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5-[(2-Ethoxyphenyl)diazenyl]quinolin-8-ol

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Abstract

The title compound, $C_{17}H_{15}N_3O_2$, exists as the *trans* isomer. The dihedral angle between the phenyl and the quinoline rings is 1.9˜1)% and the N═N bond distance is 1.249 (3) Å. In the solid, intermolecular O—H···N hydrogen bonds [2.826 (3) Å, 138°] link the molecules about inversion centres to form dimers.

Comment

5-(Arylazo)-8-quinolinol is a versatile ligand which forms complexes with metals or organometals (Basu Baul *et al.*, 1984). It was widely used as analytical reagents for qualitative detection of metal ions. The coordination centers can be the nitrogen and oxygen atoms in the quinolinol moiety or the oxygen atoms of the carboxylic group in the diazo moiety and the nitrogen atoms of the azo-group or combinations of both (Basu Baul *et al.*, 1995). In this paper we report the crystal structure of 5-(2'-ethoxyphenylazo)-8-quinolinol. The title compound exists as stable *trans*-isomeric form. The N═N bond distance[1.249 (3) Å] is similar to that in *trans*-azobenzene[1.247 Å] (Mostad & Romming, 1971). The dihedral angle between the phenyl and the quinoline rings is 1.9 (1)°. The bond distances of C9—N2[1.425 (3) Å] and C10—N3[1.416 (3) Å] are some shorter than that in *trans*-azobenzene [1.434 Å]. In the solid, there is an intermolecular hydrogen bonding O1–H1···N1A(A:1 − *x*,1 − *y*,1 − *z*)[2.826 (3) Å, 137.87°] which links the molecules to form dimer. This is very different from the suggestion that 5-(arylazo)-8-quinolinol possessed an intramolecular hydrogen bonding established between the phenolic –OH and nitrogen atom in the quinolinol moiety (Basu Baul *et al.*, 1995).

Experimental

The title compound was prepared by the usual diazotization reaction (Aiello *et al.*, 1997). Single crystals suitable for X-ray crystallography were obtained by recrystallization from CH_3CN.

Refinement

The structure was solved by direct methods and refined by full-matrix least-squares techniques. The hydroxy H atom(H1) was refined isotropically and that all H atoms bonded to C were refined as riding atoms.

Computing details

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SHELXTL* (Sheldrick, 1997a); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997c); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

CIF access

(yxz28)

Crystal data

$C_{17}H_{15}N_3O_2$	$V = 2907.08\ (8)$ Å^3
$M_r = 293.32$	$Z = 8$
Orthorhombic, *Pbca*	Mo $K\alpha$
$a = 15.6977\ (1)$ Å	$\mu = 0.09$ mm^{-1}
$b = 8.7328\ (1)$ Å	$T = 293\ (2)$ K
$c = 21.2064\ (5)$ Å	0.28 × 0.23 × 0.10 mm

Data collection

CCD area detector diffractometer	2557 independent reflections
Absorption correction: none	1992 reflections with $I > 2\sigma(I)$
12831 measured reflections	$R_{int} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$	205 parameters
$wR(F^2) = 0.136$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.23$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2557 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °)

O1—C6	1.348 (3)	N2—N3	1.249 (3)
N1—C4	1.321 (3)	N2—C9	1.425 (3)
N1—C5	1.363 (3)	N3—C10	1.416 (3)
N3—N2—C9	113.3 (2)	N2—N3—C10	115.2 (2)

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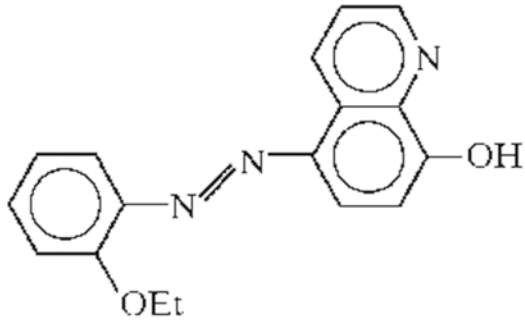
Scheme 1



supplementary materials

(yxz28)

Crystal data

$C_{17}H_{15}N_3O_2$	$D_x = 1.340$ Mg m^{-3}
$M_r = 293.32$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 4827 reflections
$a = 15.6977$ (1) Å	$\theta = 2.6$–25.0º
$b = 8.7328$ (1) Å	$\mu = 0.09$ mm^{-1}
$c = 21.2064$ (5) Å	$T = 293$ (2) K
$V = 2907.08$ (8) Å^3	Plates, orange
$Z = 8$	0.28 × 0.23 × 0.10 mm
$F_{000} = 1232$	

Data collection

CCD area detector diffractometer	1992 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.046$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 293(2)$ K	$\theta_{min} = 2.6$º
φ and θ scans	$h = -18\rightarrow18$
Absorption correction: none	$k = -9\rightarrow10$
12831 measured reflections	$l = -20\rightarrow25$
2557 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 1.4241P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.136$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.23$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2557 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
205 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0024 (5)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.40334 (11)	0.6273 (2)	0.45749 (11)	0.0581 (5)
H1	0.436 (2)	0.562 (4)	0.4771 (16)	0.099 (13)*
O2	0.43162 (13)	1.4692 (2)	0.30354 (11)	0.0771 (7)
N1	0.57027 (12)	0.6683 (2)	0.49193 (9)	0.0428 (5)
N2	0.56917 (14)	1.1666 (2)	0.38903 (10)	0.0492 (6)
N3	0.52266 (14)	1.2648 (2)	0.36356 (10)	0.0505 (6)
C1	0.57163 (15)	0.9194 (3)	0.44039 (11)	0.0392 (6)
C2	0.65676 (15)	0.9377 (3)	0.45959 (12)	0.0477 (6)
H2	0.6862	1.0266	0.4492	0.057*
C3	0.69602 (16)	0.8257 (3)	0.49321 (13)	0.0536 (7)
H3	0.7524	0.8369	0.5060	0.064*
C4	0.65028 (16)	0.6933 (3)	0.50834 (13)	0.0517 (7)
H4	0.6780	0.6178	0.5315	0.062*
C5	0.53107 (14)	0.7808 (3)	0.45810 (11)	0.0384 (6)
C6	0.44443 (15)	0.7571 (3)	0.44134 (12)	0.0441 (6)
C7	0.40156 (16)	0.8659 (3)	0.40801 (13)	0.0511 (7)
H7	0.3449	0.8495	0.3972	0.061*
C8	0.44141 (16)	1.0014 (3)	0.38988 (12)	0.0493 (7)
H8	0.4110	1.0737	0.3669	0.059*
C9	0.52484 (15)	1.0300 (3)	0.40544 (11)	0.0425 (6)
C10	0.56426 (17)	1.4021 (3)	0.34592 (12)	0.0459 (6)
C11	0.64858 (18)	1.4368 (3)	0.35909 (13)	0.0561 (7)
H11	0.6823	1.3657	0.3802	0.067*
C12	0.68306 (19)	1.5750 (3)	0.34140 (14)	0.0645 (8)
H12	0.7394	1.5981	0.3511	0.077*
C13	0.6334 (2)	1.6790 (3)	0.30910 (14)	0.0655 (8)
H13	0.6568	1.7722	0.2969	0.079*
C14	0.55003 (19)	1.6472 (3)	0.29465 (13)	0.0593 (8)
H14	0.5177	1.7177	0.2721	0.071*
C15	0.51418 (17)	1.5096 (3)	0.31382 (13)	0.0523 (7)
C16	0.37086 (19)	1.5810 (4)	0.28516 (15)	0.0690 (9)
H16A	0.3686	1.5887	0.2396	0.083*
H16B	0.3863	1.6804	0.3021	0.083*

C17	0.2865 (2)	1.5319 (4)	0.31044 (17)	0.0842 (10)
H17A	0.2437	1.6045	0.2981	0.126*
H17B	0.2891	1.5268	0.3556	0.126*
H17C	0.2723	1.4328	0.2939	0.126*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0400 (10)	0.0465 (11)	0.0878 (15)	−0.0054 (9)	−0.0064 (10)	0.0152 (10)
O2	0.0698 (14)	0.0460 (11)	0.1154 (18)	0.0013 (10)	−0.0282 (13)	0.0125 (12)
N1	0.0378 (11)	0.0378 (11)	0.0530 (12)	0.0007 (9)	0.0003 (10)	0.0034 (10)
N2	0.0518 (13)	0.0417 (12)	0.0540 (13)	0.0028 (11)	0.0011 (11)	0.0058 (10)
N3	0.0568 (13)	0.0400 (12)	0.0546 (13)	0.0001 (11)	−0.0022 (11)	0.0036 (10)
C1	0.0410 (13)	0.0357 (13)	0.0409 (13)	−0.0012 (11)	0.0037 (11)	−0.0010 (10)
C2	0.0401 (14)	0.0422 (14)	0.0610 (17)	−0.0074 (11)	0.0009 (13)	0.0018 (13)
C3	0.0401 (14)	0.0452 (15)	0.0755 (19)	−0.0016 (13)	−0.0073 (14)	0.0062 (14)
C4	0.0418 (15)	0.0436 (15)	0.0697 (19)	0.0036 (12)	−0.0069 (13)	0.0081 (13)
C5	0.0370 (13)	0.0373 (13)	0.0410 (13)	0.0029 (10)	0.0031 (11)	−0.0025 (11)
C6	0.0387 (13)	0.0396 (14)	0.0541 (16)	−0.0021 (12)	0.0021 (12)	0.0014 (12)
C7	0.0377 (14)	0.0510 (16)	0.0647 (17)	−0.0001 (12)	−0.0092 (13)	0.0028 (14)
C8	0.0488 (16)	0.0433 (15)	0.0558 (17)	0.0040 (12)	−0.0053 (12)	0.0062 (12)
C9	0.0466 (14)	0.0365 (13)	0.0445 (14)	0.0003 (11)	0.0015 (12)	0.0014 (11)
C10	0.0548 (16)	0.0391 (14)	0.0438 (14)	0.0001 (12)	0.0029 (12)	0.0026 (12)
C11	0.0581 (18)	0.0487 (16)	0.0614 (18)	0.0011 (13)	0.0006 (14)	0.0095 (14)
C12	0.0595 (18)	0.0590 (18)	0.075 (2)	−0.0115 (15)	0.0033 (16)	0.0093 (16)
C13	0.081 (2)	0.0489 (16)	0.0662 (19)	−0.0091 (16)	0.0141 (17)	0.0109 (15)
C14	0.074 (2)	0.0444 (16)	0.0591 (18)	0.0038 (14)	−0.0009 (15)	0.0109 (13)
C15	0.0622 (18)	0.0428 (15)	0.0519 (16)	0.0001 (13)	−0.0034 (14)	−0.0021 (12)
C16	0.076 (2)	0.0610 (18)	0.070 (2)	0.0109 (17)	−0.0143 (17)	0.0076 (16)
C17	0.076 (2)	0.096 (3)	0.081 (2)	0.010 (2)	−0.0014 (19)	0.009 (2)

Geometric parameters (Å, °)

O1—C6	1.348 (3)	C3—C4	1.399 (3)
O2—C15	1.361 (3)	C5—C6	1.421 (3)
O2—C16	1.419 (3)	C6—C7	1.362 (3)
N1—C4	1.321 (3)	C7—C8	1.392 (3)
N1—C5	1.363 (3)	C8—C9	1.373 (3)
N2—N3	1.249 (3)	C10—C11	1.386 (4)
N2—C9	1.425 (3)	C10—C15	1.401 (3)
N3—C10	1.416 (3)	C11—C12	1.375 (4)
C1—C2	1.406 (3)	C12—C13	1.379 (4)
C1—C5	1.419 (3)	C13—C14	1.372 (4)
C1—C9	1.421 (3)	C14—C15	1.388 (4)
C2—C3	1.358 (3)	C16—C17	1.492 (4)
C15—O2—C16	120.3 (2)	C6—C7—C8	120.9 (2)
C4—N1—C5	116.6 (2)	C9—C8—C7	121.1 (2)
N3—N2—C9	113.3 (2)	C8—C9—C1	119.7 (2)

N2—N3—C10	115.2 (2)	C8—C9—N2	124.0 (2)
C2—C1—C5	116.5 (2)	C1—C9—N2	116.3 (2)
C2—C1—C9	124.4 (2)	C11—C10—C15	119.2 (2)
C5—C1—C9	119.1 (2)	C11—C10—N3	124.9 (2)
C3—C2—C1	120.1 (2)	C15—C10—N3	115.9 (2)
C2—C3—C4	118.9 (2)	C12—C11—C10	120.8 (3)
N1—C4—C3	124.3 (2)	C11—C12—C13	119.4 (3)
N1—C5—C1	123.5 (2)	C14—C13—C12	121.1 (3)
N1—C5—C6	117.3 (2)	C13—C14—C15	119.7 (3)
C1—C5—C6	119.2 (2)	O2—C15—C14	124.3 (3)
O1—C6—C7	118.8 (2)	O2—C15—C10	116.0 (2)
O1—C6—C5	121.1 (2)	C14—C15—C10	119.7 (3)
C7—C6—C5	120.1 (2)	O2—C16—C17	107.5 (3)
C9—N2—N3—C10	−179.5 (2)	C5—C1—C9—C8	0.5 (3)
C5—C1—C2—C3	0.1 (4)	C2—C1—C9—N2	0.5 (4)
C9—C1—C2—C3	179.6 (2)	C5—C1—C9—N2	−179.9 (2)
C1—C2—C3—C4	−0.2 (4)	N3—N2—C9—C8	5.8 (4)
C5—N1—C4—C3	−0.1 (4)	N3—N2—C9—C1	−173.8 (2)
C2—C3—C4—N1	0.2 (4)	N2—N3—C10—C11	−5.0 (4)
C4—N1—C5—C1	0.0 (3)	N2—N3—C10—C15	176.2 (2)
C4—N1—C5—C6	−178.6 (2)	C15—C10—C11—C12	0.4 (4)
C2—C1—C5—N1	0.0 (3)	N3—C10—C11—C12	−178.4 (3)
C9—C1—C5—N1	−179.6 (2)	C10—C11—C12—C13	−1.2 (4)
C2—C1—C5—C6	178.6 (2)	C11—C12—C13—C14	0.4 (5)
C9—C1—C5—C6	−1.0 (3)	C12—C13—C14—C15	1.3 (5)
N1—C5—C6—O1	−1.0 (4)	C16—O2—C15—C14	−14.9 (4)
C1—C5—C6—O1	−179.7 (2)	C16—O2—C15—C10	165.2 (3)
N1—C5—C6—C7	179.4 (2)	C13—C14—C15—O2	178.0 (3)
C1—C5—C6—C7	0.8 (4)	C13—C14—C15—C10	−2.2 (4)
O1—C6—C7—C8	−179.6 (2)	C11—C10—C15—O2	−178.8 (2)
C5—C6—C7—C8	0.0 (4)	N3—C10—C15—O2	0.1 (3)
C6—C7—C8—C9	−0.5 (4)	C11—C10—C15—C14	1.3 (4)
C7—C8—C9—C1	0.3 (4)	N3—C10—C15—C14	−179.8 (2)
C7—C8—C9—N2	−179.3 (2)	C15—O2—C16—C17	−149.7 (3)
C2—C1—C9—C8	−179.0 (2)		