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(*E*)-5-(2-Thienylmethyleneamino)-quinolin-8-ol

Stéphane Dufresne, Alex N. Bourque and W. G. Skene*

Department of Chemistry, University of Montreal, CP 6128, succ. Centre-ville, Montréal, Québec, Canada H3C 3J7
Correspondence e-mail: w.skene@umontreal.ca

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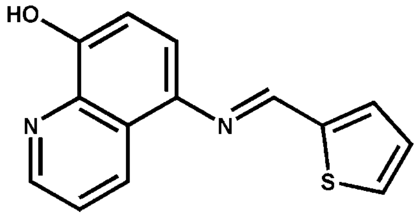
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.054; wR factor = 0.139; data-to-parameter ratio = 14.5.

Two molecules of the title compound, $C_{14}H_{10}N_2OS$, are hydrogen bonded about a center of inversion. In the molecule, the two aromatic rings are twisted by 37.27 (5)° with respect to one another. The azomethine bond is in the *E* configuration.

Related literature

For information about the utility of azomethines, see: Dufresne *et al.* (2006); Skene & Dufresne (2006). For related structures, see: Chen *et al.* (1999). For an analog with an aryl ring in place of the thienyl ring, see Manecke *et al.* (1972).



Experimental

Crystal data

$C_{14}H_{10}N_2OS$	V = 1191.07 (9) Å^3
M_r = 254.30	Z = 4
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
a = 7.6798 (4) Å	μ = 2.31 mm^{-1}
b = 9.8592 (4) Å	T = 150 (2) K
c = 15.7512 (7) Å	0.07 × 0.05 × 0.05 mm
β = 92.926 (2)°	

Data collection

Bruker SMART 6K diffractometer	31904 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2377 independent reflections
T_{min} = 0.855, T_{max} = 0.893	2152 reflections with $I > 2\sigma(I)$
	R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054	164 parameters
$wR(F^2)$ = 0.139	H-atom parameters constrained
S = 1.11	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
2377 reflections	$\Delta\rho_{min}$ = −0.62 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.84	2.27	2.927 (2)	136

Symmetry code: (i) $-x, -y, -z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *UdMX* (Marris, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2406).

References

Bruker (1997). *SHELXTL* Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, X., Zhu, X., Vittal, J. J. & You, X. (1999). *Acta Cryst.* C**55**, IUC9900095.
Dufresne, S., Bourgeaux, M. & Skene, W. G. (2006). *Acta Cryst.* E**62**, o5602–o5604.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Manecke, G., Wille, W. E. & Kossmehl, G. (1972). *Macromol. Chem.* **160**, 111–126.
Marris, T. (2004). *UdMX*. Université de Montréal, Canada.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Skene, W. G. & Dufresne, S. (2006). *Acta Cryst.* E**62**, o1116–o1117.

supporting information

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(*E*)-5-(2-Thienylmethyleneamino)quinolin-8-ol

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S1. Comment

Compound (I) was prepared as a new ligand for metal-ligand charge transfer complexes. The structure of (I) consists of quinolin-2-ol covalently linked to a thiophene unit by an azomethine bond with more stable E isomer being observed. The crystal structure has a P21/c symmetry as seen in figure 2. No solvent molecules or counter-ions were found in the crystal structure.

The bond lengths and angles of the quinolin-2-ol moiety are within 0.013 Å and 1°, respectively, to comparable structures (Chen *et al.*, 1999). The bond lengths of the azomethine bond for C5—N2, N2—C10 and C10—C11 are 1.421 (2), 1.276 (2) and 1.446 (2) Å, respectively. The bond lengths are comparable to an all thiophene azomethine analogue (Dufresne *et al.*, 2006) whose analogues bond lengths are 1.388 (3), 1.272 (3) and 1.441 (4) Å, respectively.

The mean planes of the two aryl moieties are twisted by 37.27 (5)° from the azomethine bond to which they are connected. This angle is smaller, *i.e.* 65°, (Manecke *et al.*, 1972) than its homoaryl analogue. Steric hindrance between H6 and H10 is responsible for the twist between the mean planes similar to a thiophene azomethine, whose aryl mean planes are twisted by 33° Skene *et al.*, 2006).

Hydrogen bonding takes place between two quinolin-8-ol moieties to form a supramolecular dimer. Figure 2 shows the two symmetry related hydrogen bonds between O1—H1···N1î^ and O1î^-H1î^···N1 that form the dimer. The length and the angle of this bond are 2.927 (2) Å and 136°, respectively. The two quinolin-2-ol involved in the hydrogen bonding are shifted by 0.593 Å.

S2. Experimental

The title compound was synthesized by means of an acid catalyzed condensation of 5-amino-8-hydroxyquinoline with 2-thiophenecarboxaldehyde in ethanol with catalytic trifluoroacetic acid. The reaction was held at reflux for 20 h with stirring, cooled to room temperature and the volume reduced. Ice-cold distilled water was added to this solution causing a yellow solid to precipitate. The yellow solid was collected, washed with water and then dried under reduced pressure overnight. Crystals were obtained by slow evaporation of a concentrated solution of (1) in acetone.

S3. Refinement

H atoms were placed in calculated positions (C—H = 0.95 Å) and included in the refinement in the riding-model approximation, with $U_{\rm iso}$(H) = 1.2 $U_{\rm eq}$(C). The hydrogen on the hydroxyl group was placed in calculated position (O—H = 0.84 Å, C—O—H = 109.5°) and included in the refinement in the riding-model approximation with $U_{\rm iso}$(H) = 1.5 $U_{\rm eq}$(O).

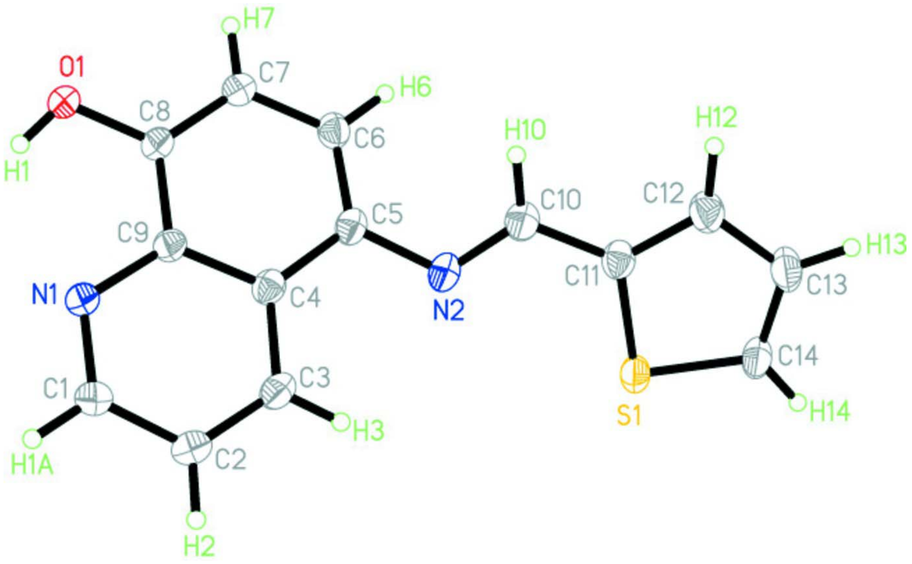

Figure 1

ORTEP representation of (I) with the numbering scheme adopted (Farrugia 1997). Ellipsoids drawn at 30% probability level.

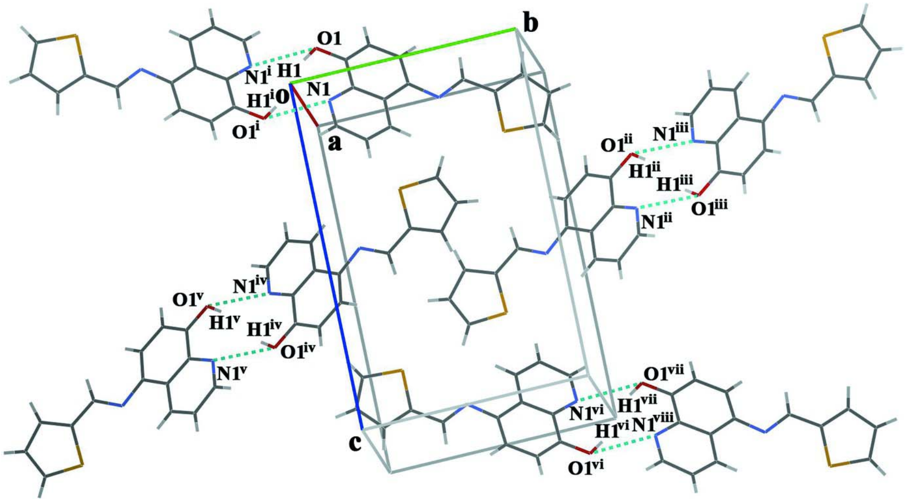

Figure 2

The lattice structure of (I) showing hydrogen bonding. [Symmetry codes: (i) -*x*, -*y*, -*z*; (ii)

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Crystal data

$C_{14}H_{10}N_2OS$
$M_r = 254.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.6798$ (4) Å
$b = 9.8592$ (4) Å
$c = 15.7512$ (7) Å
$\beta = 92.926$ (2)°

$V = 1191.07$ (9) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.418$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 7426 reflections
$\theta = 5.3$–72.9°
$\mu = 2.31$ mm^{-1}

T = 150 K
Block, yellow
0.07 × 0.05 × 0.05 mm

Data collection

Bruker SMART 6000 diffractometer
Radiation source: Rotating Anode
Montel 200 optics monochromator
Detector resolution: 5.5 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.855, $T_{\max}$ = 0.893
31904 measured reflections
2377 independent reflections
2152 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
$\theta_{\max}$ = 73.2°, $\theta_{\min}$ = 5.3°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.139$
S = 1.11
2377 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0968P)^2 + 0.157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.42 e Å^{-3}
$\Delta\rho_{\min}$ = −0.62 e Å^{-3}

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.43366 (6)	0.82399 (4)	0.21499 (3)	0.0464 (2)
O1	0.10263 (18)	0.13156 (13)	−0.09352 (7)	0.0446 (3)
H1	0.0556	0.0746	−0.0623	0.067*
N1	0.0099 (2)	0.14942 (15)	0.07194 (9)	0.0394 (3)
N2	0.29207 (18)	0.59227 (14)	0.09825 (9)	0.0406 (3)
C1	−0.0336 (3)	0.16030 (18)	0.15190 (11)	0.0449 (4)
H1A	−0.0985	0.0886	0.1752	0.054*
C2	0.0107 (3)	0.2718 (2)	0.20431 (11)	0.0460 (4)
H2	−0.0225	0.2741	0.2616	0.055*
C3	0.1018 (2)	0.37649 (19)	0.17187 (11)	0.0419 (4)
H3	0.1327	0.4524	0.2066	0.050*
C4	0.1505 (2)	0.37229 (17)	0.08639 (10)	0.0361 (4)
C5	0.2413 (2)	0.47908 (17)	0.04695 (11)	0.0373 (4)
C6	0.2810 (2)	0.46462 (17)	−0.03706 (11)	0.0393 (4)

H6	0.3422	0.5352	−0.0637	0.047*
C7	0.2332 (2)	0.34810 (17)	−0.08420 (11)	0.0397 (4)
H7	0.2618	0.3414	−0.1420	0.048*
C8	0.1456 (2)	0.24404 (17)	−0.04735 (10)	0.0367 (4)
C9	0.1013 (2)	0.25436 (16)	0.03912 (9)	0.0354 (4)
C10	0.3002 (2)	0.70894 (18)	0.06355 (12)	0.0424 (4)
H10	0.2655	0.7176	0.0050	0.051*
C11	0.3600 (2)	0.82854 (16)	0.10947 (12)	0.0420 (4)
C12	0.3671 (3)	0.95730 (19)	0.07858 (13)	0.0529 (5)
H12	0.3312	0.9809	0.0219	0.063*
C13	0.4333 (3)	1.0526 (2)	0.13929 (14)	0.0554 (5)
H13	0.4470	1.1465	0.1277	0.066*
C14	0.4751 (3)	0.99469 (18)	0.21584 (12)	0.0474 (4)
H14	0.5216	1.0430	0.2640	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0614 (3)	0.0305 (3)	0.0465 (3)	0.00053 (17)	−0.0045 (2)	−0.00223 (15)
O1	0.0632 (8)	0.0393 (7)	0.0312 (6)	−0.0135 (6)	0.0022 (5)	−0.0040 (5)
N1	0.0495 (8)	0.0376 (7)	0.0305 (7)	−0.0066 (6)	−0.0034 (6)	0.0002 (5)
N2	0.0402 (7)	0.0350 (8)	0.0461 (8)	−0.0009 (6)	−0.0026 (6)	−0.0081 (6)
C1	0.0552 (10)	0.0457 (10)	0.0337 (9)	−0.0069 (8)	0.0013 (7)	0.0017 (7)
C2	0.0556 (10)	0.0502 (10)	0.0321 (8)	−0.0020 (8)	0.0006 (7)	−0.0043 (7)
C3	0.0462 (9)	0.0418 (9)	0.0371 (8)	0.0000 (7)	−0.0033 (7)	−0.0080 (7)
C4	0.0356 (8)	0.0356 (8)	0.0365 (8)	0.0022 (7)	−0.0050 (6)	−0.0033 (6)
C5	0.0358 (8)	0.0332 (8)	0.0424 (8)	0.0004 (6)	−0.0040 (6)	−0.0045 (6)
C6	0.0392 (8)	0.0358 (9)	0.0427 (9)	−0.0050 (6)	0.0006 (6)	0.0001 (6)
C7	0.0443 (9)	0.0401 (9)	0.0347 (8)	−0.0039 (7)	0.0011 (7)	−0.0018 (7)
C8	0.0406 (8)	0.0365 (8)	0.0326 (8)	−0.0032 (7)	−0.0034 (6)	−0.0032 (6)
C9	0.0371 (8)	0.0361 (8)	0.0325 (8)	−0.0022 (6)	−0.0046 (6)	−0.0004 (6)
C10	0.0407 (9)	0.0380 (9)	0.0477 (10)	0.0032 (7)	−0.0068 (7)	−0.0028 (7)
C11	0.0409 (9)	0.0358 (9)	0.0485 (10)	0.0036 (7)	−0.0051 (7)	−0.0027 (7)
C12	0.0643 (12)	0.0380 (10)	0.0547 (11)	0.0023 (9)	−0.0136 (9)	0.0024 (8)
C13	0.0691 (13)	0.0313 (9)	0.0639 (12)	0.0002 (8)	−0.0130 (10)	0.0005 (8)
C14	0.0541 (10)	0.0317 (9)	0.0555 (11)	0.0014 (8)	−0.0064 (8)	−0.0072 (7)

Geometric parameters (Å, º)

S1—C14	1.7127 (18)	C4—C5	1.423 (2)
S1—C11	1.7289 (19)	C5—C6	1.380 (2)
O1—C8	1.358 (2)	C6—C7	1.406 (2)
O1—H1	0.8400	C6—H6	0.9500
N1—C1	1.324 (2)	C7—C8	1.372 (2)
N1—C9	1.367 (2)	C7—H7	0.9500
N2—C10	1.276 (2)	C8—C9	1.424 (2)
N2—C5	1.421 (2)	C10—C11	1.446 (2)
C1—C2	1.406 (3)	C10—H10	0.9500

C1—H1A	0.9500	C11—C12	1.362 (3)
C2—C3	1.361 (3)	C12—C13	1.417 (3)
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.416 (2)	C13—C14	1.358 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C9	1.421 (2)	C14—H14	0.9500

C14—S1—C11	91.92 (9)	C8—C7—H7	119.7
C8—O1—H1	109.5	C6—C7—H7	119.7
C1—N1—C9	117.25 (14)	O1—C8—C7	119.70 (14)
C10—N2—C5	118.84 (15)	O1—C8—C9	120.47 (14)
N1—C1—C2	123.84 (17)	C7—C8—C9	119.83 (15)
N1—C1—H1A	118.1	N1—C9—C4	123.30 (14)
C2—C1—H1A	118.1	N1—C9—C8	117.39 (14)
C3—C2—C1	119.08 (16)	C4—C9—C8	119.30 (15)
C3—C2—H2	120.5	N2—C10—C11	122.82 (17)
C1—C2—H2	120.5	N2—C10—H10	118.6
C2—C3—C4	120.04 (16)	C11—C10—H10	118.6
C2—C3—H3	120.0	C12—C11—C10	126.81 (18)
C4—C3—H3	120.0	C12—C11—S1	110.52 (14)
C3—C4—C9	116.48 (15)	C10—C11—S1	122.67 (13)
C3—C4—C5	123.54 (15)	C11—C12—C13	113.39 (18)
C9—C4—C5	119.98 (15)	C11—C12—H12	123.3
C6—C5—N2	123.97 (15)	C13—C12—H12	123.3
C6—C5—C4	118.63 (15)	C14—C13—C12	112.38 (18)
N2—C5—C4	117.35 (15)	C14—C13—H13	123.8
C5—C6—C7	121.74 (16)	C12—C13—H13	123.8
C5—C6—H6	119.1	C13—C14—S1	111.78 (14)
C7—C6—H6	119.1	C13—C14—H14	124.1
C8—C7—C6	120.53 (16)	S1—C14—H14	124.1

C9—N1—C1—C2	−0.8 (3)	C3—C4—C9—N1	0.8 (2)
N1—C1—C2—C3	0.8 (3)	C5—C4—C9—N1	−178.15 (15)
C1—C2—C3—C4	0.1 (3)	C3—C4—C9—C8	179.70 (15)
C2—C3—C4—C9	−0.8 (2)	C5—C4—C9—C8	0.7 (2)
C2—C3—C4—C5	178.12 (16)	O1—C8—C9—N1	−2.1 (2)
C10—N2—C5—C6	33.6 (2)	C7—C8—C9—N1	178.23 (15)
C10—N2—C5—C4	−149.15 (16)	O1—C8—C9—C4	178.95 (14)
C3—C4—C5—C6	−179.47 (15)	C7—C8—C9—C4	−0.7 (2)
C9—C4—C5—C6	−0.6 (2)	C5—N2—C10—C11	−176.40 (16)
C3—C4—C5—N2	3.2 (2)	N2—C10—C11—C12	−177.5 (2)
C9—C4—C5—N2	−177.93 (14)	N2—C10—C11—S1	2.4 (3)
N2—C5—C6—C7	177.58 (15)	C14—S1—C11—C12	−0.57 (17)
C4—C5—C6—C7	0.4 (2)	C14—S1—C11—C10	179.52 (17)
C5—C6—C7—C8	−0.4 (3)	C10—C11—C12—C13	−179.54 (19)
C6—C7—C8—O1	−179.12 (15)	S1—C11—C12—C13	0.6 (2)
C6—C7—C8—C9	0.5 (3)	C11—C12—C13—C14	−0.2 (3)
C1—N1—C9—C4	0.0 (2)	C12—C13—C14—S1	−0.2 (2)

C1—N1—C9—C8	−178.93 (16)	C11—S1—C14—C13	0.45 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.84	2.27	2.927 (2)	136

Symmetry code: (i) $-x, -y, -z$.