FT-IR, <sup>1</sup>H NMR and X-ray crystallographic techniques.

### EXPERIMENTAL

All the reagents were of AR grade and used without further purification. IR spectra (4000-400 cm<sup>-1</sup>), as KBr pellets, were recorded on a Nicolet FT-IR 510P spectrometer. <sup>1</sup>H NMR spectra were measured with a Bruker ALP 80 NMR spectrometer (CD<sub>3</sub>COCD<sub>3</sub> as solvent, TMS as internal standard).

**Synthesis of (*E*)-*N*-(4-(diethylamino)benzylidene)-naphthalen-1-amine:** A mixture of 4-(diethylamino)benzaldehyde (1.77 g, 0.01 mol) and α-naphthylamine (2.19 g, 0.01 mol) in ethyl alcohol (20 mL) was refluxed for 2 h. After cooling, filtration and drying, the desired compound was obtained. 10 mg of the present compound were dissolved in 15 mL ethyl alcohol and the solution was kept at room temperature. The single crystal suitable for X-ray determination was obtained by evaporation from ethyl alcohol solution after 1 week. Elemental analysis: calcd. (%) for C<sub>21</sub>H<sub>22</sub>N<sub>2</sub>: C 83.44, H 7.29, N 9.27. Found (%): C 83.25, H 7.32, N 9.31. Selected IR data (KBr, ν<sub>max</sub>, cm<sup>-1</sup>): 1636 (s, C=N), 1418 (s, C-N). <sup>1</sup>H NMR data (CD<sub>3</sub>COCD<sub>3</sub>, ppm): δ = 8.41 (s, 1H), 7.73 (d, 3H), 7.45 (d, 2H), 7.31 (d, 4H), 6.65 (d, 2H), 3.42 (m, 4H), 1.15 (m, 6H).

**Data collection and structure determination:** A selected crystal of the present Schiff base was mounted on a SMART CCD diffractometer. The reflection data were measured at 298 K, using a graphite monochromator MoK<sub>α</sub> (λ = 0.71073 Å) radiation with an ω-2θ scan mode. The total reflections were 16870 with 6145 independent ones (R<sub>int</sub> = 0.0539), of which 439 were observed with I > 2σ(I). Intensities were corrected for Lorentz and polarization effects and empirical absorption and all data were corrected using SADABB<sup>16</sup> program.

The structure was solved by direct methods using SHELXS-97<sup>17</sup> program. All the non-hydrogen atoms were refined on F<sup>2</sup> anisotropically by full-matrix least squares method. All hydrogen atoms were placed in the geometrically

calculated positions. The contributions of these hydrogen atoms were included in the structure factor calculations. The atomic scattering factors and anomalous dispersion corrections were taken from International Table for X-ray crystallography<sup>18</sup>. The final least-square cycle gave  $R = 0.0620$  and  $\omega R = 0.1374$  ( $w = 1/[\sigma^2(F_o^2) + 0.0000P^2 + 2.9151P]$ , where  $P = (F_o^2 + 2F_c^2)/3$ ).  $S = 1.054$ ,  $(\Delta/\sigma)_{\max} = 0.000$ ,  $(\Delta\rho)_{\min} = -0.260$  and  $(\Delta\rho)_{\max} = 0.445 \text{ e}/\text{\AA}^3$ . CIF file containing complete information on the studied structure was deposited with CCDC, deposition number

778615 and is freely available upon request from the following web site: [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

## RESULTS AND DISCUSSION

The selected bond distances and bond angles are given in Table-1. A displacement ellipsoid plot with atomic numbering scheme is shown in Fig. 1 and a perspective view of the crystal packing in the unit cell in Fig. 2.

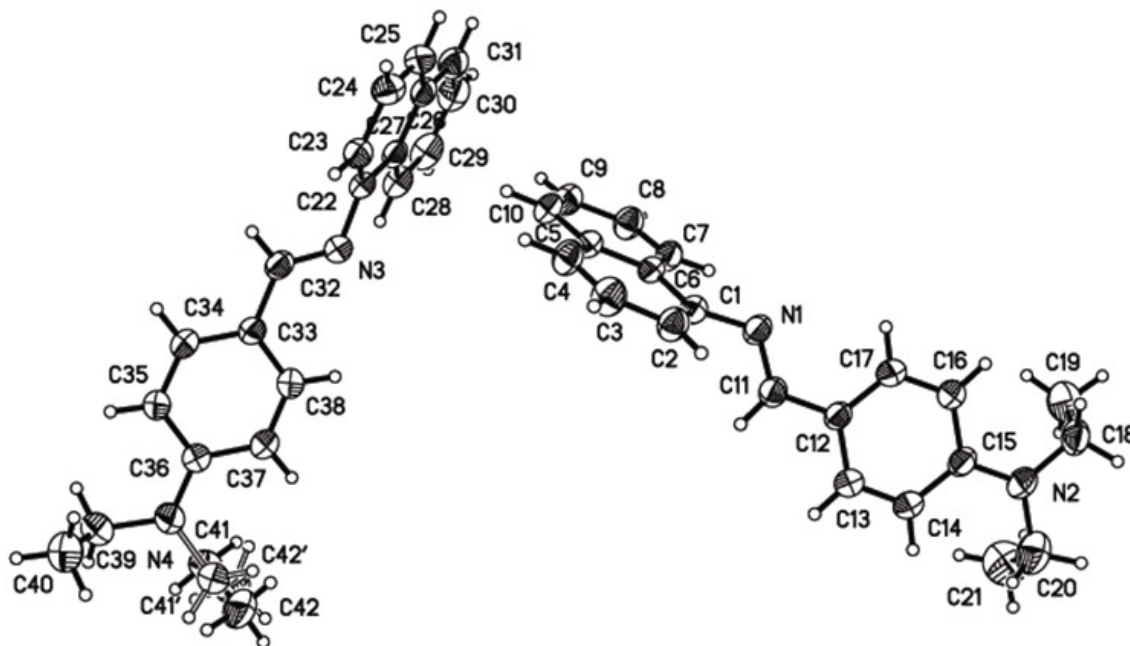


Fig. 1. Molecular structure with atomic numbering scheme

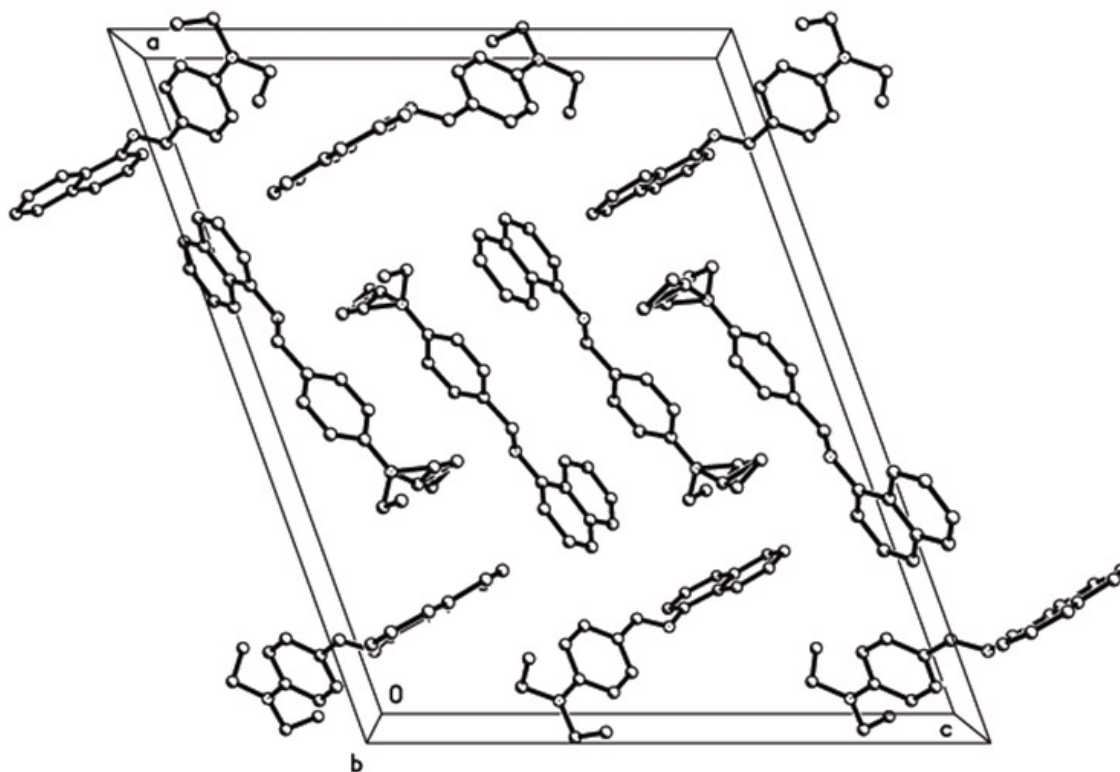


Fig. 2. View of crystal packing down the *b*-axis

TABLE-1  
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (°)

Bond	Length (Å)	Bond	Angle (°)
N(1)-C(11)	1.265(4)	C(11)-N(1)-C(1)	119.6(3)
N(1)-C(1)	1.412(5)	C(15)-N(2)-C(18)	121.4(4)
N(2)-C(15)	1.362(5)	C(15)-N(2)-C(20)	121.4(4)
N(2)-C(18)	1.460(5)	C(18)-N(2)-C(20)	117.0(4)
N(2)-C(20)	1.475(6)	C(32)-N(3)-C(22)	119.9(4)
N(3)-C(32)	1.277(5)	C(36)-N(4)-C(39)	121.9(4)
N(3)-C(22)	1.402(5)	C(36)-N(4)-C(41)	121.4(8)
N(4)-C(36)	1.362(5)	C(39)-N(4)-C(41)	111.5(8)
N(4)-C(39)	1.462(6)	C(36)-N(4)-C(41')	118.7(7)
N(4)-C(41)	1.51(4)	C(39)-N(4)-C(41')	117.8(7)
N(4)-C(41')	1.55(3)	C(41)-N(4)-C(41')	37.1(8)

As seen from Fig. 1, the asymmetric unit of the present compound consists of two independent molecules which adopt E configuration with respect to the C=N double bond. Each molecule contains one naphthalene ring and one benzene ring and the two rings are connected by C=N double bond. In the two molecules, the C1-N1=C11-C12 and C22-N3=C32-C33 torsion angles between naphthalene ring and benzene ring are  $-175.5(4)^\circ$  and  $-174.9(4)^\circ$ , respectively. The dihedral angles between the naphthalene ring and the benzene ring are  $71.7(2)^\circ$  and  $57.4(2)^\circ$ , respectively. However, in the similar compound, 4-chloro-N-[4-(diethylamino)benzylidene]aniline<sup>19</sup>, the dihedral angles between the two aromatic rings are  $64.0(1)^\circ$  and  $66.5(1)^\circ$ , respectively. This may be due to the presence of the naphthalene. The bond lengths and bond angles in the title compound are as expected. The C11-N1 and C32-N3 are double bonds and their bond lengths are 1.265(4) and 1.277(5) Å, respectively. In the compound<sup>19</sup>, however, the corresponding double bond lengths are 1.273(2) Å (C11-N2) and 1.269(2) Å (C28-N4), respectively. While the other N-C [1.362(5)-1.475(6) Å] in the title compound are found to have normal single bond length and in good agreement with the values reported in the

literature<sup>19</sup>. As seen from Figs. 1 and 2, one of the two ethyls in one of the two independent molecules is disorder.

#### ACKNOWLEDGEMENTS

This work was supported by Shandong Provincial Natural Science Foundation, China (No. ZR2009BL027).

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