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1,1'-[(1*E*,11*E*)-5,8-Dioxa-2,11-diazonia-dodeca-1,11-diene-1,12-diyl]dinaphthalen-2-olate

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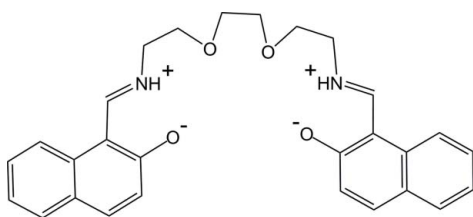
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.068; wR factor = 0.224; data-to-parameter ratio = 13.3.

The title compound, $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_4$, crystallizes in a zwitterionic form with deprotonated naphthol hydroxy groups and protonated imine N atoms. The asymmetric unit contains one half-molecule located on a twofold rotation axis. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds occur and the two bicyclic ring systems form a dihedral angle of $64.2(1)^\circ$. In the crystal, weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into layers parallel to the bc plane.

Related literature

For applications of Schiff bases in coordination chemistry, see: Osowle (2008). For related structures, see: Etemadi *et al.* (2004); Liu *et al.* (2010); Farag *et al.* (2010; 2011).



Experimental

Crystal data

$\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_4$
 $M_r = 456.52$
 Orthorhombic, $Pcca$
 $a = 44.704(4)$ Å
 $b = 6.3576(6)$ Å
 $c = 8.2074(9)$ Å
 $V = 2332.6(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.50 \times 0.37 \times 0.11$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
 $T_{\min} = 0.958$, $T_{\max} = 0.991$
 9099 measured reflections
 2060 independent reflections
 1162 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.081$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.224$
 $S = 1.00$
 2060 reflections
 155 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.86	1.85	2.542 (5)	136
$\text{C14}-\text{H14B}\cdots\text{O2}^i$	0.97	2.58	3.382 (5)	141
$\text{C12}-\text{H12B}\cdots\text{O1}^{ii}$	0.97	2.50	3.257 (5)	134

Symmetry codes: (i) $x, -y + 1, z + \frac{1}{2}$; (ii) $x, y - 1, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5066).

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supporting information

Acta Cryst. (2011). E67, o1074 [doi:10.1107/S1600536811012219]

1,1'-[(1*E*,11*E*)-5,8-Dioxa-2,11-diazoniadodeca-1,11-diene-1,12-diyl]dinaphthalen-2-olate

Yanju Liu, Kai Liu, Zhiqiang Cao and Meiju Niu

S1. Comment

Schiff bases have various applications in coordination chemistry (Oswole, 2008). Herewith we present the title compound (I), which is a new crowned Schiff base.

In (I) (Fig. 1), all bond lengths and angles are usual and comparable with those observed in the related compounds (Etemadi *et al.*, 2004; Liu *et al.*, 2010; Farag *et al.*, 2010, 2011). Each molecule is situated on a twofold rotational axis. Intramolecular N—H···O hydrogen bonds (Table 1) influence the molecular conformation - two bicycles form a dihedral angle of 64.2 (1)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 1) link molecules into layers parallel to *bc* plane.

S2. Experimental

The title compound was synthesized by adding drop-wise a solution of 3,6-dioxa-1,8-diaminooctane (1.48 g, 10 mmol) in absolute methanol (10 mL) to a methanol solution (20 mL) of 2-hydroxy-1-naphthaldehyde (3.4438 g, 20 mmol) under stirring at room temperature. The resultant reaction mixture was then refluxed for 5 h, cooled and concentrated under reduced pressure, and then the residue was retained at -268 K for overnight. The bright yellow crystal suitable for X-ray analysis was formed, filtered and dried under reduced pressure. Yield: 82%. Analysis found: C 73.07, H 6.06, N 6.51%; calculated for C₂₈H₂₈N₂O₄ (M_r=456.54): C 73.66, H 6.18, N 6.14%.

S3. Refinement

C-bound H atoms were geometrically positioned [C—H 0.93–0.97 Å]. Atom H1A was located on a difference map, but placed in idealized position [N—H 0.86 Å]. All H atoms were refined as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{iso}}$ of the parent atom.

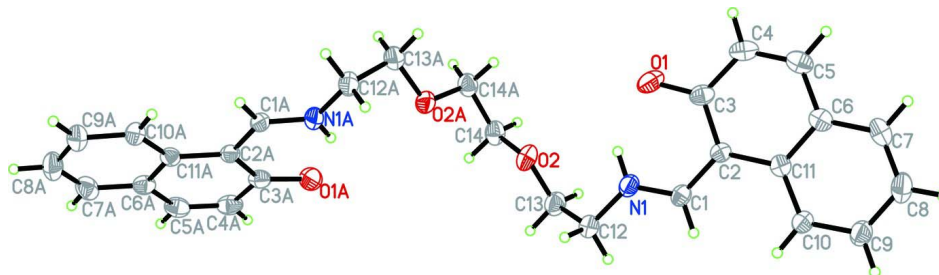


Figure 1

The molecular structure of (I) showing the atomic labels and 30% probability displacement ellipsoids. Symmetry code: (A) $-x + 1/2, -y + 1, z$.

1,1'-[(1E,11E)-5,8-Dioxa-2,11-diazoniadodeca-1,11-diene-1,12-diyl]dinaphthalen-2-olate*Crystal data*C₂₈H₂₈N₂O₄ $M_r = 456.52$ Orthorhombic, *Pcca*

Hall symbol: -P 2a 2ac

 $a = 44.704$ (4) Å $b = 6.3576$ (6) Å $c = 8.2074$ (9) Å $V = 2332.6$ (4) Å³ $Z = 4$ $F(000) = 968$ $D_x = 1.300$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1398 reflections

 $\theta = 2.7$ – 22.6° $\mu = 0.09$ mm⁻¹ $T = 298$ K

Block, yellow

 $0.50 \times 0.37 \times 0.11$ mm*Data collection*Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.958$, $T_{\max} = 0.991$

9099 measured reflections

2060 independent reflections

1162 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.081$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.8^\circ$ $h = -53 \rightarrow 28$ $k = -7 \rightarrow 7$ $l = -9 \rightarrow 9$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.068$ $wR(F^2) = 0.224$ $S = 1.00$

2060 reflections

155 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.088P)^2 + 3.8548P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.21$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ e Å⁻³Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0055 (16)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.16369 (7)	0.4462 (5)	0.0795 (4)	0.0479 (10)
H1A	0.1720	0.5537	0.1239	0.058*
C2	0.11853 (8)	0.6454 (6)	0.0728 (5)	0.0370 (9)
O1	0.15974 (7)	0.8123 (5)	0.2021 (4)	0.0628 (10)
O2	0.22075 (6)	0.4082 (5)	0.2116 (4)	0.0541 (9)
C11	0.08741 (8)	0.6581 (6)	0.0253 (5)	0.0388 (10)
C1	0.13580 (8)	0.4646 (6)	0.0400 (5)	0.0402 (10)
H1	0.1266	0.3525	-0.0129	0.048*
C6	0.07056 (9)	0.8373 (6)	0.0703 (5)	0.0464 (11)
C3	0.13216 (10)	0.8124 (6)	0.1618 (5)	0.0463 (11)
C10	0.07267 (8)	0.5045 (7)	-0.0672 (5)	0.0469 (11)
H10	0.0832	0.3861	-0.1007	0.056*

C12	0.18209 (9)	0.2620 (7)	0.0560 (6)	0.0505 (12)
H12A	0.1941	0.2793	-0.0415	0.061*
H12B	0.1694	0.1397	0.0410	0.061*
C4	0.11373 (10)	0.9880 (7)	0.2060 (6)	0.0557 (12)
H4	0.1220	1.0978	0.2657	0.067*
C5	0.08502 (10)	0.9976 (7)	0.1636 (5)	0.0550 (12)
H5	0.0739	1.1138	0.1962	0.066*
C8	0.02674 (10)	0.6990 (9)	-0.0632 (6)	0.0650 (14)
H8	0.0067	0.7120	-0.0922	0.078*
C7	0.04054 (10)	0.8504 (8)	0.0252 (6)	0.0572 (13)
H7	0.0296	0.9677	0.0571	0.069*
C9	0.04327 (9)	0.5238 (8)	-0.1096 (6)	0.0553 (12)
H9	0.0342	0.4182	-0.1703	0.066*
C13	0.20210 (9)	0.2282 (7)	0.1994 (6)	0.0566 (13)
H13A	0.1903	0.2115	0.2979	0.068*
H13B	0.2141	0.1027	0.1840	0.068*
C14	0.24105 (8)	0.4015 (7)	0.3430 (5)	0.0507 (12)
H14A	0.2542	0.2808	0.3319	0.061*
H14B	0.2302	0.3884	0.4449	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0378 (17)	0.048 (2)	0.059 (2)	-0.0032 (15)	-0.0074 (17)	0.0003 (19)
C2	0.041 (2)	0.035 (2)	0.035 (2)	-0.0033 (17)	-0.0019 (18)	0.0031 (18)
O1	0.066 (2)	0.0485 (19)	0.074 (2)	-0.0125 (15)	-0.0256 (18)	0.0002 (17)
O2	0.0413 (15)	0.064 (2)	0.057 (2)	-0.0092 (14)	-0.0097 (14)	0.0128 (16)
C11	0.042 (2)	0.042 (2)	0.032 (2)	0.0007 (18)	0.0030 (18)	0.0051 (19)
C1	0.039 (2)	0.042 (2)	0.040 (2)	-0.0059 (17)	-0.0017 (19)	0.0059 (19)
C6	0.054 (2)	0.043 (2)	0.043 (3)	0.000 (2)	0.007 (2)	0.004 (2)
C3	0.059 (3)	0.037 (2)	0.043 (3)	-0.006 (2)	-0.005 (2)	0.004 (2)
C10	0.043 (2)	0.050 (2)	0.049 (3)	-0.003 (2)	-0.004 (2)	-0.003 (2)
C12	0.040 (2)	0.054 (3)	0.057 (3)	0.000 (2)	-0.003 (2)	-0.003 (2)
C4	0.081 (3)	0.041 (2)	0.046 (3)	-0.011 (3)	-0.004 (3)	-0.002 (2)
C5	0.074 (3)	0.043 (3)	0.049 (3)	0.006 (2)	0.006 (2)	0.001 (2)
C8	0.040 (2)	0.091 (4)	0.064 (3)	0.005 (3)	-0.001 (2)	0.001 (3)
C7	0.052 (3)	0.061 (3)	0.059 (3)	0.017 (2)	0.008 (2)	0.005 (3)
C9	0.045 (2)	0.068 (3)	0.053 (3)	-0.001 (2)	-0.006 (2)	-0.005 (3)
C13	0.047 (2)	0.054 (3)	0.069 (3)	0.000 (2)	-0.011 (2)	0.004 (3)
C14	0.044 (2)	0.067 (3)	0.042 (3)	0.004 (2)	-0.003 (2)	0.005 (2)

Geometric parameters (Å, °)

N1—C1	1.294 (4)	C12—C13	1.494 (6)
N1—C12	1.444 (5)	C12—H12A	0.9700
N1—H1A	0.8600	C12—H12B	0.9700
C2—C1	1.410 (5)	C4—C5	1.331 (6)
C2—C3	1.426 (5)	C4—H4	0.9300

C2—C11	1.447 (5)	C5—H5	0.9300
O1—C3	1.276 (5)	C8—C7	1.354 (7)
O2—C14	1.410 (5)	C8—C9	1.390 (6)
O2—C13	1.420 (5)	C8—H8	0.9300
C11—C10	1.402 (5)	C7—H7	0.9300
C11—C6	1.415 (5)	C9—H9	0.9300
C1—H1	0.9300	C13—H13A	0.9700
C6—C7	1.394 (6)	C13—H13B	0.9700
C6—C5	1.429 (6)	C14—C14 ⁱ	1.486 (8)
C3—C4	1.434 (6)	C14—H14A	0.9700
C10—C9	1.365 (5)	C14—H14B	0.9700
C10—H10	0.9300		
C1—N1—C12	126.1 (4)	C5—C4—C3	121.6 (4)
C1—N1—H1A	117.0	C5—C4—H4	119.2
C12—N1—H1A	117.0	C3—C4—H4	119.2
C1—C2—C3	118.1 (3)	C4—C5—C6	122.9 (4)
C1—C2—C11	121.4 (3)	C4—C5—H5	118.6
C3—C2—C11	120.5 (4)	C6—C5—H5	118.6
C14—O2—C13	114.1 (3)	C7—C8—C9	118.3 (4)
C10—C11—C6	116.9 (4)	C7—C8—H8	120.8
C10—C11—C2	124.0 (4)	C9—C8—H8	120.8
C6—C11—C2	119.1 (4)	C8—C7—C6	122.6 (4)
N1—C1—C2	123.6 (4)	C8—C7—H7	118.7
N1—C1—H1	118.2	C6—C7—H7	118.7
C2—C1—H1	118.2	C10—C9—C8	120.9 (5)
C7—C6—C11	119.4 (4)	C10—C9—H9	119.5
C7—C6—C5	122.3 (4)	C8—C9—H9	119.5
C11—C6—C5	118.2 (4)	O2—C13—C12	106.9 (4)
O1—C3—C2	123.1 (4)	O2—C13—H13A	110.3
O1—C3—C4	119.3 (4)	C12—C13—H13A	110.3
C2—C3—C4	117.6 (4)	O2—C13—H13B	110.3
C9—C10—C11	121.9 (4)	C12—C13—H13B	110.3
C9—C10—H10	119.1	H13A—C13—H13B	108.6
C11—C10—H10	119.1	O2—C14—C14 ⁱ	108.7 (3)
N1—C12—C13	110.7 (4)	O2—C14—H14A	109.9
N1—C12—H12A	109.5	C14 ⁱ —C14—H14A	109.9
C13—C12—H12A	109.5	O2—C14—H14B	109.9
N1—C12—H12B	109.5	C14 ⁱ —C14—H14B	109.9
C13—C12—H12B	109.5	H14A—C14—H14B	108.3
H12A—C12—H12B	108.1		
C1—C2—C11—C10	-5.6 (6)	C2—C11—C10—C9	-179.8 (4)
C3—C2—C11—C10	177.6 (4)	C1—N1—C12—C13	-140.1 (4)
C1—C2—C11—C6	175.9 (4)	O1—C3—C4—C5	178.5 (4)
C3—C2—C11—C6	-0.9 (6)	C2—C3—C4—C5	-1.1 (7)
C12—N1—C1—C2	177.0 (4)	C3—C4—C5—C6	-0.8 (7)
C3—C2—C1—N1	-3.4 (6)	C7—C6—C5—C4	-179.3 (5)

C11—C2—C1—N1	179.8 (4)	C11—C6—C5—C4	1.8 (7)
C10—C11—C6—C7	1.5 (6)	C9—C8—C7—C6	0.3 (7)
C2—C11—C6—C7	-179.9 (4)	C11—C6—C7—C8	-1.1 (7)
C10—C11—C6—C5	-179.5 (4)	C5—C6—C7—C8	180.0 (4)
C2—C11—C6—C5	-0.9 (6)	C11—C10—C9—C8	0.5 (7)
C1—C2—C3—O1	5.5 (6)	C7—C8—C9—C10	0.0 (7)
C11—C2—C3—O1	-177.7 (4)	C14—O2—C13—C12	179.6 (3)
C1—C2—C3—C4	-175.0 (4)	N1—C12—C13—O2	-61.2 (5)
C11—C2—C3—C4	1.8 (6)	C13—O2—C14—C14 ⁱ	-179.5 (4)
C6—C11—C10—C9	-1.3 (6)		

Symmetry code: (i) $-x+1/2, -y+1, z$.

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1A...O1	0.86	1.85	2.542 (5)	136
C14—H14B...O2 ⁱⁱ	0.97	2.58	3.382 (5)	141
C12—H12B...O1 ⁱⁱⁱ	0.97	2.50	3.257 (5)	134

Symmetry codes: (ii) $x, -y+1, z+1/2$; (iii) $x, y-1, z$.