Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: www.tandfonline.com/journals/gmcl20

Crystal Structure Analysis and Conformational Isomerism of 2,2'-(4,4'-oxybis(4,1-phenylene)bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-1-ol

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To cite this article: V. Krishna, Srinivas Basavoju & A. Ramachandraiah (2012) Crystal Structure Analysis and Conformational Isomerism of 2,2'-(4,4'-oxybis(4,1-phenylene)bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-1-ol, Molecular Crystals and Liquid Crystals, 562:1, 265-290, DOI: 10.1080/10426507.2012.669675

To link to this article: https://doi.org/10.1080/10426507.2012.669675

Mol. Cryst. Liq. Cryst., Vol. 562: pp. 265–290, 2012

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/10426507.2012.669675

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Crystal Structure Analysis and Conformational Isomerism of 2,2′-(4,4′-oxybis(4,1-phenylene) bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene) dinaphthalen-1-ol

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*A helicate Schiff base ligand 2,2′-(4,4′-oxybis(4,1-phenylene)bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-1-ol (NDADPE) was synthesized from 1-hydroxynaphthalene-2-carbaldehyde and 4-(4-aminophenoxy)benzenamine. The compound (NDADPE) was characterized by FT-IR, ^{1}H NMR, and mass spectroscopic and elemental analyses. The structure of NDADPE was solved by single crystal X-ray diffraction method. The crystal structure reveals that there are two conformers (**A** and **B**). Conformer **A** forms a supramolecular dimer with bifurcated C—H···O hydrogen bond synthon, whereas conformer **B** forms a supramolecular helix due to bifurcated C—H···O hydrogen bond synthons. Hirshfeld surface analyses were carried out on the two conformers to explain the conformational isomerism.*

Keywords 2D fingerprint plots; conformational isomers; helical structure; helicate schiff base; hirshfeld surface analysis; molecular modeling; single crystal X-ray diffraction; spectroscopic studies

Introduction

Schiff base ligands with aromatic spacer groups [1–5] as building blocks for metal-assisted self-assembly (metallosupramolecular chemistry) [6–16] have attracted considerable attention to demonstrate the requirements for the close and tunable control of the coordination sphere of the metal ion and the weak aromatic interactions between the spacer groups in the ligand. Many transition metal complexes with these Schiff base ligands are employed in homogeneous and heterogeneous catalysis [17–23]. Helical metallo-supramolecular arrays have attracted much attention because of the fundamental role of helicity in biology and the potential applications in the fields of asymmetric catalysis and nonlinear optical materials [24]. Huang and coworkers have reported two flexible Schiff base ligands, such as bis-[(N,N′-3,5-di-tert-butylsalicylidene)-4,4′-diaminodiphenyl]ether and bis-[(N,N′-3-tert-butyl-5-methylsalicylidene)-4,4′-diaminodiphenyl]ether, which formed double helical

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Table 1. Crystallographic and experimental data of NDADPE

Empirical formula	$C_{34}\ H_{24}\ N_2\ O_3$
Formula weight	508.55
Temperature	293 (2) K
Wavelength	1.54184 Å
Crystal system	Triclinic
Space group	*P*-1
Unit cell dimensions	
a/Å	$a = 5.2902\ (6)$
b/Å	$b = 20.195\ (2)$
c/Å	$c = 23.832\ (3)$
$\alpha/^\circ$	$\alpha = 87.860\ (9)$
$\beta/^\circ$.	$\beta = 86.386\ (9)$
$\gamma/^\circ$	$\gamma = 85.286\ (9)$
Volume	2531.1 (5) $Å^3$
Z	4
Density (calculated)	1.335 mg m^{-3}
Absorption coefficient	0.684 mm^{-1}
F(000)	1064
Crystal size	0.42 × 0.18 × 0.08 mm^3
Theta range for data collection	2.83° to 58.92°.
Index ranges	$-5<=h<=4, -21<=k<=22, -26<=l<=25$
Reflections collected	11,529
Independent reflections	7043 [R(int) = 0.0580]
Completeness to theta = 58.92°	96.9%
Absorption correction	multiscan
Max. and min. transmission	0.947 and 0.8623
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	7043/0/736
Goodness-of-fit on F^2	0.973
Final R indices [I > 2 sigma (I)]	R1 = 0.0649, wR2 = 0.1796
R indices (all data)	R1 = 0.1492, wR2 = 0.1280
Extinction coefficient	0.00048 (7)
Largest diff. peak and hole	0.162 and −0.199 e.$Å^{-3}$
CCDC	833469

dinuclear complexes with Cu(II) ion in which Cu(II) has a pseudo-tetrahedral coordination sphere with two-wrapped ligands. Recently, a progress toward the employment of more synthetically amenable Schiff's base ligand types has occurred and this has increased knowledge of the self-assembly process greatly [25–27]. Within these studies, it has been proposed that weak intramolecular (interstrand) face-to-face and edge-to-face π—π interactions between the aromatic rings of these ligands that gives rise to helicate formation in the solid-state structure. In this paper, we report the crystal structure analysis and conformational isomerism of Schiff base ligand 2,2′-(4,4′-oxybis(4,1-phenylene)bis

Table 2. Geometrical parameters of hydrogen bonds in NDADPE

Compound	D—H···A[a]	D—A (Å)	H···A (Å)	D—H···A (°)
NDADPE	Intra O(2)—H(2)···N(2)	2.519 (6)	1.61 (11)	153 (11)
	Intra O(3)—H(3A)···N(1)	2.510 (6)	1.69 (10)	136 (8)
	Intra O(5)—H(5)···N(4)	2.525 (6)	1.60 (8)	154 (9)
	Intra O(6)—H(6A)···N(3)	2.504 (6)	1.67 (8)	139 (6)
	C(9)—H(9)···O(3)	3.235 (7)	2.4	141
	C(43)—H(43)···O(2)	3.087 (6)	2.37	134

[a]All of the C—H, N—H, and O—H bonds are neutron normalized to 1.083, 1.009, and 0.983Å respectively.

(azan-1-yl-1ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-1-ol, (NDADPE), which exhibited features of helicate type crystal packing.

Experimental

Materials and Methods

All reagents and solvents were of analytical grade and used without further purification. 1-hydroxynaphthalene-2-carbaldehyde was prepared in the same manner as reported in the literature [28].

Physical Measurements

^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX FT-NMR spectrometer; using tetramethylsilane (TMS) as an internal reference at room temperature operating at 400 MHz. Infrared absorption spectra (4000–400 cm^{-1}) were obtained from a JASCO FT/IR-5300 spectrometer. Mass spectra (ESI-MS) were determined on Perkin Elmer (SCIEX API- 2000, ESI) at 12.5 Ev. Carbon, nitrogen, and hydrogen elemental analyses were performed on a LECO CHNS-932 analyzer. Melting points were determined on DSC Model: Q 10 TA Instruments. Thermal analyses of these samples were performed on a Thermal Advantage DSC Q2000 V9.8 Build 296 (TA Instrument, USA) module which was calibrated for temperature and cell constants using indium and sapphire. The instrument was equipped with refrigerator cooling system (RCS). The crystals (3–5 mg) were crimped in aluminium pans (nonhermetic) (30 μL) scanned at a heating rate of 10°C min^{-1} in the range 30°C–300°C under a dry nitrogen atmosphere (flow rate 50 mL min^{-1}). The data were collected by TA Instruments Universal Analysis 2000 V4.3A software. The single crystal X-ray data was collected on Bruker-Nonius SMART APEX CCD diffractometer at 293 (2) K using graphite-monochromated Cu Kα radiation ($\lambda = 1.54184$ Å).

Synthesis of NDADPE

The ligand was prepared by a usual Schiff-base condensation in methanol (50 mL) of 1-hydroxynaphthalene-2-carbaldehyde (1.72 g, 10 mmol) with 4-(4-aminophenoxy) benzenamine (2.02 g, 5 mmol). The reaction mixture was stirred and refluxed for 30 min. A

Table 3. Bond lengths(Å), bond angles(°) for NDADPE

Atoms	XRD data (q_1)	Molecular modeling data (q_2)	$\Delta q = q_1 - q_2$
O(2)−C(15)	1.301 (6)	1.361	0.06
C(14)−C(13)	1.425 (7)	1.431	0.006
C(36)−C(37)	1.364 (6)	1.376	0.0156
C(34)−C(33)	1.412 (6)	1.419	0.0065
C(44)−C(45)	1.367 (6)	1.373	0.0061
C(48)−C(49)	1.422 (7)	1.429	0.0067
C(34)−C(25)	1.448 (6)	1.439	−0.0093
C(23)−C(21)	1.404 (6)	1.411	0.0066
C(33)−C(28)	1.420 (7)	1.424	0.0038
C(10)−C(11)	1.371 (7)	1.373	0.0022
C(57)−C(55)	1.410 (7)	1.420	0.0095
C(41)−C(46)	1.389 (7)	1.389	0.0003
C(34)−C(25)	1.448 (6)	1.478	0.0297
N(3)−C(58)	1.284 (6)	1.284	0.0003
N(4)−C(44)	1.429 (6)	1.432	0.0032
C(34)−C(32)	1.401 (6)	1.402	0.0006
C(57)−C(56)	1.396 (7)	1.397	0.0012
C(34)−C(32)	1.401 (6)	1.402	0.0007
C(23)−C(21)	1.404 (6)	1.403	−0.0013
C(54)−C(53)	1.391 (7)	1.394	0.003
C(34)−C(32)	1.401 (6)	1.402	0.0007
O(1)−C(5)	1.382 (6)	1.369	−0.0135
O(6)−C(60)	1.315 (6)	1.368	0.0532
C(22)−C(18)	1.392 (7)	1.402	0.0103
C(10)−C(9)	1.390 (7)	1.397	0.0068
C(20)−C(19)	1.390 (7)	1.401	0.0114
C(54)−C(53)	1.391 (7)	1.401	0.0104
C(56)−C(52)	1.409 (7)	1.394	−0.0148
C(34)−C(32)	1.401 (6)	1.403	0.0023
C(58)−C(59)	1.428 (7)	1.432	0.0043
O(3)−C(26)	1.303 (6)	1.361	0.058
N(3)−C(58)	1.289 (6)	1.285	−0.0037
C(34)−C(25)	1.448 (6)	1.477	0.0285
C(24)−C(25)	1.428 (6)	1.425	−0.0032
C(43)−C(42)	1.364 (6)	1.372	0.0076
C(33)−C(28)	1.420 (7)	1.422	0.002
C(44)−C(43)	1.389 (6)	1.389	0
C(14)−C(23)	1.441 (6)	1.442	0.0008
C(6)−C(1)	1.378 (6)	1.374	−0.0044
C(67)−C(62)	1.426 (7)	1.428	0.0015
C(57)−C(56)	1.396 (7)	1.410	0.0136
C(58)−C(59)	1.428 (7)	1.431	0.0028
C(6)−C(1)	1.375 (6)	1.376	0.0011

Table 3. Bond lengths(Å), bond angles(°) for NDADPE *(Continued)*

Atoms	XRD data (q_1)	Molecular modeling data (q_2)	$\Delta q = q_1 - q_2$
C(34)−C(33)	1.412 (6)	1.418	0.0056
O(5)−H(5)	0.980 (2)	0.971	−0.0094
C(41)−O(4)−C(39)	116.6 (4)	116.7	0.1000
C(13)−N(2)−C(10)	125.9 (5)	125.9	0.0000
C(58)−N(3)−C(36)	124.4 (5)	124.5	0.1000
C(24)−N(1)−C(2)	124.1 (5)	124.3	0.2000
C(49)−O(5)−H(5)	103 (6)	103.0	0.0000
C(47)−N(4)−C(44)	124.8 (5)	124.9	0.1000
C(26)−O(3)−H(3A)	113 (6)	113.0	0.0000
C(60)−O(6)−H(6A)	113 (5)	113.0	0.0000
C(45)−C(44)−C(43)	119.6 (5)	119.7	0.1000
C(45)−C(44)−N(4)	116.8 (5)	116.9	0.1000
C(43)−C(44)−N(4)	123.5 (5)	123.6	0.1000
C(5)−O(1)−C(7)	116.2 (4)	116.2	0.0000
C(32)−C(34)−C(33)	117.6 (5)	117.7	0.1000
C(32)−C(34)−C(25)	123.3 (4)	123.3	0.0000
C(33)−C(34)−C(25)	119 (5)	119.1	0.1000
C(37)−C(36)−C(35)	118.3 (5)	118.4	0.1000
C(37)−C(36)−N(3)	117.1 (5)	117.1	0.0000
C(35)−C(36)−N(3)	124.6 (5)	124.7	0.1000
C(47)−C(48)−C(49)	118.9 (5)	118.9	0.0000
C(47)−C(48)−C(57)	121.8 (5)	121.8	0.0000
C(49)−C(48)−C(57)	119.4 (5)	119.3	−0.1000
C(42)−C(41)−O(4)	117.1 (5)	117.2	0.1000
C(42)−C(41)−C(46)	120.1 (5)	120.1	0.0000
O(4)−C(41)−C(46)	122.7 (5)	122.8	0.1000
C(56)−C(57)−C(55)	116.2 (5)	116.1	−0.1000
C(56)−C(57)−C(48)	120 (5)	120.0	0.0000
C(55)−C(57)−C(48)	123.8 (5)	123.9	0.1000
C(15)−C(14)−C(13)	119.1 (5)	119.1	0.0000
C(15)−C(14)−C(23)	119.2 (5)	119.2	0.0000
C(13)−C(14)−C(23)	121.7 (5)	121.7	0.0000
N(1)−C(24)−C(25)	121.6 (5)	121.7	0.1000
N(1)−C(24)−H(24)	117 (2)	117.0	0.0000
C(25)−C(24)−H(24)	122 (2)	122.0	0.0000
C(26)−C(25)−C(24)	118.7 (5)	118.8	0.1000
C(26)−C(25)−C(34)	119.6 (4)	119.7	0.1000
C(24)−C(25)−C(34)	121.7 (5)	121.8	0.1000
C(42)−C(43)−C(44)	120.2 (5)	120.3	0.1000
C(42)−C(43)−H(43)	119.9 (5)	120.0	0.1000
C(8)−C(7)−C(12)	120.5 (5)	120.5	0.0000

(Continued on next page)

Table 3. Bond lengths(Å), bond angles(°) for NDADPE *(Continued)*

Atoms	XRD data (q_1)	Molecular modeling data (q_2)	$\Delta q = q_1 - q_2$
C(8)−C(7)−O(1)	118.2 (5)	118.3	0.1000
C(12)−C(7)−O(1)	121.2 (5)	121.2	0.0000
N(2)−C(13)−C(14)	122.4 (5)	122.5	0.1000
N(2)−C(13)−H(13)	117 (3)	117.1	0.1000
C(14)−C(13)−H(13)	120 (3)	120.0	0.0000
C(57)−C(56)−C(52)	121.0 (5)	121.1	0.1000
C(57)−C(56)−C(51)	119.1 (5)	119.1	0.0000
C(52)−C(56)−C(51)	119.9 (5)	119.9	0.0000
N(3)−C(58)−C(59)	123.0 (5)	123.0	0.0000
N(3)−C(58)−H(58)	119 (3)	119.0	0.0000
C(59)−C(58)−H(58)	117 (3)	117.0	0.0000
O(3)−C(26)−C(25)	122.6 (5)	122.7	0.1000
O(3)−C(26)−C(27)	118.0 (5)	118.0	0.0000
C(25)−C(26)−C(27)	119.4 (5)	119.5	0.1000
C(3)−C(2)−C(1)	118.7 (5)	118.8	0.1000
C(3)−C(2)−N(1)	116.1 (5)	116.1	0.0000
C(1)−C(2)−N(1)	125.2 (5)	125.3	0.1000
N(4)−C(47)−C(48)	121.8 (5)	121.8	0.0000
N(4)−C(47)−H(47)	119 (3)	119.0	0.0000
C(48)−C(47)−H(47)	120 (3)	120.0	0.0000
C(29)−C(33)−C(34)	119.7 (5)	119.8	0.1000
C(29)−C(33)−C(28)	121.6 (5)	121.6	0.0000
C(34)−C(33)−C(28)	118.7 (5)	118.7	0.0000
C(21)−C(23)−C(22)	116.8 (5)	116.8	0.0000
C(21)−C(23)−C(14)	123.7 (5)	123.8	0.1000
C(22)−C(23)−C(14)	119.5 (5)	119.5	0.0000
C(11)−C(10)−C(9)	117.5 (5)	117.6	0.1000
C(11)−C(10)−N(2)	117.2 (5)	117.3	0.1000
C(9)−C(10)−N(2)	125.2 (5)	125.2	0.0000
C(66)−C(68)−C(67)	116.5 (5)	116.5	0.0000
C(66)−C(68)−C(59)	124.0 (5)	124.0	0.0000
C(67)−C(68)−C(59)	119.4 (5)	119.5	0.1000
C(31)−C(32)−C(34)	121.4 (5)	121.4	0.0000
C(31)−C(32)−H(32)	119.3	119.3	0.0000
C(34)−C(32)−H(32)	119.3	119.3	0.0000
C(15)−O(2)−H(2)	105 (8)	105.0	0.0000
C(39)−C(40)−C(35)	120.9 (5)	121.0	0.1000
C(39)−C(40)−H(40)	119.5	119.6	0.1000
C(36)−C(37)−C(38)	121.5 (5)	121.6	0.1000
C(36)−C(37)−H(37)	119.3	119.4	0.1000
C(6)−C(5)−C(4)	119.5 (5)	119.6	0.1000
C(6)−C(5)−O(1)	119.3 (5)	119.4	0.1000

Table 3. Bond lengths(Å), bond angles(°) for NDADPE *(Continued)*

Atoms	XRD data (q_1)	Molecular modeling data (q_2)	$\Delta q = q_1 - q_2$
C(4)−C(5)−O(1)	121.1 (5)	121.1	0.0000
C(18)−C(22)−C(23)	120.5 (5)	120.5	0.0000
C(44)−C(45)−C(46)	120.8 (5)	120.9	0.1000
C(44)−C(45)−H(45)	119.6	119.7	0.1000
C(8)−C(9)−C(10)	121.0 (5)	121	0.0000
C(8)−C(9)−H(9)	119.5	119.6	0.1000
C(7)−C(8)−C(9)	119.7 (5)	119.6	−0.1000
C(7)−C(8)−H(8)	120.2	120.3	0.1000
C(2)−C(1)−C(6)	120.6 (5)	120.6	0.0000
C(2)−C(1)−H(1)	119.7	119.8	0.1000
C(54)−C(55)−C(57)	121.8 (5)	121.9	0.1000
C(54)−C(55)−H(55)	119.1	119.1	0.0000
C(28)−C(27)−C(26)	120.4 (5)	120.4	0.0000
C(28)−C(27)−H(27)	119.8	119.9	0.1000
C(58)−C(59)−C(68)	122.7 (5)	122.7	0.0000
C(58)−C(59)−C(60)	117.1 (5)	117.1	0.0000
C(68)−C(59)−C(60)	120.1 (5)	120.2	0.1000
O(5)−C(49)−C(48)	122.7 (5)	122.8	0.1000
O(5)−C(49)−C(50)	118.8 (6)	118.8	0.0000
C(48)−C(49)−C(50)	118.4 (5)	118.5	0.1000
C(40)−C(39)−C(38)	119.6 (5)	119.6	0.0000
C(40)−C(39)−O(4)	118.4 (5)	118.5	0.1000
C(38)−C(39)−O(4)	122.0 (5)	122	0.0000
C(50)−C(51)−H(51)	118.9	118.9	0.0000
C(32)−C(31)−C(30)	121.1 (5)	121.2	0.1000
C(32)−C(31)−H(31)	119.4	119.5	0.1000
C(10)−C(11)−C(12)	121.1 (5)	121.4	0.3000
C(10)−C(11)−H(11)	119.5	119.6	0.1000
C(40)−C(35)−C(36)	119.9 (5)	120	0.1000
C(36)−C(35)−H(35)	120.0	120.1	0.1000
C(30)−C(29)−C(33)	121.2 (5)	121.3	0.1000
C(30)−C(29)−H(29)	119.4	119.5	0.1000
C(62)−C(61)−C(60)	122.0 (6)	122	0.0000
C(62)−C(61)−H(61)	119.0	119	0.0000
C(27)−C(28)−C(33)	122.8 (5)	122.9	0.1000
C(27)−C(28)−H(28)	118.6	118.7	0.1000
C(29)−C(30)−C(31)	119.0 (5)	190.1	71.1000
C(29)−C(30)−H(30)	120.5	120.5	0.0000
O(2)−C(15)−C(16)	119.4 (6)	119.5	0.1000
O(2)−C(15)−C(14)	121.4 (5)	121	−0.4000
C(16)−C(15)−C(14)	119.2 (5)	119.2	0.0000
C(19)−C(18)−C(22)	121.3 (5)	121.4	0.1000
C(19)−C(18)−H(18)	119.4	119.5	0.1000

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Table 3. Bond lengths(Å), bond angles(°) for NDADPE *(Continued)*

Atoms	XRD data (q_1)	Molecular modeling data (q_2)	$\Delta q = q_1 - q_2$
C(21)−C(20)−C(19)	121.5 (5)	121.7	0.2000
C(21)−C(20)−H(20)	119.3	119.2	−0.1000
C(55)−C(54)−C(53)	121.2 (5)	121.2	0.0000
C(55)−C(54)−H(54)	119.4	119.3	−0.1000
C(2)−C(3)−C(4)	121.0 (6)	121	0.0000
C(2)−C(3)−H(3)	119.5	119.6	0.1000
C(17)−C(16)−C(15)	121.6 (5)	121.6	0.0000
C(17)−C(16)−H(16)	119.2	119.5	0.3000
C(63)−C(67)−C(68)	120.1 (6)	120.3	0.2000
C(63)−C(67)−C(62)	121.5 (6)	121.3	−0.2000
C(68)−C(67)−C(62)	118.4 (5)	118.5	0.1000
C(53)−C(52)−C(56)	121.2 (5)	121.3	0.1000
C(53)−C(52)−H(52)	119.4	119.4	0.0000
C(18)−C(19)−C(20)	118.4 (5)	118.5	0.1000
C(18)−C(19)−H(19)	120.8	120.7	−0.1000
C(16)−C(17)−C(22)	121.6 (5)	121.7	0.1000
C(16)−C(17)−H(17)	119.2	119.3	0.1000
C(64)−C(63)−C(67)	121.6 (6)	121.7	0.1000
C(3)−C(4)−C(5)	119.8 (5)	119.9	0.1000
C(66)−C(65)−C(64)	120.8 (6)	120.9	0.1000
C(66)−C(65)−H(65)	119.6	119.5	−0.1000
C(65)−C(66)−C(68)	121.7 (6)	121.8	0.1000
C(65)−C(66)−H(66)	119.1	119.2	0.1000
C(7)−C(12)−C(11)	120.0 (5)	120	0.0000
C(52)−C(53)−C(54)	118.5 (6)	118.6	0.1000
C(52)−C(53)−H(53)	120.7	120.7	0.0000
C(61)−C(62)−C(67)	122.0 (6)	122	0.0000
C(61)−C(62)−H(62)	119.0	119	0.0000
C(63)−C(64)−C(65)	119.2 (6)	119.3	0.1000
C(63)−C(64)−H(64)	120.4	120.5	0.1000
C(65)−C(64)−H(64)	120.4	120.5	0.1000
C(37)−C(38)−C(39)	119.6 (5)	119.7	0.1000
C(37)−C(38)−H(38)	120.2	120.1	−0.1000
C(23)−C(22)−C(17)	118.9 (5)	119.1	0.2000
C(45)−C(46)−H(46)	120.5	120.6	0.1000
C(20)−C(21)−C(23)	121.5 (5)	121.6	0.1000
C(20)−C(21)−H(21)	119.3	119.3	0.0000
O(6)−C(60)−C(59)	122.7 (5)	122.9	0.2000
C(61)−C(60)−C(59)	118.0 (5)	118.0	0.0000
C(43)−C(42)−C(41)	120.2 (5)	120.5	0.3000
C(5)−C(6)−C(1)	120.3 (5)	120.5	0.2000
C(5)−C(6)−H(6)	119.8	120.0	0.2000
C(51)−C(50)−C(49)	120.9 (6)	121.0	0.1000
C(51)−C(50)−H(50)	119.5	120.0	0.5000

Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($A^2 \times 10^3$) for NDADPE. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

Atoms	*x*	*y*	*z*	U(eq)
O(4)	9331 (6)	8270 (2)	4498 (1)	84 (1)
N(2)	3719 (8)	7134 (2)	6597 (2)	72 (1)
N(3)	5552 (8)	7654 (2)	2478 (2)	68 (1)
N(1)	6182 (8)	5285 (2)	2981 (2)	68 (1)
O(5)	−1620 (8)	10, 218 (2)	6222 (2)	87 (1)
N(4)	2440 (8)	9478 (2)	6080 (2)	68 (1)
O(3)	10, 135 (7)	4553 (2)	2737 (2)	87 (1)
O(6)	2191 (8)	7057 (2)	2060 (2)	85 (1)
C(44)	4274 (10)	9169 (2)	5685 (2)	62 (1)
O(1)	−529 (6)	6147 (2)	4746 (1)	87 (1)
C(34)	8300 (9)	5546 (2)	1480 (2)	60 (1)
C(36)	6684 (10)	7788 (2)	2973 (2)	63 (1)
C(48)	557 (9)	9711 (2)	6995 (2)	59 (1)
C(41)	7576 (10)	8570 (3)	4880 (2)	67 (1)
C(57)	611 (9)	9629 (2)	7600 (2)	61 (1)
C(14)	4666 (9)	7231 (2)	7558 (2)	60 (1)
C(24)	6290 (9)	5528 (2)	2464 (2)	64 (1)
C(25)	8242 (9)	5299 (2)	2059 (2)	57 (1)
C(43)	6464 (10)	8799 (2)	5840 (2)	67 (1)
C(7)	534 (10)	6393 (3)	5209 (2)	70 (2)
C(13)	3312 (10)	6971 (2)	7128 (2)	67 (1)
C(56)	−1275 (10)	9957 (2)	7947 (2)	67 (1)
C(58)	5844 (11)	7987 (3)	2009 (2)	67 (1)
C(26)	10, 161 (10)	4823 (3)	2233 (2)	68 (1)
C(2)	4378 (10)	5517 (2)	3407 (2)	66 (1)
C(47)	2461 (10)	9393 (3)	6627 (2)	66 (1)
C(33)	10, 343 (9)	5322 (2)	1106 (2)	65 (1)
C(23)	4200 (9)	7044 (2)	8142 (2)	62 (1)
C(10)	2524 (10)	6881 (3)	6144 (2)	67 (1)
C(68)	4747 (10)	8267 (2)	1015 (2)	68 (1)
C(32)	6415 (10)	5997 (2)	1267 (2)	68 (1)
O(2)	7049 (9)	7857 (2)	6882 (2)	103 (1)
C(40)	9610 (10)	8308 (3)	3502 (2)	72 (2)
C(37)	5622 (11)	7541 (3)	3465 (2)	80 (2)
C(5)	1156 (10)	5947 (3)	4306 (2)	74 (2)
C(22)	5614 (10)	7310 (2)	8553 (2)	66 (1)
C(46)	5432 (10)	8956 (3)	4719 (2)	75 (2)
C(21)	2375 (9)	6609 (2)	8338 (2)	69 (1)
C(60)	2562 (10)	7416 (3)	1595 (2)	71 (2)
C(42)	8077 (10)	8496 (2)	5441 (2)	69 (1)
C(6)	614 (10)	6139 (3)	3765 (2)	76 (2)
C(50)	−3383 (10)	10, 439 (3)	7137 (3)	81 (2)

(Continued on next page)

Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($A^2 \times 10^3$) for NDADPE. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor *(Continued)*

Atoms	x	y	z	U(eq)
C(45)	3785 (10)	9247 (3)	5129 (2)	71 (1)
C(9)	528 (10)	6472 (3)	6198 (2)	76 (2)
C(8)	−425 (10)	6220 (3)	5730 (2)	77 (2)
C(1)	2220 (10)	5927 (3)	3318 (2)	74 (2)
C(55)	2493 (10)	9226 (3)	7874 (2)	72 (2)
C(27)	12, 214 (10)	4621 (3)	1843 (3)	77 (2)
C(59)	4448 (10)	7886 (2)	1530 (2)	64 (1)
C(49)	−1465 (10)	10, 115 (2)	6760 (3)	70 (2)
C(39)	8422 (10)	8096 (3)	3991 (2)	71 (1)
C(51)	−3266 (11)	10, 361 (3)	7697 (3)	82 (2)
C(31)	6520 (10)	6206 (3)	715 (2)	75 (2)
C(11)	3418 (11)	7053 (3)	5610 (2)	93 (2)
C(35)	8795 (10)	8157 (3)	2995 (2)	75 (2)
C(29)	10, 414 (11)	5552 (3)	541 (2)	78 (2)
C(61)	1112 (10)	7322 (3)	1131 (3)	77 (2)
C(28)	12, 271 (10)	4862 (3)	1312 (2)	75 (2)
C(30)	8533 (11)	5988 (3)	345 (2)	80 (2)
C(15)	6547 (11)	7678 (3)	7405 (3)	76 (2)
C(18)	5224 (10)	7129 (3)	9119 (2)	81 (2)
C(20)	2025 (10)	6442 (3)	8896 (2)	79 (2)
C(54)	2455 (10)	9156 (3)	8446 (2)	78 (2)
C(3)	4857 (11)	5305 (3)	3948 (2)	89 (2)
C(16)	7930 (10)	7926 (3)	7830 (3)	79 (2)
C(67)	3244 (11)	8152 (3)	564 (2)	73 (2)
C(52)	−1253 (11)	9887 (3)	8537 (2)	85 (2)
C(19)	3427 (11)	6702 (3)	9297 (2)	86 (2)
C(17)	7474 (10)	7757 (3)	8376 (3)	79 (2)
C(63)	3529 (12)	8518 (3)	55 (3)	95 (2)
C(4)	3269 (11)	5519 (3)	4398 (2)	94 (2)
C(65)	6740 (12)	9100 (3)	415 (3)	90 (2)
C(66)	6514 (10)	8752 (3)	918 (2)	78 (2)
C(12)	2445 (12)	6804 (3)	5144 (2)	104 (2)
C(53)	579 (11)	9493 (3)	8786 (3)	89 (2)
C(62)	1411 (11)	7673 (3)	646 (3)	84 (2)
C(64)	5247 (13)	8982 (3)	−21 (3)	99 (2)
C(38)	6445 (11)	7692 (3)	3972 (2)	90 (2)
C(53)	579 (11)	9493 (3)	8786 (3)	89 (2)
C(62)	1411 (11)	7673 (3)	646 (3)	84 (2)
C(64)	5247 (13)	8982 (3)	−21 (3)	99 (2)
C(38)	6445 (11)	7692 (3)	3972 (2)	90 (2)

Table 5. Anisotropic displacement parameters ($A^2 \times 10^3$) for NDADPE anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2\ a^{*2}$ U11 + ... + 2 $h \times k \times a^* \times b^* \times$ U12]

Atoms	U11	U22	U33	U23	U13	U12
O(4)	79 (3)	121 (3)	54 (2)	−15 (2)	−6 (2)	−8 (2)
N(2)	85 (3)	84 (3)	49 (3)	5 (2)	−18 (2)	−9 (2)
N(3)	85 (3)	66 (3)	52 (3)	−12 (2)	−9 (2)	1 (2)
N(1)	78 (3)	74 (3)	53 (3)	−2 (2)	1 (2)	−10 (2)
O(5)	103 (3)	81 (3)	78 (3)	0 (2)	−31 (2)	6 (2)
N(4)	81 (3)	67 (3)	56 (3)	−3 (2)	−14 (2)	−2 (2)
O(3)	102 (3)	78 (3)	77 (3)	10 (2)	−7 (2)	7 (2)
O(6)	110 (3)	78 (3)	69 (3)	−8 (2)	3 (2)	−18 (2)
C(44)	73 (4)	62 (3)	52 (3)	−1 (2)	−14 (3)	−12 (3)
O(1)	72 (2)	138 (3)	54 (2)	−19 (2)	−2 (2)	−15 (2)
C(34)	69 (3)	59 (3)	54 (3)	−9 (2)	−6 (3)	−9 (3)
C(36)	75 (4)	61 (3)	52 (3)	−8 (2)	−8 (3)	9 (3)
C(48)	69 (3)	52 (3)	58 (3)	−5 (2)	−8 (3)	−8 (2)
C(41)	67 (4)	83 (4)	54 (3)	−3 (3)	−6 (3)	−12 (3)
C(57)	67 (3)	54 (3)	63 (3)	−1 (3)	−2 (3)	−12 (3)
C(14)	71 (4)	53 (3)	57 (3)	−5 (2)	−14 (3)	−4 (2)
C(24)	64 (4)	60 (3)	68 (4)	−10 (3)	−7 (3)	−3 (3)
C(25)	59 (3)	54 (3)	58 (3)	−7 (2)	−9 (3)	−7 (2)
C(43)	85 (4)	74 (3)	42 (3)	1 (3)	−15 (3)	−5 (3)
C(7)	65 (4)	104 (4)	44 (3)	−6 (3)	−8 (3)	−11 (3)
C(13)	85 (4)	58 (3)	58 (4)	0 (3)	−14 (3)	−1 (3)
C(56)	73 (4)	56 (3)	72 (4)	2 (3)	−3 (3)	−4 (3)
C(58)	78 (4)	65 (3)	58 (4)	−13 (3)	−9 (3)	4 (3)
C(26)	74 (4)	65 (3)	65 (4)	−5 (3)	−6 (3)	−8 (3)
C(2)	71 (4)	70 (3)	56 (3)	−5 (3)	−3 (3)	−12 (3)
C(47)	71 (4)	63 (3)	67 (4)	−4 (3)	−17 (3)	−3 (3)
C(33)	59 (3)	69 (3)	67 (4)	−13 (3)	1 (3)	−10 (3)
C(23)	75 (4)	52 (3)	56 (3)	−9 (2)	0 (3)	1 (3)
C(10)	75 (4)	79 (4)	49 (3)	1 (3)	−15 (3)	0 (3)
C(68)	78 (4)	65 (3)	61 (4)	−15 (3)	−5 (3)	6 (3)
C(32)	76 (4)	67 (3)	59 (4)	−7 (3)	3 (3)	−4 (3)
O(2)	120 (3)	114 (3)	79 (3)	27 (3)	−22 (3)	−39 (3)
C(40)	78 (4)	80 (4)	59 (4)	−8 (3)	−3 (3)	−10 (3)
C(37)	96 (4)	85 (4)	61 (4)	1 (3)	−9 (3)	−22 (3)
C(5)	73 (4)	103 (4)	49 (3)	−18 (3)	−4 (3)	−15 (3)
C(22)	81 (4)	60 (3)	59 (4)	−9 (3)	−12 (3)	1 (3)
C(46)	81 (4)	98 (4)	46 (3)	5 (3)	−11 (3)	−14 (3)
C(21)	84 (4)	68 (3)	56 (3)	−11 (3)	−12 (3)	3 (3)
C(60)	78 (4)	68 (4)	67 (4)	−17 (3)	−7 (3)	4 (3)
C(42)	74 (4)	74 (3)	60 (4)	0 (3)	−17 (3)	−6 (3)
C(6)	70 (4)	95 (4)	63 (4)	−14 (3)	−10 (3)	−2 (3)
C(50)	62 (4)	66 (3)	112 (5)	4 (3)	−8 (4)	5 (3)

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Table 5. Anisotropic displacement parameters ($A^2 \times 10^3$) for NDADPE anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U11 + \ldots + 2 h \times k \times a^* \times b^* \times U12]$ *(Continued)*

Atoms	U11	U22	U33	U23	U13	U12
C(45)	78 (4)	82 (4)	55 (4)	2 (3)	−22 (3)	2 (3)
C(9)	88 (4)	88 (4)	51 (3)	1 (3)	1 (3)	−13 (3)
C(8)	88 (4)	94 (4)	51 (3)	−6 (3)	−6 (3)	−18 (3)
C(1)	86 (4)	85 (4)	52 (3)	−4 (3)	−9 (3)	−8 (3)
C(55)	73 (4)	82 (4)	60 (4)	−6 (3)	−2 (3)	4 (3)
C(27)	77 (4)	64 (3)	89 (5)	−6 (3)	−10 (3)	2 (3)
C(59)	76 (4)	60 (3)	56 (3)	−15 (3)	0 (3)	3 (3)
C(49)	76 (4)	58 (3)	79 (4)	−3 (3)	−18 (3)	−4 (3)
C(39)	78 (4)	81 (4)	54 (4)	−7 (3)	−5 (3)	4 (3)
C(51)	87 (4)	73 (4)	81 (4)	−1 (3)	6 (4)	7 (3)
C(31)	84 (4)	82 (4)	60 (4)	−3 (3)	−4 (3)	−6 (3)
C(11)	108 (5)	117 (5)	59 (4)	5 (3)	−2 (3)	−45 (4)
C(35)	75 (4)	90 (4)	58 (4)	−2 (3)	1 (3)	−7 (3)
C(29)	80 (4)	85 (4)	69 (4)	−27 (3)	14 (3)	−12 (3)
C(61)	82 (4)	69 (4)	83 (4)	−9 (3)	−17 (3)	−8 (3)
C(28)	66 (4)	86 (4)	74 (4)	−21 (3)	4 (3)	−8 (3)
C(30)	98 (4)	92 (4)	51 (3)	−9 (3)	0 (3)	−11 (3)
C(15)	86 (4)	72 (4)	70 (4)	4 (3)	−17 (3)	−4 (3)
C(18)	87 (4)	98 (4)	59 (4)	−19 (3)	−22 (3)	1 (3)
C(20)	96 (4)	86 (4)	54 (4)	−2 (3)	−3 (3)	−12 (3)
C(54)	87 (4)	81 (4)	65 (4)	1 (3)	−16 (3)	2 (3)
C(3)	96 (4)	107 (5)	59 (4)	7 (3)	−5 (3)	14 (3)
C(16)	77 (4)	67 (4)	94 (5)	5 (3)	−19 (4)	−15 (3)
C(67)	82 (4)	71 (4)	64 (4)	−12 (3)	−12 (3)	7 (3)
C(52)	96 (5)	84 (4)	71 (4)	−11 (3)	15 (4)	2 (3)
C(19)	104 (5)	113 (5)	43 (3)	−7 (3)	−11 (3)	−16 (4)
C(17)	84 (4)	71 (4)	84 (4)	−11 (3)	−28 (3)	−1 (3)
C(63)	115 (5)	101 (5)	67 (4)	−8 (4)	−23 (4)	8 (4)
C(4)	98 (5)	128 (5)	51 (4)	8 (3)	−2 (3)	3 (4)
C(65)	106 (5)	87 (4)	75 (4)	3 (4)	2 (4)	−5 (3)
C(66)	86 (4)	73 (4)	75 (4)	−13 (3)	−6 (3)	−7 (3)
C(12)	109 (5)	163 (6)	46 (4)	−1 (4)	0 (3)	−58 (5)
C(53)	104 (5)	96 (4)	66 (4)	−7 (3)	−4 (4)	0 (4)
C(62)	96 (5)	79 (4)	79 (4)	−18 (3)	−20 (4)	5 (3)
C(64)	126 (6)	94 (5)	74 (5)	3 (4)	−4 (4)	0 (4)
C(38)	99 (5)	115 (5)	58 (4)	11 (3)	−2 (3)	−40 (4)

yellow precipitate was filtered, washed by a small amount of methanol and dried in vacuo. (1.98 g, yield: 96%).

M.p. 194.38°C, Anal. Calcd. for $C_{34}H_{24}N_2O_3$: C: 80.3; H: 4.8; N: 5.5%. Found: C: 80.1; H: 4.6; N: 5.7%. IR (KBr, cm^{-1}): 3045.87 (Ar−H), 1620.35 (C=N), 1541.26 (C=C), 1323.29 (C−O). ^{1}H NMR (400 MHz, $CDCl_3$, internal reference TMS): δ ppm: 15.54 (2H,

Table 6. Torsion/Dihedral Angles (Deg.) in conformer **A**

C(7)−O(1)−C(5)−C(4)	50.5 (7)
C(7)−O(1)−C(5)−C(6)	−134.3 (5)
C(5)−O(1)−C(7)−C(8)	−139.8 (5)
C(5)−O(1)−C(7)−C(12)	41.4 (7)
C(24)−N(1)−C(2)−C(1)	13.7 (9)
C(24)−N(1)−C(2)−C(3)	−166.8 (5)
C(2)−N(1)−C(24)−C(25)	176.5 (5)
C(13)−N(2)−C(10)−C(9)	7.4 (9)
C(13)−N(2)−C(10)−C(11)	−172.5 (5)
C(10)−N(2)−C(13)−C(14)	177.6 (5)
C(6)−C(1)−C(2)−N(1)	−178.3 (5)
C(6)−C(1)−C(2)−C(3)	2.2 (9)
C(2)−C(1)−C(6)−C(5)	0.4 (9)
N(1)−C(2)−C(3)−C(4)	177.8 (5)
C(1)−C(2)−C(3)−C(4)	−2.7 (9)
C(2)−C(3)−C(4)−C(5)	0.6 (9)
C(3)−C(4)−C(5)−O(1)	177.2 (5)
C(3)−C(4)−C(5)−C(6)	2.0 (9)
O(1)−C(5)−C(6)−C(1)	−177.8 (5)
C(4)−C(5)−C(6)−C(1)	−2.5 (9)
O(1)−C(7)−C(8)−C(9)	−177.8 (5)
C(12)−C(7)−C(8)−C(9)	1.0 (9)
O(1)−C(7)−C(12)−C(11)	178.4 (5)
C(8)−C(7)−C(12)−C(11)	−0.4 (9)
C(7)−C(8)−C(9)−C(10)	−2.6 (9)
C(8)−C(9)−C(10)−N(2)	−176.4 (5)
C(8)−C(9)−C(10)−C(11)	3.5 (9)
N(2)−C(10)− C(11)−C(12)	176.9 (5)
C(9)−C(10)−C(11)−C(12)	−3.0 (9)
C(10)−C(11)−C(12)−C(7)	1.5 (9)
N(2)−C(13)−C(14)−C(15)	−0.7 (8)
N(2)−C(13)−C(14)−C(23)	−179.8 (5)
C(13)−C(14)−C(15)−O(2)	0.3 (8)
C(13)−C(14)−C(15)−C(16)	−178.7 (5)
C(23)−C(14)−C(15)−O(2)	179.4 (5)
C(23)−C(14)−C(15)−C(16)	0.4 (8)
C(13)−C(14)−C(23)−C(21)	−1.3 (7)
C(13)−C(14)−C(23)−C(22)	179.3 (5)
C(15)−C(14)−C(23)−C(21)	179.6 (5)
C(15)−C(14)−C(23)−C(22)	0.2 (7)
O(2)−C(15)−C(16)−C(17)	179.8 (5)
C(14)−C(15)−C(16)−C(17)	−1.2 (9)
C(15)−C(16)−C(17)−C(22)	1.3 (9)
C(16)−C(17)−C(22)−C(18)	178.3 (5)
C(16)−C(17)−C(22)−C(23)	−0.6 (8)
C(22)−C(18)−C(19)−C(20)	1.2 (9)

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Table 6. Torsion/Dihedral Angles (Deg.) in conformer **A** *(Continued)*

C(19)−C(18)−C(22)−C(17)	179.6 (5)
C(19)−C(18)−C(22)−C(23)	−1.5 (8)
C(18)−C(19)−C(20)−C(21)	−0.9 (9)
C(19)−C(20)−C(21)−C(23)	1.0 (8)
C(20)−C(21)−C(23)−C(14)	179.3 (5)
C(20)−C(21)−C(23)−C(22)	−1.3 (7)
C(17)−C(22)−C(23)−C(14)	−0.1 (7)
C(17)−C(22)−C(23)−C(21)	−179.6 (4)
C(18)−C(22)−C(23)−C(14)	−179.0 (5)
C(18)−C(22)−C(23)−C(21)	1.5 (7)
N(1)−C(24)−C(25)−C(26)	−3.2 (8)
N(1)−C(24)−C(25)−C(34)	177.1 (5)
C(24)−C(25)−C(26)−O(3)	4.5 (8)
C(24)−C(25)−C(26)−C(27)	−176.0 (5)
C(34)−C(25)−C(26)−O(3)	−175.8 (5)
C(34)−C(25)−C(26)−C(27)	3.8 (7)
C(24)−C(25)−C(34)−C(32)	−3.2 (7)
C(24)−C(25)−C(34)−C(33)	177.2 (5)
C(26)−C(25)−C(34)−C(32)	177.1 (5)
C(26)−C(25)−C(34)−C(33)	−2.5 (7)
O(3)−C(26)−C(27)−C(28)	177.1 (5)
C(25)−C(26)−C(27)−C(28)	−2.5 (8)
C(26)−C(27)−C(28)−C(33)	0.0 (9)
C(27)−C(28)−C(33)−C(29)	−178.1 (6)
C(27)−C(28)−C(33)−C(34)	1.3 (9)
C(33)−C(29)−C(30)−C(31)	0.0 (9)
C(30)−C(29)−C(33)−C(28)	179.1 (6)
C(30)−C(29)−C(33)−C(34)	−0.2 (9)
C(29)−C(30)−C(31)−C(32)	0.7 (9)
C(30)−C(31)−C(32)−C(34)	−1.2 (8)
C(31)−C(32)−C(34)−C(25)	−178.7 (5)
C(31)−C(32)−C(34)−C(33)	1.0 (7)
C(28)−C(33)−C(34)−C(25)	0.0 (7)
C(28)−C(33)−C(34)−C(32)	−179.6 (5)
C(29)−C(33)−C(34)−C(25)	179.4 (5)
C(29)−C(33)−C(34)−C(32)	−0.3 (7)

s, OH), 9.38 (2H, s, C=N), 7.16 (4H, d, aminophenyl), 7.38 (4H, d, aminophenyl), 7.40 (4H, s, naphthyl), 7.54 (4H, s, naphthyl) δ 7.73–8.14 (4H, m, naphthyl). Mass (ESI): *m/z* 509 $[M+H]^+$.

Crystallization

The 20 mg of the compound NDADPE was dissolved in chloroform and gently heated to aid the complete dissolution. The solution was allowed to slow evaporation at room temperature. Prismatic shaped crystals were obtained after about 10 days.

Table 7. Torsion/Dihedral Angles (Deg.) in conformer **B**

C(41)−O(4)−C(39)−C(38)	−55.9 (7)
C(41)−O(4)−C(39)−C(40)	127.3 (6)
C(39)−O(4)−C(41)−C(42)	153.5 (5)
C(39)−O(4)−C(41)−C(46)	−29.1 (8)
C(58)−N(3)−C(36)−C(35)	−19.3 (8)
C(58)−N(3)−C(36)−C(37)	160.3 (5)
C(36)−N(3)−C(58)−C(59)	−172.9 (5)
C(47)−N(4)−C(44)−C(43)	−7.6 (7)
C(47)−N(4)−C(44)−C(45)	172.0 (5)
C(44)−N(4)−C(47)−C(48)	−179.3 (4)
C(40)−C(35)−C(36)−N(3)	174.2 (5)
C(40)−C(35)−C(36)−C(37)	−5.4 (8)
C(36)−C(35)−C(40)−C(39)	1.0 (9)
N(3)−C(36)−C(37)−C(38)	−174.3 (5)
C(35)−C(36)−C(37)−C(38)	5.4 (8)
C(36)−C(37)−C(38)−C(39)	−0.9 (9)
C(37)−C(38)−C(39)−O(4)	179.6 (5)
C(37)−C(38)−C(39)−C(40)	−3.6 (9)
O(4)−C(39)−C(40)−C(35)	−179.6 (5)
C(38)−C(39)−C(40)−C(35)	3.5 (9)
O(4)−C(41)−C(42)−C(43)	177.8 (4)
C(46)−C(41)−C(42)−C(43)	0.3 (8)
O(4)−C(41)−C(46)−C(45)	−179.1 (5)
C(42)−C(41)−C(46)−C(45)	−1.8 (9)
C(41)−C(42)−C(43)−C(44)	1.7 (7)
C(42)−C(43)−C(44)−N(4)	177.4 (4)
C(42)−C(43)−C(44)−C(45)	−2.2 (7)
N(4)−C(44)−C(45)−C(46)	−178.9 (5)
C(43)−C(44)−C(45)−C(46)	0.7 (8)
C(44)−C(45)−C(46)−C(41)	1.3 (9)
N(4)−C(47)−C(48)−C(49)	2.1 (8)
N(4)−C(47)−C(48)−C(57)	−178.6 (5)
C(47)−C(48)−C(49)−O(5)	−1.5 (8)
C(47)−C(48)−C(49)−C(50)	−179.7 (5)
C(57)−C(48)−C(49)−O(5)	179.1 (5)
C(57)−C(48)−C(49)−C(50)	0.9 (7)
C(47)−C(48)−C(57)−C(55)	−1.1 (7)
C(47)−C(48)−C(57)−C(56)	179.1 (5)
C(49)−C(48)−C(57)−C(55)	178.3 (5)
C(49)−C(48)−C(57)−C(56)	−1.5 (7)
O(5)−C(49)−C(50)−C(51)	−178.3 (5)
C(48)−C(49)−C(50)−C(51)	−0.1 (8)
C(49)−C(50)−C(51)−C(56)	−0.3 (9)
C(50)−C(51)−C(56)−C(52)	−179.4 (6)
C(50)−C(51)−C(56)−C(57)	−0.3 (8)
C(56)−C(52)−C(53)−C(54)	−0.1 (9)
C(53)−C(52)−C(56)−C(51)	178.4 (5)

(Continued on next page)

Table 7. Torsion/Dihedral Angles (Deg.) in conformer **B** *(Continued)*

C(53)−C(52)−C(56)−C(57)	−0.8 (8)
C(52)−C(53)−C(54)−C(55)	1.3 (9)
C(53)−C(54)−C(55)−C(57)	−1.6 (9)
C(54)−C(55)−C(57)−C(48)	−179.2 (5)
C(54)−C(55)−C(57)−C(56)	0.6 (8)
C(51)−C(56)−C(57)−C(48)	1.2 (7)
C(51)−C(56)−C(57)−C(55)	−178.7 (5)
C(52)−C(56)−C(57)−C(48)	−179.7 (5)
C(52)−C(56)−C(57)−C(55)	0.5 (7)
N(3)−C(58)−C(59)−C(60)	3.3 (8)
N(3)−C(58)−C(59)−C(68)	178.7 (5)
C(58)−C(59)−C(60)−O(6)	−3.9 (8)
C(58)−C(59)−C(60)−C(61)	178.3 (5)
C(68)−C(59)−C(60)−O(6)	−179.5 (5)
C(68)−C(59)−C(60)−C(61)	2.8 (8)
C(58)−C(59)−C(68)−C(66)	3.2 (8)
C(58)−C(59)−C(68)−C(67)	−178.1 (5)
C(60)−C(59)−C(68)−C(66)	178.5 (5)
C(60)−C(59)−C(68)−C(67)	−2.9 (8)
O(6)−C(60)−C(61)−C(62)	−179.9 (6)
C(59)−C(60)−C(61)−C(62)	−2.1 (9)
C(60)−C(61)−C(62)−C(67)	1.4 (9)
C(61)−C(62)−C(67)−C(63)	−179.7 (6)
C(61)−C(62)−C(67)−C(68)	−1.4 (9)
C(67)−C(63)−C(64)−C(65)	−0.5 (10)
C(64)−C(63)−C(67)−C(62)	179.0 (6)
C(64)−C(63)−C(67)−C(68)	0.6 (9)
C(63)−C(64)−C(65)−C(66)	0.5 (10)
C(64)−C(65)−C(66)−C(68)	−0.6 (9)
C(65)−C(66)−C(68)−C(59)	179.5 (5)
C(65)−C(66)−C(68)−C(67)	0.8 (8)
C(62)−C(67)−C(68)−C(59)	2.1 (8)
C(62)−C(67)−C(68)−C(66)	−179.2 (5)
C(63)−C(67)−C(68)−C(59)	−179.5 (5)
C(63)−C(67)−C(68)−C(66)	−0.8 (8)

X-Ray Crystallography

Diffraction quality crystals of NDADPE were chosen and the single crystal X-ray diffraction data were collected. No absorption correction was applied. The lattice parameters were determined from least-squares analysis, and reflection data were integrated using the program SHELXTL [29a]. The structure was solved by direct methods using SHELXS-97 and refined by full-matrix least squares on F^2 with anisotropic displacement parameters for non-H atoms, using SHELXL-97 [29b]. The positions of all aromatic and aliphatic C−H hydrogen atoms were calculated geometrically, and a riding model was used in the refinement, with C−H distances in the range of 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. All the

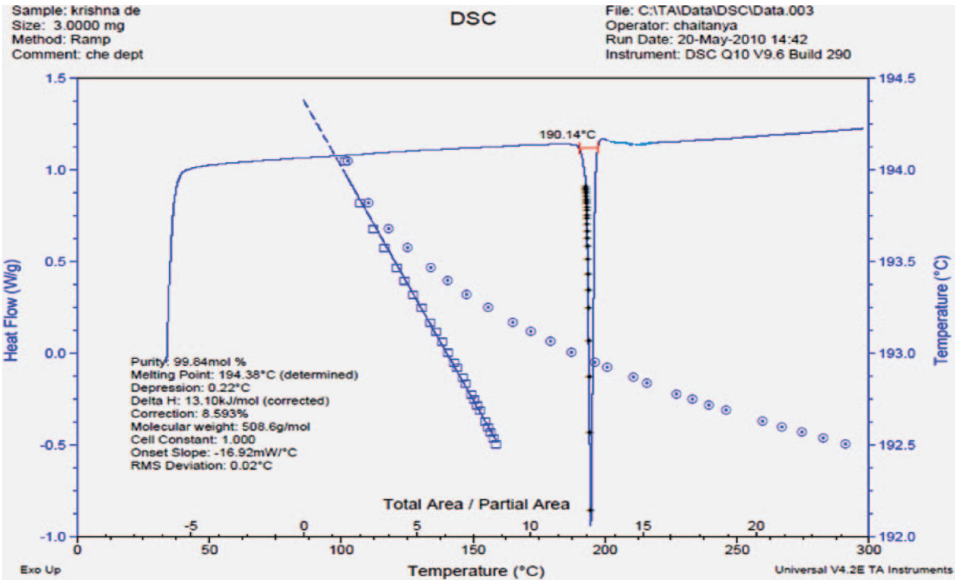

Figure 1. DSC thermogram of Schiff base NDADPE.

O—H and N—H hydrogens were refined from difference Fourier maps. The software used to prepare material for publication was Mercury 2.3 (Build RC4), ORTEP-3 and X-Seed [30]. The details of crystal data and refinements are given in Table 1. The geometrical parameters of hydrogen bonds in NDADPE are listed in Table 2. The selected bond lengths and bond angles of all the nonhydrogen atoms are given in Table 3. In Table 4, the list of atomic coordinates and equivalent isotropic displacement parameters of the nonhydrogen atoms are given. Table 5 lists the anisotropic displacement parameters of the nonhydrogen

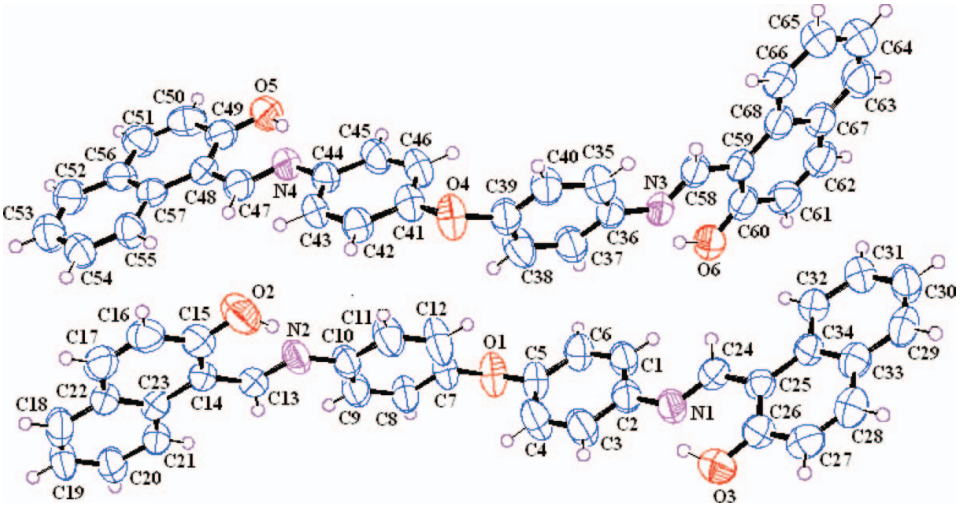

Figure 2. ORTEP representation of conformers **A** and **B** of NDADPE ligand. Thermal ellipsoids are drawn at 50% probability level.

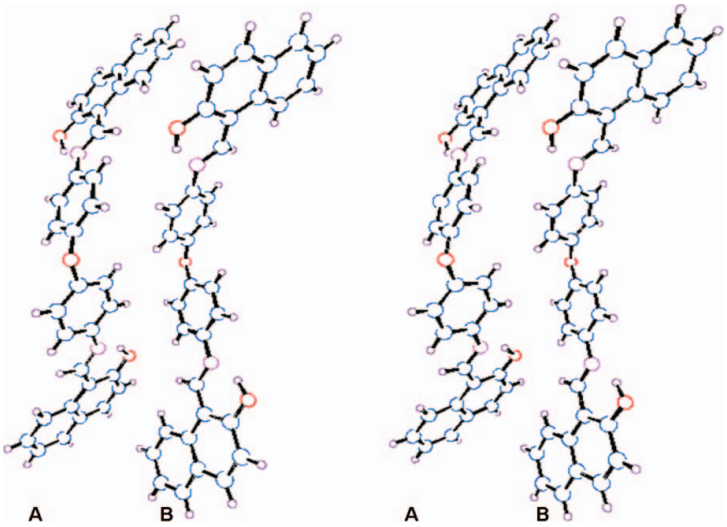

Figure 3. Stereovision of the two conformers **A** and **B**, of NDADPE in the asymmetric unit.

atoms. The lists of torsion and dihedral angles (deg.) in NDADPE are given in Tables 6 and 7.

Results and Discussion

The DSC thermogram of Schiff base NDADPE was given in Fig. 1. The endotherm at 194.38°C corresponds to its melting point. It was observed that there is no any endotherm before the melting point. It means that no solvent molecule has included in the crystal structure. The change in enthalpy (ΔH) of fusion in 13.0 KJ/mol

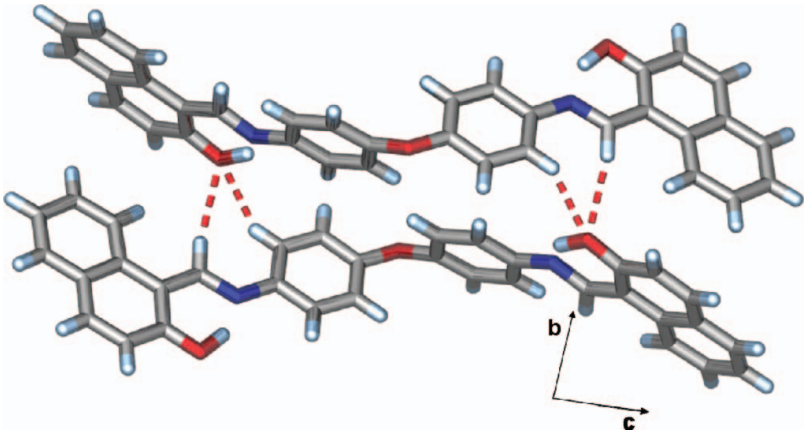

Figure 4. Supramolecular dimer of conformer **A.** The two conformer **A** molecules interacting with bifurcated C—H···O hydrogen bonds.

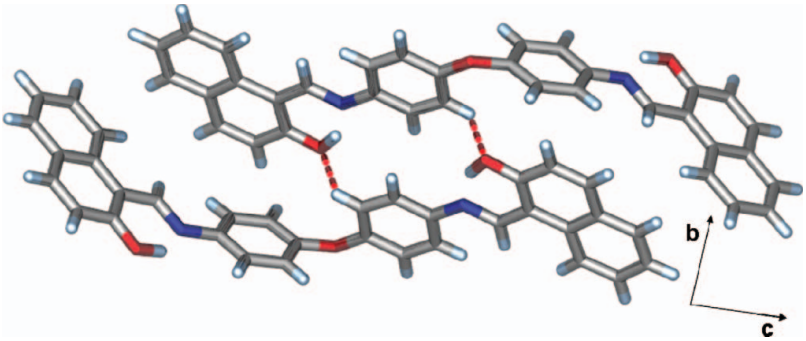

Figure 5. Supramolecular dimer of conformer **B**. The two conformer **B** molecules interacting with the C—H···O hydrogen bonds.

Crystal Structure Analysis

The compound NDADPE was crystallizes in triclinic *P*-1 space group with two symmetry independent molecules in the asymmetric unit ($Z' = 2$) (Fig. 2). These two molecules are conformational isomers (Fig. 3). The torsion angles at diphenylether linkage in two molecules are different with each other (C6—C5—O1—C7; −134.25°; C42—C41—O4—C39; 153.5°). In the two conformers, the four naphthol O—H groups form intramolecular interactions with imine N-atoms with O—H···N hydrogen bonds. The two conformers (**A** and **B**) form different hydrogen bonding in the crystal structure. The inversion related molecules of conformer **A**, forms a supramolecular dimer with bifurcated

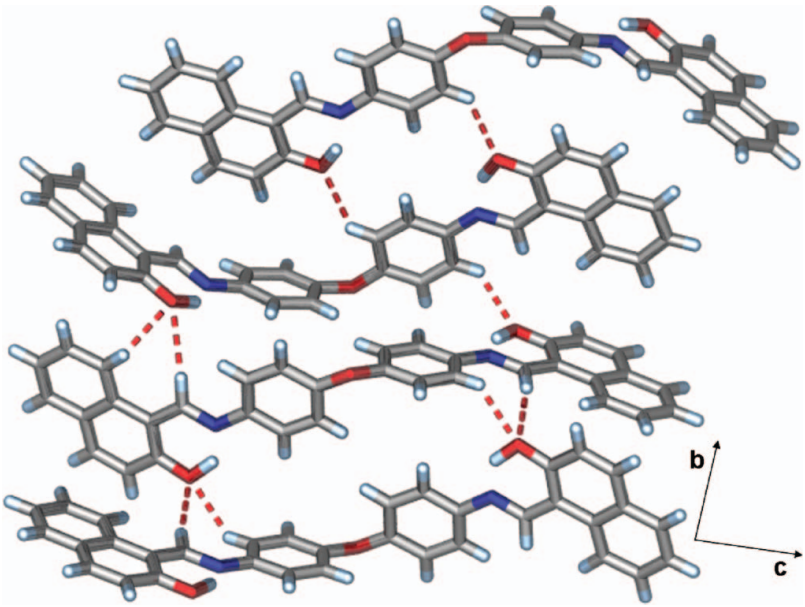

Figure 6. Spatial disposition of the two conformers **A** and **B** as viewed along the bc-planes. The two supramolecular dimers of conformer **A** and **B** connect via C—H···O and bifurcated C—H···O hydrogen bonds.

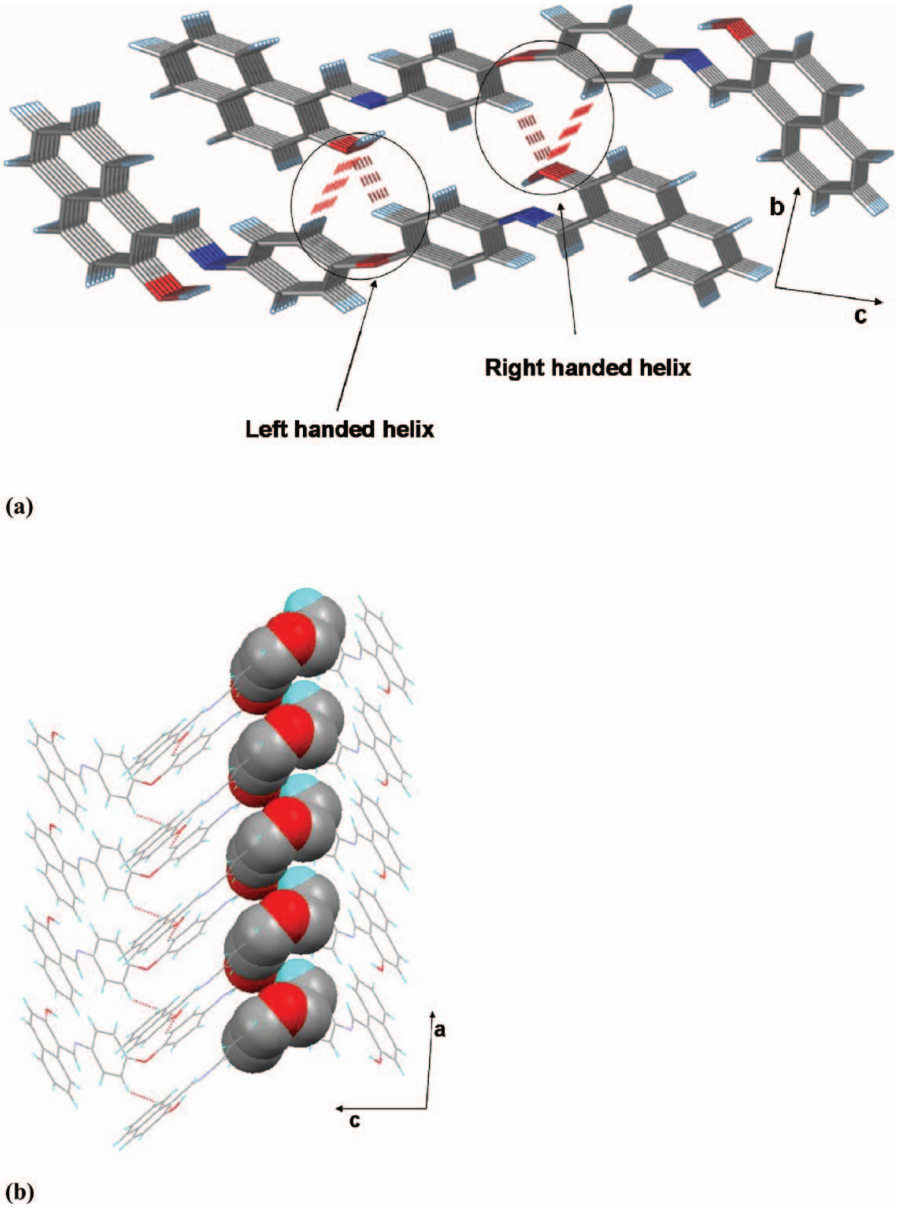

Figure 7. Molecular packing array of conformer **B**, (a) viewed along bc plane; ((b) viewed along ac plane (atomic segments forming the helical structure shown in CPK spacefilling manner).

C—H···O hydrogen bond synthon (Fig. 4). Conformer **B**, also forms a supramolecular dimer synthon with C—H···O hydrogen bonds (Fig. 5). The two dimers of **A** and **B** conformers interact with the single and bifurcated C—H···O hydrogen bonds (Fig. 6). But the molecules of conformer **B**, form a supramolecular helix due to bifurcated C—H···O hydrogen bond synthons [Figs. 7(a) and (b)]. The dimers of conformer **A** and helices of

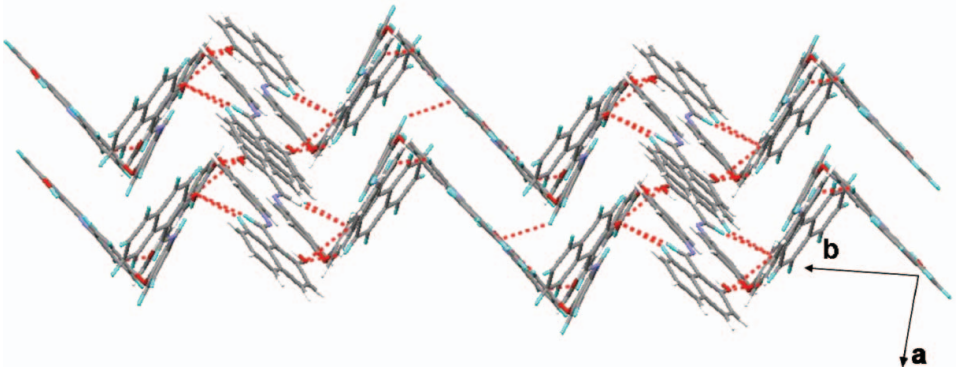

Figure 8. Corrugated layered structure formed by conformers, **A** and **B** of NDADPE, viewed over ab plane.

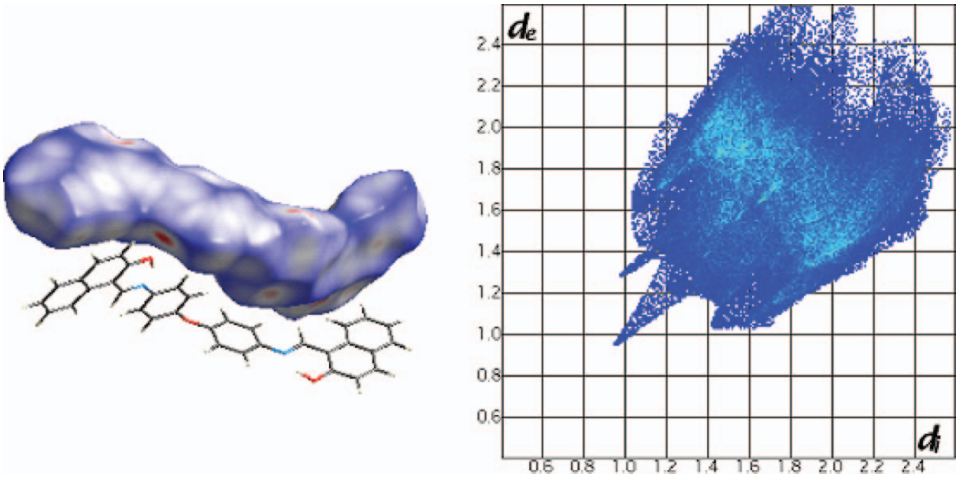

Figure 9. Hirshfeld surface and 2D fingerplots of conformer **A**.

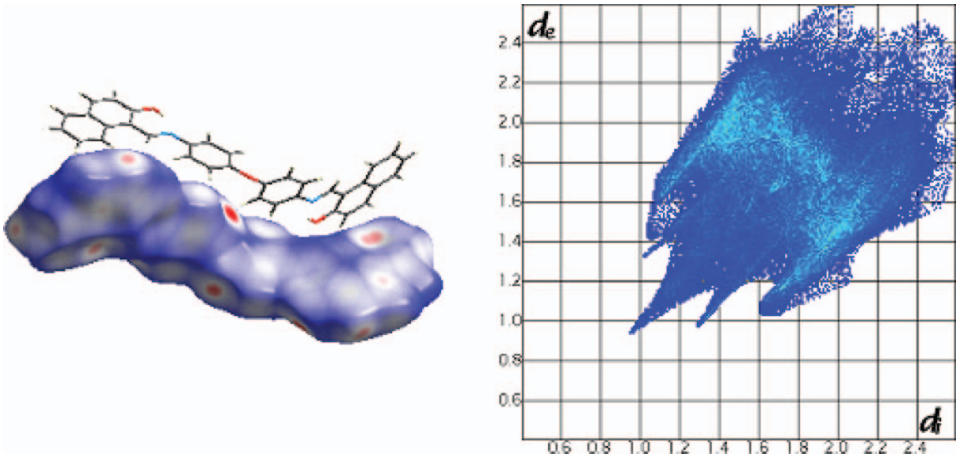

Figure 10. Hirshfeld surface and 2D fingerplots of conformer **B.**

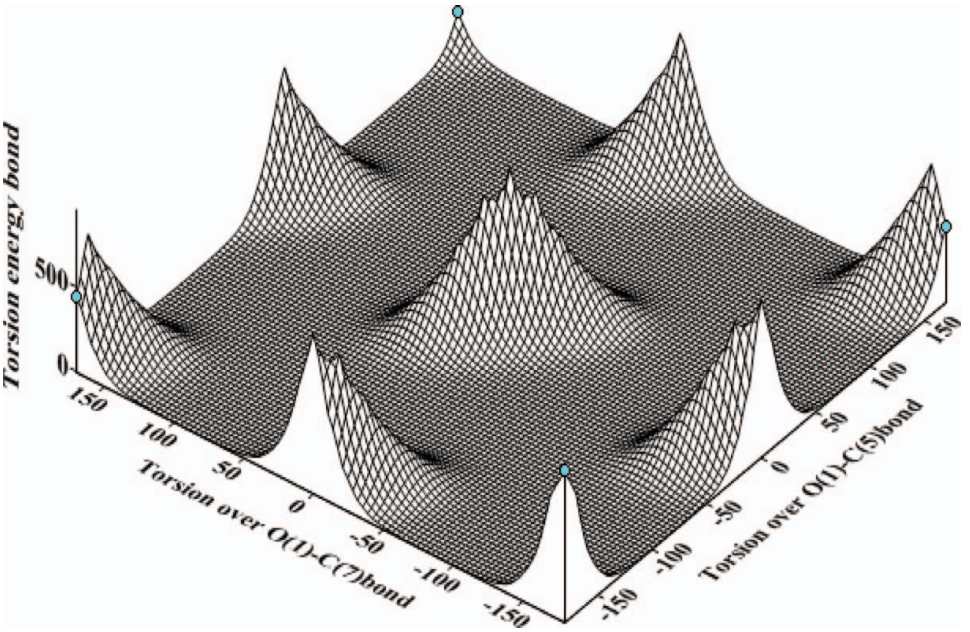

Figure 11. Stereographic view of NDADPE after global minimization.

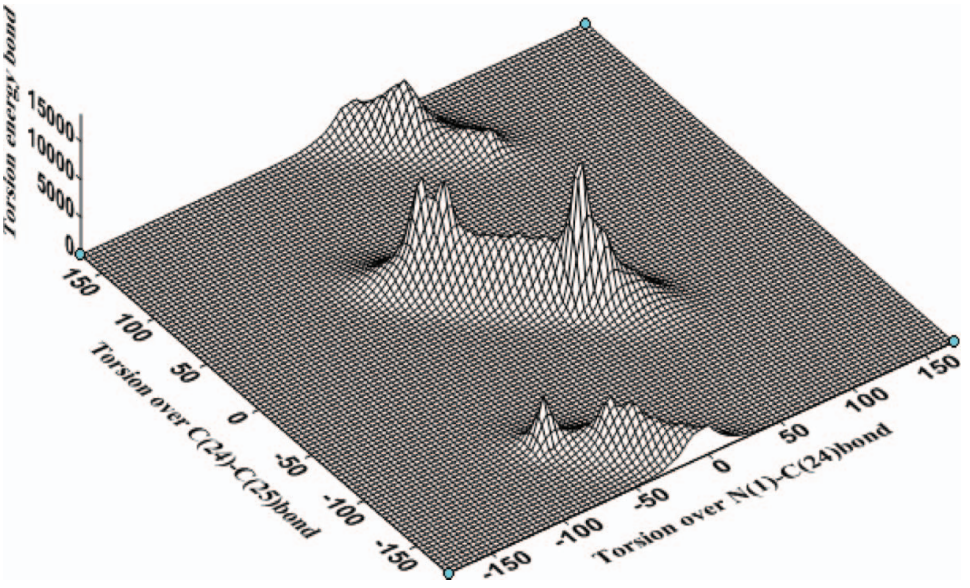

Figure 12. Double dihedral torsional energy plot over C(7)−O(1)−C(5) bonds; (left) over O(1)−C(7) and (right) O(1)−C(5) bonds from -180 to $+180^{\circ}$ (Refer to Figure 2 for numbering scheme for the atoms).

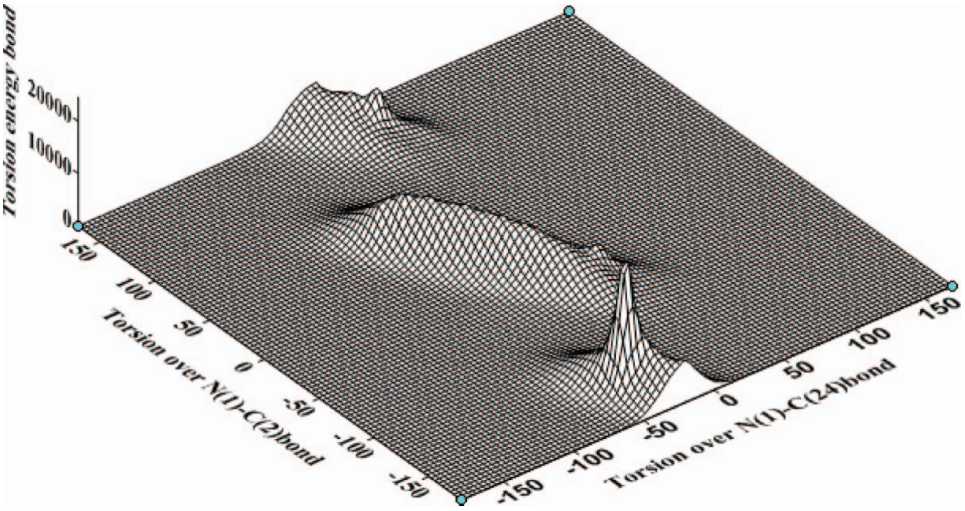

Figure 13. Double dihedral torsional energy plot over N(1)—C(24)—C(25) bonds; (left) over C(24)—C(25) and (right) N(1)—C(24) bonds from −180 to +180° (Refer to Figure 2 for numbering scheme for atoms).

conformer **B** connect via C—H···O hydrogen bonding. The overall molecular aggregation of conformers, **A** and **B** of NDADPE results in a corrugated tape like structure (Fig. 8), and in such corrugation conformers of **B** form a helical structure due to the bifurcated C—H···O interactions, [Figs. 7(a) and 9b)].

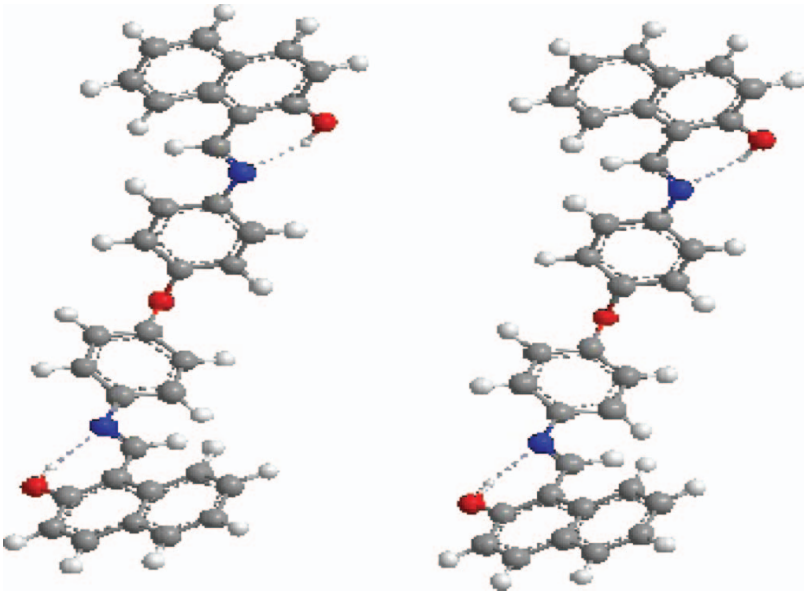

Figure 14. Double dihedral torsional energy plot over C(2)—N(1)—C(24) bonds; (left) over N(1)—C(2) (right) and N(1)—C(24) bonds from −180 to +180° (Refer to Figure 2 for numbering scheme for atoms).

Hirshfeld Surface Analysis

The challenging task is to successfully differentiate the conformational polymorphism by analyzing the crystal structures. The Hirshfeld surface analysis makes it easy to differentiate the polymorphic forms and conformational polymorphism [31]. Since the Hirshfeld surfaces are directly depend on the molecular environment, these are unique in the given crystal structure and they depend on the number of crystallographically independent molecules present in the asymmetric unit. Here, we have applied the Hirshfeld surface and 2D fingerprint plots to analyze the conformers **A** and **B** of NDADPE.

The 2D fingerprint plots of Schiff base NDADPE were derived from the Hirshfeld surface by plotting the fraction of points on the surface as a function of the pair (d_i, d_e). Each point on the standard 2D graph represents a bin formed by discrete intervals of d_i and d_e (0.01 × 0.01 Å), and the points are colored as a function of the fraction of surface points in that bin, with a range from blue (relatively fewer points) through green (moderate fraction). The Hirshfeld surfaces and 2D fingerprint plots are given in Figs. 9 and 10. It is obvious from this analysis that the two conformers **A** and **B** of NDADPE are completely different and exhibit the conformational isomerism. Inspection of the fingerprint plots in Figs. 9 and 10 highlights the major differences between the two conformers. Conformer **A** features the diffuse region of the blue points between the hydrogen bond spikes, while this feature is sharpened with extra spike in the fingerprint plot of conformer **B**.

Molecular Modeling

Molecular modeling was performed on NDADPE using chemoffice ultra (version 11.0) to arrive at the molecular geometric properties and an intramolecular interaction such as H-bonding, etc. In Table 2 are placed some of the intramolecular and intermolecular hydrogen bond lengths and angles whereas in Table 3, some of the most relevant bond lengths and bond angles obtained from X-ray crystallography along with those from molecular modeling are shown. There is an excellent agreement among the respective experimental and modeled data. In Figs 11, 12, and 13 are shown the torsional energy conformational 3D plots. It is found that the free molecule has the stablest disposition with C(7)−O(1)−C(5)−C(6) dihedral angle of C(7)−O(1)−C(5) and with C(2)−N(1)−C(24)−C(25) of C(2)−N(1)−C(24) this is slightly different from the experimental X-ray data of Tables 6 and 7. The actual dihedral angles at their moieties toward slight positive and negative drifts in comparison to the gases our state free molecule is understood to be the reason for the conformational isomerism as on crystallization and molecular aggregation. The structure of the energy minimized free molecule is shown in Fig. 14 in its stereographic projection.

Conclusion

We have presented herein the structural and characterization of new bis-N,O-bidentate Schiff base bis bidentate ligand. The compound NDADPE 2,2′-(4,4′-oxybis(4,1-phenylene)bis(azan-1-yl-1ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-1-ol was synthesized and confirmed by IR, ^{1}H NMR, and Mass spectral data. Its crystal structure was determined by single crystal X-ray diffraction. The crystal structure was described in terms of supramolecular dimers and helices and the overall structure corrugated layered structure. The Hirshfeld surface analysis associated with 2D fingerprint plots reveal the existence of conformational isomerism of the NDADPE. The structural geometry data such as bond

lengths, bond angles, and dihedral angles obtain by molecular modeling lie as near mean to those of conformers **A** and **B** suggesting the conformational isomerism in NDADPE.

Supplementary Material

Supplementary material consists of IR, ^{1}H NMR, and Mass spectral data. Crystallographic information file is available in .cif format.

Acknowledgments

V. KRISHNA expresses his thanks to the Director, NIT Warangal for the Institute Research Fellowship. The authors thank Dr. P. Raghavaiah, scientific officer, University of Hyderabad, Hyderabad for the help in collecting the single crystal X-ray diffraction data.

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