

1-[(*E*)-(2-Phenoxyanilino)methylene]-naphthalen-2(1*H*)-oneErsin Temel,<sup>a</sup> Erbil Ağar<sup>b</sup> and Orhan Büyükgüngör<sup>a\*</sup><sup>a</sup>Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, TR-55139 Samsun, Turkey, and <sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, TR-55139 Samsun, Turkey

Correspondence e-mail: orhanb@omu.edu.tr

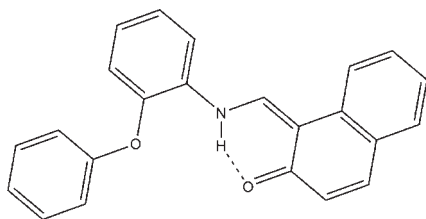
Received 9 April 2010; accepted 14 April 2010

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å;  $R$  factor = 0.115;  $wR$  factor = 0.331; data-to-parameter ratio = 10.9.

The molecule of the title compound,  $\text{C}_{23}\text{H}_{17}\text{NO}_2$ , a Schiff base derived from 2-hydroxy-1-naphthaldehyde, crystallizes in the keto–amine tautomeric form. The dihedral angle between the aniline and hydroxybenzene rings is  $77.41$  ( $17$ ) $^\circ$ , whereas the planes of the naphthaldehyde and fused aniline benzene rings are nearly coplanar, making a dihedral angle of  $8.29$  ( $15$ ) $^\circ$ . Intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonding, a characteristic hydrogen bond for Schiff bases, helps to stabilize the molecular structure. Weak intermolecular  $\text{C}-\text{H}\cdots\pi$  interactions are present in the crystal structure.

## Related literature

For Schiff bases, see: Caligaris *et al.* (1972); Caligaris & Randaccio *et al.* (1987); Salman *et al.* (1990); Popović *et al.* (2001); Garnovskii *et al.* (1993); Pyrz *et al.* (1985); Hadjoudis *et al.* (1987). For the HOMA (harmonic oscillator model of aromaticity) index, see: Krygowski *et al.* (1993). For similar structures, see: Özek *et al.* (2004); Takano *et al.* (2009).



## Experimental

## Crystal data

$\text{C}_{23}\text{H}_{17}\text{NO}_2$   
 $M_r = 339.38$   
 Monoclinic,  $C2/c$   
 $a = 14.6428$  (11) Å  
 $b = 5.6297$  (3) Å  
 $c = 42.602$  (3) Å  
 $\beta = 101.175$  (6) $^\circ$

$V = 3445.3$  (4) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.40 \times 0.34 \times 0.23$  mm

## Data collection

Stoe IPDSII diffractometer  
 Absorption correction: integration  
 ( $X$ -RED; Stoe & Cie, 2002)  
 $T_{\min} = 0.960$ ,  $T_{\max} = 0.996$   
 11832 measured reflections

2565 independent reflections  
 1522 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.212$   
 $\theta_{\max} = 23.6^\circ$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.115$   
 $wR(F^2) = 0.331$   
 $S = 1.06$   
 2565 reflections

236 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.39$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å,  $^\circ$ ).

Cg1 and Cg2 are the centroids of the C1–C6 and C18–C23 rings, respectively.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}$	0.86	1.87	2.561 (6)	136
$\text{C11}-\text{H11}\cdots\text{Cg1}^{\text{i}}$	0.93	2.87	3.664 (7)	144
$\text{C19}-\text{H19}\cdots\text{Cg2}^{\text{ii}}$	0.93	2.90	3.674 (7)	142

Symmetry codes: (i)  $x + \frac{1}{2}, y - \frac{1}{2}, z$ ; (ii)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$ .

Data collection:  $X$ -AREA (Stoe & Cie, 2002); cell refinement:  $X$ -AREA; data reduction:  $X$ -RED32 (Stoe & Cie, 2002); program(s) used to solve structure:  $SHELXS97$  (Sheldrick, 2008); program(s) used to refine structure:  $SHELXL97$  (Sheldrick, 2008); molecular graphics:  $ORTEP-3$  for Windows (Farrugia, 1997); software used to prepare material for publication:  $WinGX$  (Farrugia, 1999).

The authors wish to acknowledge the Faculty of Arts and Sciences of Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDSII diffractometer (purchased under grant No. F279 of the University Research Grant of Ondokuz Mayıs University).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2256).

## References

- Caligaris, M., Nardin, G. & Randaccio, L. (1972). *Coord. Chem. Rev.* **7**, 385–403.
- Caligaris, M. & Randaccio, L. (1987). *Comprehensive Coordination Chemistry*, Vol. 2, edited by G. Wilkinson, R. D. Gillard & J. A. McCleverty, pp. 715–738. Oxford: Pergamon Press.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Garnovskii, A. D., Nivorozhkin, A. L. & Minkin, V. I. (1993). *Coord. Chem. Rev.* **126**, 1–69.
- Hadjoudis, E., Vittorakis, M. & Moustakali-Mavridis, I. (1987). *Tetrahedron*, **43**, 1345–1360.
- Krygowski, T. M. (1993). *J. Chem. Inf. Comput. Sci.* **33**, 70–78.
- Özek, A., Yüce, S., Albayrak, Ç., Odabaşoğlu, M. & Büyükgüngör, O. (2004). *Acta Cryst.* **E60**, o1162–o1164.
- Popović, Z., Roje, V., Pavlović, G., Matković-Čalogović, D. & Giester, G. (2001). *J. Mol. Struct.* **597**, 39–47.
- Pyrz, J. W., Roe, A. L., Stern, L. J. & Que, L. Jr (1985). *J. Am. Chem. Soc.* **107**, 614–620.
- Salman, S. R., Shawkat, S. H. & Al-Obaidi, G. M. (1990). *Can. J. Spectrosc.* **35**, 25–27.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stoe & Cie (2002).  $X$ -RED32 and  $X$ -AREA. Stoe & Cie, Darmstadt, Germany.
- Takano, K., Takahashi, M., Fukushima, T. & Shibahara, T. (2009). *Acta Cryst.* **E65**, o3127.

**supplementary materials**

*Acta Cryst.* (2010). E66, o1131 [ doi:10.1107/S1600536810013851 ]

## 1-[(*E*)-(2-Phenoxyanilino)methylene]naphthalen-2(1*H*)-one

E. Temel, E. Agar and O. Büyükgüngör

### Comment

2-Hydroxy-Schiff bases are formed by reactions of salicylaldehyde and 2-hydroxy-1-naphthaldehyde with various amines (Caligaris *et al.*, 1972). In this study, a Schiff base derivatived from 2-hydroxy-1-naphthaldehyde was examined. In contrast to salicylaldimine derivatives, the Schiff bases of 2-hydroxy-1-naphthaldehyde have been rarely investigated (Salman *et al.*, 1990; Popović *et al.*, 2001). Schiff base ligands play a vital role in coordination chemistry due to their metal binding ability (Garnovskii *et al.*, 1993). In addition, Schiff bases and their metal complexes have wide applications in biological systems (Pyrz *et al.*, 1985).

The structure of *o*-hydroxy aromatic Schiff base has drawn attention due to their keto-enol tautomerism in recent years (Hadjoudis *et al.*, 1987). As being characteristic feature of Schiff bases there are two alternative intra-molecular hydrogen bonds depending on the type of tautomer. The structure with intra-molecular N—H···O hydrogen bond is called keto tautomer. The enol tautomer is, on the other hand, a structure involving O—H···N type hydrogen bond (Caligaris & Randaccio *et al.*, 1987). Details of hydrogen bond geometry are given in Table 1.

The proton transfer responsible for the tautomerization requires a small amount of energy which can be obtained by temperature change or light (Caligaris & Randaccio *et al.*, 1987). The proton transfer reaction causes the bond distances to deviate from the ideal value 1.338 Å, which leads to a decrease in aromaticity of the ring. In order to investigate deformation in  $\pi$ -electron delocalization of aromatic rings, HOMA (Harmonic Oscillator Model of Aromaticity) index is a useful tool. The HOMA index is equal to unity for purely aromatic systems and zero for non-aromatic systems (Krygowski *et al.*, 1993). The HOMA index of the naphthalene ring of (I) was calculated as 0.670, which is fairly less than the HOMA index of aromatic naphthalene. It can be inferred that  $\pi$ -electron delocalization of the ring involving proton transfer is considerably deformed.

The molecule of (I) is generated by connecting 2-phenoxyaniline and naphthaldehyde units through a nitrogen bridge (Figure 1). Rings A(C1—C6), B(C7—C12) and C(C14—C23) are planar and the dihedral angles between them are A/B=77.41 (17)°, A/C=79.24 (15)°, B/C=8.29 (15)°. The hydrogen atom in the title compound (I) is located on nitrogen atom, thus the keto-amine tautomer is favored over the phenol-imine form. The presence of keto form can be also confirmed by N1—C13 and C15—O1 bond lengths. The C15—O1 bond length of 1.276 (7)Å indicates double-bond character while the N1—C13 bond length of 1.315 (6)Å indicates single-bond character. Similar results are also reported in the literature (Özek *et al.*, 2004; Takano *et al.*, 2009]. Intermolecular weak C—H··· $\pi$  ring interactions are also present in the crystal structure (Figure 2). Details of C—H··· $\pi$  contacts are given in Table 1.

### Experimental

(*E*)-1-((2-phenoxyphenylamino)methyl)naphthalen-2-ol was prepared by refluxing a mixture of a solution containing 2-hydroxy-1-naphthaldehyde (17.2 mg, 0.1 mmol) in ethanol (30 ml) and a solution containing 2-phenoxyaniline (18.5 mg, 0.1 mmol) in ethanol (20 ml). The reaction mixture was stirred for 1 h. under reflux. Single crystals of the title compound for x-ray analysis were obtained by slow evaporation of an ethanol solution (Yield 68%; m.p. 411-413 K ).

## Refinement

H atoms attached to carbon atoms were placed in calculated positions with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The coordinates of the amine hydrogen obtained from a difference map and refined isotropically with  $\text{N—H} = 0.86\text{\AA}$  constrain.

## Figures

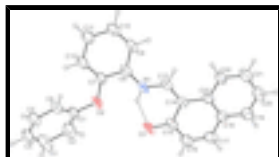


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability.

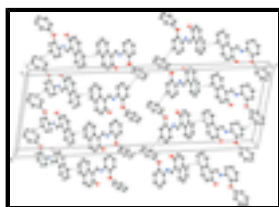


Fig. 2. The packing of (I), showing weak  $\text{C—H}\cdots\pi$  ring interactions with dashed lines.

## 1-[(E)-(2-Phenoxyanilino)methylene]naphthalen-2(1H)-one

### Crystal data

$\text{C}_{23}\text{H}_{17}\text{NO}_2$

$M_r = 339.38$

Monoclinic,  $C2/c$

Hall symbol:  $-C\ 2yc$

$a = 14.6428\ (11)\ \text{\AA}$

$b = 5.6297\ (3)\ \text{\AA}$

$c = 42.602\ (3)\ \text{\AA}$

$\beta = 101.175\ (6)^\circ$

$V = 3445.3\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1424$

$D_x = 1.309\ \text{Mg m}^{-3}$

Melting point = 411–413 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2565 reflections

$\theta = 2.8\text{--}24.1^\circ$

$\mu = 0.08\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Long prismatic rod, yellow

$0.40 \times 0.34 \times 0.23\ \text{mm}$

### Data collection

Stoe IPDSII  
diffractometer

Radiation source: fine-focus sealed tube  
plane graphite

Detector resolution:  $6.67\ \text{pixels mm}^{-1}$   
rotation scans

Absorption correction: integration  
(X-RED; Stoe & Cie, 2002)

$T_{\text{min}} = 0.960$ ,  $T_{\text{max}} = 0.996$

11832 measured reflections

2565 independent reflections

1522 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.212$

$\theta_{\text{max}} = 23.6^\circ$ ,  $\theta_{\text{min}} = 2.8^\circ$

$h = -16\text{--}16$

$k = -6\text{--}6$

$l = -47\text{--}47$

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.115$	H-atom parameters constrained
$wR(F^2) = 0.331$	$w = 1/[\sigma^2(F_o^2) + (0.1935P)^2]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2565 reflections	$(\Delta/\sigma)_{\max} = 0.001$
236 parameters	$\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0045 (17)

*Special details*

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

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4850 (4)	-0.3268 (11)	0.56310 (14)	0.0576 (16)
C2	0.4649 (5)	-0.1907 (13)	0.53616 (18)	0.0748 (19)
H2	0.4969	-0.0499	0.5345	0.090*
C3	0.3949 (5)	-0.2683 (15)	0.51107 (16)	0.082 (2)
H3	0.3797	-0.1800	0.4923	0.099*
C4	0.3489 (4)	-0.4759 (13)	0.51444 (17)	0.075 (2)
H4	0.3040	-0.5314	0.4976	0.090*
C5	0.3683 (4)	-0.6017 (12)	0.54216 (17)	0.0704 (19)
H5	0.3345	-0.7383	0.5445	0.084*
C6	0.4377 (4)	-0.5276 (11)	0.56682 (15)	0.0641 (18)
H6	0.4517	-0.6144	0.5857	0.077*
C7	0.6418 (3)	-0.3294 (10)	0.59332 (14)	0.0513 (14)

## supplementary materials

---

C8	0.7060 (3)	-0.2112 (10)	0.61624 (13)	0.0482 (14)
C9	0.7982 (4)	-0.2933 (11)	0.62192 (15)	0.0581 (16)
H9	0.8429	-0.2158	0.6369	0.070*
C10	0.8231 (4)	-0.4821 (11)	0.60609 (16)	0.0622 (17)
H10	0.8845	-0.5343	0.6106	0.075*
C11	0.7588 (4)	-0.5997 (11)	0.58329 (15)	0.0627 (17)
H11	0.7767	-0.7284	0.5722	0.075*
C12	0.6665 (4)	-0.5216 (12)	0.57725 (15)	0.0639 (18)
H12	0.6220	-0.6002	0.5623	0.077*
C13	0.7229 (4)	0.1281 (10)	0.65264 (13)	0.0503 (14)
H13	0.7872	0.1090	0.6571	0.060*
C14	0.6860 (4)	0.3109 (9)	0.66784 (13)	0.0487 (14)
C15	0.5865 (4)	0.3495 (10)	0.66058 (15)	0.0571 (16)
C16	0.5513 (4)	0.5463 (12)	0.67467 (18)	0.0716 (19)
H16	0.4876	0.5747	0.6699	0.086*
C17	0.6065 (4)	0.6971 (12)	0.69492 (18)	0.0704 (19)
H17	0.5797	0.8235	0.7039	0.084*
C18	0.7048 (4)	0.6642 (10)	0.70262 (14)	0.0548 (15)
C19	0.7619 (5)	0.8288 (11)	0.72230 (14)	0.0647 (17)
H19	0.7347	0.9566	0.7308	0.078*
C20	0.8549 (5)	0.8045 (12)	0.72899 (16)	0.0730 (19)
H20	0.8916	0.9137	0.7422	0.088*
C21	0.8956 (5)	0.6163 (13)	0.71618 (17)	0.078 (2)
H21	0.9599	0.5976	0.7213	0.094*
C22	0.8435 (4)	0.4567 (12)	0.69608 (16)	0.0691 (19)
H22	0.8728	0.3352	0.6870	0.083*
C23	0.7448 (4)	0.4752 (9)	0.68896 (12)	0.0481 (14)
N1	0.6738 (3)	-0.0213 (8)	0.63234 (10)	0.0493 (12)
H1	0.6146	0.0005	0.6285	0.059*
O1	0.5318 (2)	0.2158 (8)	0.64128 (11)	0.0720 (14)
O2	0.5522 (2)	-0.2386 (8)	0.58887 (11)	0.0755 (15)

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.031 (3)	0.080 (4)	0.055 (4)	0.002 (3)	-0.008 (3)	-0.025 (3)
C2	0.060 (4)	0.084 (4)	0.075 (5)	-0.016 (3)	-0.001 (3)	-0.005 (4)
C3	0.072 (5)	0.116 (6)	0.050 (4)	-0.011 (4)	-0.010 (3)	0.007 (4)
C4	0.044 (3)	0.098 (5)	0.072 (5)	-0.011 (3)	-0.014 (3)	-0.016 (4)
C5	0.045 (3)	0.084 (4)	0.075 (5)	-0.010 (3)	-0.005 (3)	-0.007 (4)
C6	0.049 (3)	0.074 (4)	0.060 (4)	0.001 (3)	-0.013 (3)	-0.002 (3)
C7	0.028 (3)	0.066 (3)	0.055 (3)	0.000 (2)	-0.003 (2)	-0.011 (3)
C8	0.033 (3)	0.068 (3)	0.042 (3)	-0.005 (2)	0.003 (2)	-0.006 (3)
C9	0.031 (3)	0.075 (4)	0.062 (4)	-0.010 (3)	-0.007 (3)	0.001 (3)
C10	0.032 (3)	0.077 (4)	0.074 (4)	-0.003 (3)	0.003 (3)	-0.008 (3)
C11	0.043 (3)	0.080 (4)	0.064 (4)	0.001 (3)	0.008 (3)	-0.014 (3)
C12	0.033 (3)	0.093 (4)	0.061 (4)	-0.007 (3)	-0.002 (3)	-0.022 (3)
C13	0.033 (3)	0.071 (3)	0.043 (3)	0.000 (3)	-0.003 (2)	-0.001 (3)

C14	0.034 (3)	0.062 (3)	0.046 (3)	-0.003 (2)	-0.003 (2)	0.001 (3)
C15	0.029 (3)	0.069 (4)	0.069 (4)	-0.003 (3)	0.000 (3)	-0.001 (3)
C16	0.034 (3)	0.090 (5)	0.088 (5)	0.006 (3)	0.004 (3)	-0.012 (4)
C17	0.048 (3)	0.080 (4)	0.083 (5)	0.014 (3)	0.012 (3)	-0.004 (4)
C18	0.049 (3)	0.061 (3)	0.050 (3)	0.004 (3)	0.000 (3)	0.000 (3)
C19	0.073 (4)	0.065 (4)	0.050 (3)	0.008 (3)	-0.001 (3)	-0.008 (3)
C20	0.070 (4)	0.082 (4)	0.060 (4)	-0.009 (4)	-0.005 (3)	-0.013 (3)
C21	0.045 (3)	0.106 (5)	0.075 (5)	-0.004 (3)	-0.007 (3)	-0.021 (4)
C22	0.038 (3)	0.093 (4)	0.070 (4)	0.003 (3)	-0.007 (3)	-0.034 (3)
C23	0.041 (3)	0.058 (3)	0.040 (3)	0.003 (2)	-0.004 (2)	0.002 (2)
N1	0.027 (2)	0.071 (3)	0.045 (3)	-0.006 (2)	-0.0059 (19)	-0.006 (2)
O1	0.0240 (19)	0.097 (3)	0.087 (3)	-0.0049 (19)	-0.009 (2)	-0.018 (3)
O2	0.028 (2)	0.106 (3)	0.083 (3)	0.004 (2)	-0.013 (2)	-0.047 (3)

*Geometric parameters (Å, °)*

C1—C6	1.351 (9)	C12—H12	0.9300
C1—C2	1.364 (9)	C13—N1	1.315 (6)
C1—O2	1.414 (6)	C13—C14	1.381 (8)
C2—C3	1.400 (9)	C13—H13	0.9300
C2—H2	0.9300	C14—C15	1.447 (7)
C3—C4	1.370 (10)	C14—C23	1.451 (7)
C3—H3	0.9300	C15—O1	1.276 (7)
C4—C5	1.359 (10)	C15—C16	1.404 (9)
C4—H4	0.9300	C16—C17	1.359 (9)
C5—C6	1.377 (8)	C16—H16	0.9300
C5—H5	0.9300	C17—C18	1.425 (8)
C6—H6	0.9300	C17—H17	0.9300
C7—C12	1.366 (8)	C18—C23	1.395 (8)
C7—O2	1.387 (6)	C18—C19	1.411 (8)
C7—C8	1.387 (7)	C19—C20	1.344 (10)
C8—N1	1.400 (7)	C19—H19	0.9300
C8—C9	1.403 (8)	C20—C21	1.379 (10)
C9—C10	1.347 (9)	C20—H20	0.9300
C9—H9	0.9300	C21—C22	1.368 (8)
C10—C11	1.383 (8)	C21—H21	0.9300
C10—H10	0.9300	C22—C23	1.422 (8)
C11—C12	1.397 (8)	C22—H22	0.9300
C11—H11	0.9300	N1—H1	0.8600
C6—C1—C2	122.4 (5)	N1—C13—H13	117.6
C6—C1—O2	119.9 (6)	C14—C13—H13	117.6
C2—C1—O2	117.4 (6)	C13—C14—C15	118.8 (5)
C1—C2—C3	118.3 (7)	C13—C14—C23	121.8 (5)
C1—C2—H2	120.9	C15—C14—C23	119.3 (5)
C3—C2—H2	120.9	O1—C15—C16	120.3 (5)
C4—C3—C2	119.2 (7)	O1—C15—C14	122.0 (5)
C4—C3—H3	120.4	C16—C15—C14	117.7 (5)
C2—C3—H3	120.4	C17—C16—C15	122.9 (5)
C5—C4—C3	120.8 (6)	C17—C16—H16	118.6

## supplementary materials

---

C5—C4—H4	119.6	C15—C16—H16	118.6
C3—C4—H4	119.6	C16—C17—C18	120.9 (6)
C4—C5—C6	120.3 (6)	C16—C17—H17	119.5
C4—C5—H5	119.9	C18—C17—H17	119.5
C6—C5—H5	119.9	C23—C18—C19	120.0 (5)
C1—C6—C5	118.9 (6)	C23—C18—C17	119.4 (5)
C1—C6—H6	120.5	C19—C18—C17	120.5 (6)
C5—C6—H6	120.5	C20—C19—C18	121.3 (6)
C12—C7—O2	124.0 (5)	C20—C19—H19	119.4
C12—C7—C8	121.5 (5)	C18—C19—H19	119.4
O2—C7—C8	114.4 (5)	C19—C20—C21	119.5 (6)
C7—C8—N1	117.7 (5)	C19—C20—H20	120.2
C7—C8—C9	117.5 (5)	C21—C20—H20	120.2
N1—C8—C9	124.8 (5)	C22—C21—C20	121.4 (6)
C10—C9—C8	121.3 (5)	C22—C21—H21	119.3
C10—C9—H9	119.4	C20—C21—H21	119.3
C8—C9—H9	119.4	C21—C22—C23	120.4 (6)
C9—C10—C11	121.0 (6)	C21—C22—H22	119.8
C9—C10—H10	119.5	C23—C22—H22	119.8
C11—C10—H10	119.5	C18—C23—C22	117.3 (5)
C10—C11—C12	118.8 (6)	C18—C23—C14	119.9 (5)
C10—C11—H11	120.6	C22—C23—C14	122.8 (5)
C12—C11—H11	120.6	C13—N1—C8	128.1 (4)
C7—C12—C11	119.9 (5)	C13—N1—H1	115.9
C7—C12—H12	120.1	C8—N1—H1	115.9
C11—C12—H12	120.1	C7—O2—C1	118.3 (4)
N1—C13—C14	124.8 (5)		
C6—C1—C2—C3	-2.5 (11)	C14—C15—C16—C17	-1.0 (11)
O2—C1—C2—C3	-176.5 (6)	C15—C16—C17—C18	1.0 (11)
C1—C2—C3—C4	0.3 (11)	C16—C17—C18—C23	0.4 (10)
C2—C3—C4—C5	2.4 (12)	C16—C17—C18—C19	176.6 (7)
C3—C4—C5—C6	-3.0 (11)	C23—C18—C19—C20	-1.8 (10)
C2—C1—C6—C5	1.9 (10)	C17—C18—C19—C20	-177.9 (7)
O2—C1—C6—C5	175.7 (6)	C18—C19—C20—C21	0.6 (11)
C4—C5—C6—C1	0.9 (10)	C19—C20—C21—C22	1.7 (12)
C12—C7—C8—N1	-177.8 (5)	C20—C21—C22—C23	-2.7 (12)
O2—C7—C8—N1	1.2 (8)	C19—C18—C23—C22	0.7 (9)
C12—C7—C8—C9	1.2 (9)	C17—C18—C23—C22	176.9 (6)
O2—C7—C8—C9	-179.8 (5)	C19—C18—C23—C14	-177.9 (5)
C7—C8—C9—C10	-1.0 (9)	C17—C18—C23—C14	-1.7 (9)
N1—C8—C9—C10	177.9 (6)	C21—C22—C23—C18	1.5 (10)
C8—C9—C10—C11	1.0 (10)	C21—C22—C23—C14	-180.0 (6)
C9—C10—C11—C12	-1.1 (10)	C13—C14—C23—C18	176.9 (5)
O2—C7—C12—C11	179.8 (6)	C15—C14—C23—C18	1.6 (8)
C8—C7—C12—C11	-1.4 (10)	C13—C14—C23—C22	-1.6 (9)
C10—C11—C12—C7	1.3 (10)	C15—C14—C23—C22	-176.9 (6)
N1—C13—C14—C15	-2.0 (9)	C14—C13—N1—C8	-179.5 (5)
N1—C13—C14—C23	-177.4 (5)	C7—C8—N1—C13	-173.3 (5)
C13—C14—C15—O1	2.0 (9)	C9—C8—N1—C13	7.7 (9)



C23—C14—C15—O1	177.4 (6)	C12—C7—O2—C1	-9.5 (10)
C13—C14—C15—C16	-175.8 (6)	C8—C7—O2—C1	171.6 (6)
C23—C14—C15—C16	-0.3 (9)	C6—C1—O2—C7	84.9 (8)
O1—C15—C16—C17	-178.8 (7)	C2—C1—O2—C7	-101.0 (7)

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

Cg1 and Cg2 are the centroids of the C1–C6 and C18–C23 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O1	0.86	1.87	2.561 (6)	136
C11—H11 $\cdots$ Cg1 <sup>i</sup>	0.93	2.87	3.664 (7)	144
C19—H19 $\cdots$ Cg2 <sup>ii</sup>	0.93	2.90	3.674 (7)	142

Symmetry codes: (i)  $x+1/2, y-1/2, z$ ; (ii)  $-x+3/2, y+1/2, -z+3/2$ .

Fig. 1

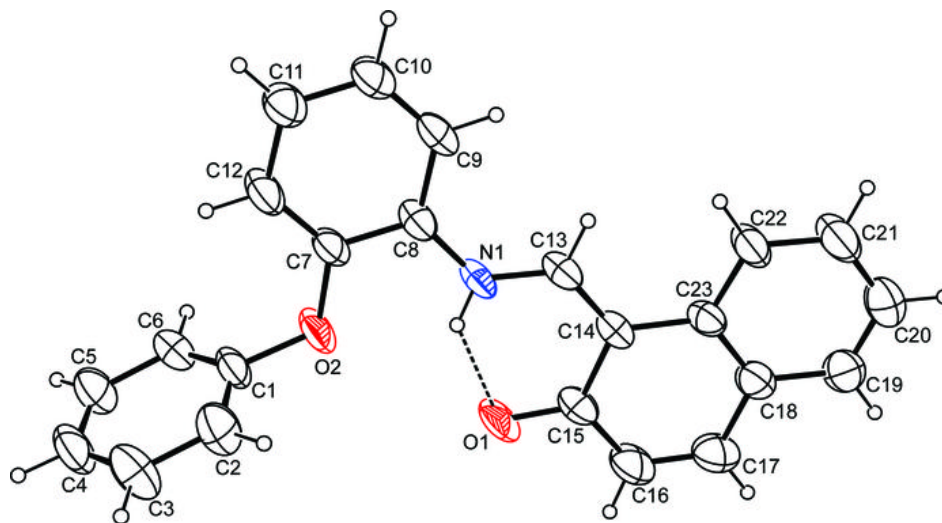


Fig. 2

