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1,1′-{(Hexane-1,6-diyl)bis[(azaniumylylidene)methanylylidene]}bis(naphthalen-2-olate)

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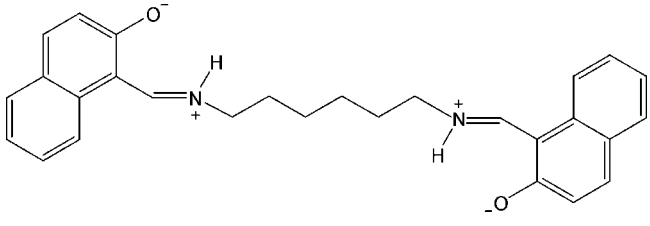
The whole molecule of the title Schiff base compound, $C_{28}H_{28}N_2O_2$, is generated by inversion symmetry. It is formed from two units of *ortho*-hydroxynaphthaldehyde bridged with 1,6-diaminohexane. The N atoms are protonated and, thus, the structure is a bis-zwitterionic compound in the solid state. The zwitterion shows strong intramolecular N—H···O hydrogen bonds between the iminium N and the naphthalenolate O atoms.

Keywords: crystal structure; 1,6-diaminohexane; 2-hydroxy-1-naphthaldehyde; hydrogen bonding; elemental analysis..

CCDC reference: 1032693

1. Related literature

For the synthesis of similar compounds, see: Ramos Silva *et al.* (2009); Li *et al.* (2007); Zhu *et al.* (2006); Sampath Kumar *et al.* (2010); Bhattacharjee *et al.* (2012). For their applications, see: Ourari *et al.* (2006, 2008); Ouari *et al.* (2010, 2015). For related crystal structures, see: Yuan & Li (2013); Paul & Kubicki (2009). For the biological activity of Schiff bases, see: Zayed *et al.* (2015); Abou-Hussein & Linert (2014); Sadeek *et al.* (2013).



2. Experimental

2.1. Crystal data

$C_{28}H_{28}N_2O_2$
M_r = 424.52
Orthorhombic, *Pbcn*
a = 23.722 (1) Å
b = 8.8117 (3) Å
c = 10.3903 (5) Å
V = 2171.90 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 173 K
0.36 × 0.16 × 0.08 mm

2.2. Data collection

Nonius KappaCCD diffractometer
17177 measured reflections
2500 independent reflections
1285 reflections with $I > 2\sigma(I)$
R_{int} = 0.082

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.160
S = 0.99
2500 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1	0.99 (3)	1.74 (3)	2.587 (2)	141 (2)

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 200); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2013*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: MW2128).

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Acta Cryst. (2015). E**71**, o51–o52 [https://doi.org/10.1107/S2056989014027236]

1,1′-{(Hexane-1,6-diyl)bis[(azaniumylylidene)methanylylidene]}bis-(naphthalen-2-olate)

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S1. Experimental

The Schiff base ligand was prepared in 67% yield by condensation between 58 mg (0.5 mmole) of 1,6-diaminohexane and 172 mg (1 mmole) of 2-hydroxy-1-naphthaldehyde in methanol (12 mL). The mixture was refluxed and stirred under a nitrogen atmosphere for 3 hours. The precipitate obtained was filtered, washed with methanol and diethyl ether and dried in vacuum overnight. The product was recrystallized from dimethyl sulfoxide at room temperature over a period of a week. The yellow, single crystals of $C_{28}H_{28}O_2N_2$ obtained were of X-ray quality. Elemental analysis: calculated for $C_{28}H_{28}O_2N_2$: C 79.20, H 6.65, N 6.60%; found: C 78.84, H 6.63, N 6.78%.

S1.1. Refinement

The iminium H atom was located from a difference Fourier map and refined isotropically. C-bound H atoms were included in calculated positions and treated as riding atoms: C—H = 0.95 Å (CH) or 0.99 Å (CH_2) with Uiso(H) = 1.2Ueq(C—$H_{aromatics}$). .

S2. Results and discussion

The synthesis of the Schiff base is similar to those described in the literature (Ramos Silva *et al.*, 2009; Li *et al.*, 2007; Zhu *et al.*, 2006; Sampath Kumar *et al.*, 2010; Bhattacharjee *et al.*, 2012). These ligands are also currently applied in coordination chemistry for the synthesis of Schiff base complexes of transition metals (Ouari *et al.*, 2015; Ouari *et al.*, 2010; Ourari *et al.*, 2008; Ourari *et al.*, 2006). Compounds of the type of the title molecule possess diverse biological properties such as anti-anxiety, anti-depressant (Zayed *et al.*, 2015) and anti-tumor activities as well as antibacterial and fungicidal properties (Abou-Hussein *et al.*, 2014; Sadeek *et al.*, 2013). We report here the synthesis of title compound and its crystal structure.

A perspective view of the title molecule, which has crystallographically- imposed centrosymmetry, is shown in Fig. 1. The intramolecular N1—H1N···O1 hydrogen bond forces the O1–C1–C10–C11–N1 unit into near planarity (rms deviation 0.005 Å) with the consequence that the naphthalene portion is nearly co-planar with it (dihedral angle 1.20 (8)°).

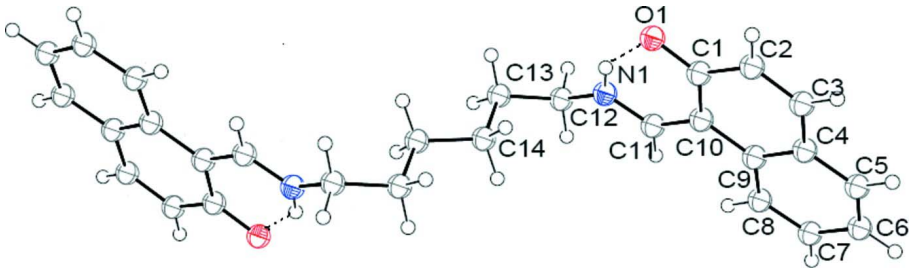

Figure 1

The molecular geometry of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines. Only the non-H atoms of the asymmetric unit are labelled.

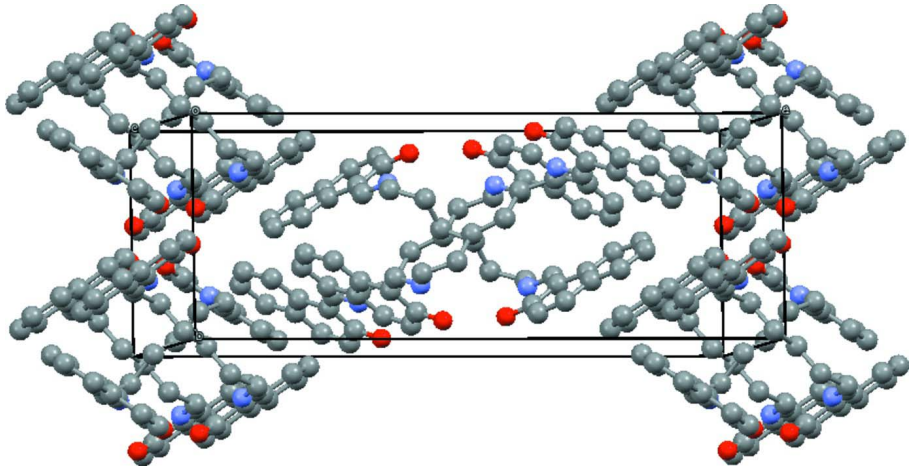

Figure 2

Crystal packing of the title compound viewed along the *c* axis.

1,1′-{(Hexane-1,6-diyl)bis[(azaniumylylidene)methanylylidene]}bis(naphthalen-2-olate)

Crystal data

$C_{28}H_{28}N_2O_2$
M_r = 424.52
Orthorhombic, *Pbcn*
a = 23.722 (1) Å
b = 8.8117 (3) Å
c = 10.3903 (5) Å
V = 2171.90 (16) Å^3
Z = 4
F(000) = 904
D_x = 1.298 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 8957 reflections
θ = 1.0–27.5°
μ = 0.08 mm^{-1}
T = 173 K
Prism, yellow
0.36 × 0.16 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and ω scans
17177 measured reflections
2500 independent reflections
1285 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −25→30
k = −10→11
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.160$
$S = 0.99$
2500 reflections
150 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0047 (14)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.42177 (9)	0.1531 (2)	0.9435 (2)	0.0314 (5)
C2	0.42703 (9)	0.1083 (2)	1.0765 (2)	0.0336 (5)
H2	0.4575	0.0451	1.1016	0.040*
C3	0.38971 (9)	0.1541 (2)	1.1655 (2)	0.0351 (5)
H3	0.3951	0.1236	1.2524	0.042*
C4	0.34205 (8)	0.2473 (2)	1.1344 (2)	0.0319 (5)
C5	0.30246 (9)	0.2899 (2)	1.2282 (2)	0.0389 (6)
H5	0.3080	0.2583	1.3147	0.047*
C6	0.25612 (9)	0.3759 (2)	1.1985 (2)	0.0411 (6)
H6	0.2297	0.4032	1.2631	0.049*
C7	0.24870 (9)	0.4224 (2)	1.0717 (2)	0.0416 (6)
H7	0.2167	0.4820	1.0497	0.050*
C8	0.28662 (9)	0.3839 (2)	0.9781 (2)	0.0359 (5)
H8	0.2806	0.4182	0.8926	0.043*
C9	0.33434 (8)	0.2945 (2)	1.0054 (2)	0.0294 (5)
C10	0.37521 (8)	0.24816 (18)	0.90863 (19)	0.0283 (5)
C11	0.36924 (9)	0.2932 (2)	0.7794 (2)	0.0311 (5)
H11	0.3382	0.3570	0.7587	0.037*
C12	0.39177 (9)	0.2937 (2)	0.55205 (19)	0.0372 (6)
H12A	0.3640	0.3775	0.5496	0.045*
H12B	0.3747	0.2051	0.5084	0.045*
C13	0.44381 (9)	0.3416 (2)	0.4786 (2)	0.0369 (5)
H13A	0.4324	0.3750	0.3914	0.044*
H13B	0.4689	0.2526	0.4687	0.044*
C14	0.47653 (8)	0.4686 (2)	0.54272 (19)	0.0370 (6)
H14A	0.4932	0.4303	0.6238	0.044*
H14B	0.4502	0.5518	0.5650	0.044*
N1	0.40291 (8)	0.25438 (18)	0.68565 (17)	0.0333 (5)

O1	0.45832 (6)	0.10907 (16)	0.86112 (13)	0.0403 (4)
H1N	0.4349 (11)	0.195 (2)	0.720 (2)	0.064 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0307 (12)	0.0318 (11)	0.0318 (13)	−0.0016 (8)	0.0006 (10)	−0.0004 (9)
C2	0.0348 (12)	0.0326 (10)	0.0335 (13)	0.0006 (9)	−0.0050 (10)	0.0047 (9)
C3	0.0394 (14)	0.0371 (11)	0.0289 (12)	−0.0057 (9)	−0.0019 (10)	0.0040 (9)
C4	0.0311 (12)	0.0321 (10)	0.0325 (13)	−0.0056 (8)	−0.0003 (10)	−0.0021 (9)
C5	0.0399 (14)	0.0449 (13)	0.0319 (13)	−0.0090 (10)	0.0026 (11)	−0.0056 (9)
C6	0.0307 (13)	0.0495 (13)	0.0431 (15)	−0.0056 (10)	0.0073 (11)	−0.0162 (10)
C7	0.0332 (13)	0.0438 (12)	0.0479 (15)	0.0008 (9)	0.0003 (11)	−0.0105 (11)
C8	0.0347 (13)	0.0363 (11)	0.0367 (13)	0.0009 (9)	−0.0002 (10)	−0.0019 (9)
C9	0.0290 (12)	0.0284 (10)	0.0308 (12)	−0.0039 (8)	−0.0008 (9)	−0.0017 (8)
C10	0.0277 (11)	0.0299 (10)	0.0275 (12)	−0.0009 (8)	−0.0026 (9)	0.0014 (8)
C11	0.0289 (12)	0.0301 (10)	0.0342 (13)	−0.0002 (8)	−0.0012 (10)	−0.0004 (9)
C12	0.0397 (13)	0.0426 (12)	0.0293 (13)	−0.0013 (9)	−0.0026 (10)	0.0060 (9)
C13	0.0398 (13)	0.0430 (12)	0.0281 (12)	0.0014 (10)	0.0010 (10)	0.0041 (9)
C14	0.0413 (13)	0.0409 (11)	0.0288 (13)	0.0008 (10)	0.0005 (10)	0.0027 (9)
N1	0.0349 (10)	0.0380 (10)	0.0271 (11)	0.0018 (8)	0.0010 (9)	0.0042 (7)
O1	0.0383 (9)	0.0474 (8)	0.0352 (9)	0.0120 (7)	0.0035 (7)	0.0006 (7)

Geometric parameters (Å, º)

C1—O1	1.279 (2)	C8—H8	0.9500
C1—C10	1.433 (3)	C9—C10	1.455 (3)
C1—C2	1.442 (3)	C10—C11	1.408 (3)
C2—C3	1.343 (3)	C11—N1	1.305 (2)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.434 (3)	C12—N1	1.455 (2)
C3—H3	0.9500	C12—C13	1.511 (3)
C4—C5	1.405 (3)	C12—H12A	0.9900
C4—C9	1.416 (3)	C12—H12B	0.9900
C5—C6	1.370 (3)	C13—C14	1.516 (3)
C5—H5	0.9500	C13—H13A	0.9900
C6—C7	1.391 (3)	C13—H13B	0.9900
C6—H6	0.9500	C14—C14^{i}	1.528 (4)
C7—C8	1.367 (3)	C14—H14A	0.9900
C7—H7	0.9500	C14—H14B	0.9900
C8—C9	1.408 (3)	N1—H1N	0.99 (3)
O1—C1—C10	122.03 (18)	C11—C10—C1	118.94 (18)
O1—C1—C2	119.99 (18)	C11—C10—C9	120.86 (18)
C10—C1—C2	117.97 (19)	C1—C10—C9	120.19 (18)
C3—C2—C1	121.40 (19)	N1—C11—C10	125.19 (19)
C3—C2—H2	119.3	N1—C11—H11	117.4
C1—C2—H2	119.3	C10—C11—H11	117.4

C2—C3—C4	122.44 (19)	N1—C12—C13	113.56 (17)
C2—C3—H3	118.8	N1—C12—H12A	108.9
C4—C3—H3	118.8	C13—C12—H12A	108.9
C5—C4—C9	119.48 (19)	N1—C12—H12B	108.9
C5—C4—C3	121.6 (2)	C13—C12—H12B	108.9
C9—C4—C3	118.92 (18)	H12A—C12—H12B	107.7
C6—C5—C4	121.9 (2)	C12—C13—C14	113.75 (18)
C6—C5—H5	119.1	C12—C13—H13A	108.8
C4—C5—H5	119.1	C14—C13—H13A	108.8
C5—C6—C7	118.5 (2)	C12—C13—H13B	108.8
C5—C6—H6	120.7	C14—C13—H13B	108.8
C7—C6—H6	120.7	H13A—C13—H13B	107.7
C8—C7—C6	121.1 (2)	C13—C14—C14^{i}	112.7 (2)
C8—C7—H7	119.4	C13—C14—H14A	109.1
C6—C7—H7	119.4	C14^{i}—C14—H14A	109.1
C7—C8—C9	121.7 (2)	C13—C14—H14B	109.1
C7—C8—H8	119.2	C14^{i}—C14—H14B	109.1
C9—C8—H8	119.2	H14A—C14—H14B	107.8
C8—C9—C4	117.29 (18)	C11—N1—C12	122.55 (19)
C8—C9—C10	123.64 (18)	C11—N1—H1N	109.7 (14)
C4—C9—C10	119.06 (17)	C12—N1—H1N	127.7 (14)
O1—C1—C2—C3	179.21 (17)	C3—C4—C9—C10	−0.5 (3)
C10—C1—C2—C3	−0.1 (3)	O1—C1—C10—C11	0.7 (3)
C1—C2—C3—C4	1.1 (3)	C2—C1—C10—C11	180.00 (16)
C2—C3—C4—C5	177.89 (18)	O1—C1—C10—C9	179.50 (17)
C2—C3—C4—C9	−0.8 (3)	C2—C1—C10—C9	−1.2 (3)
C9—C4—C5—C6	0.3 (3)	C8—C9—C10—C11	0.8 (3)
C3—C4—C5—C6	−178.41 (18)	C4—C9—C10—C11	−179.71 (16)
C4—C5—C6—C7	−0.4 (3)	C8—C9—C10—C1	−178.02 (17)
C5—C6—C7—C8	−0.1 (3)	C4—C9—C10—C1	1.5 (3)
C6—C7—C8—C9	0.7 (3)	C1—C10—C11—N1	0.5 (3)
C7—C8—C9—C4	−0.8 (3)	C9—C10—C11—N1	−178.29 (17)
C7—C8—C9—C10	178.74 (17)	N1—C12—C13—C14	−53.7 (2)
C5—C4—C9—C8	0.3 (3)	C12—C13—C14—C14^{i}	−171.2 (2)
C3—C4—C9—C8	179.01 (16)	C10—C11—N1—C12	174.49 (16)
C5—C4—C9—C10	−179.23 (16)	C13—C12—N1—C11	139.58 (19)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1	0.99 (3)	1.74 (3)	2.587 (2)	141 (2)