

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-Diethylamino-6-[(Z)-(4-hydroxy-anilino)methylidene]cyclohexa-2,4-dienone

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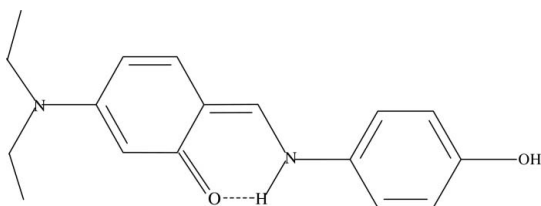
Received 3 November 2010; accepted 8 November 2010

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.065; wR factor = 0.189; data-to-parameter ratio = 15.1.

In the molecule of the title compound, $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2$, the aromatic rings are oriented at a dihedral angle of 6.23 (22)°. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding involving the amine H atom and the carbonyl O atom affects the conformation of the molecule. One of the ethyl arms is disordered over two conformations, with occupancies of 0.59 (2) and 0.41 (2). The crystal packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, and weak $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For general background to Schiff bases, see: Odabaşoğlu *et al.* (2004); Hadjoudis *et al.* (1987); Hodnett & Dunn (1970); Misra *et al.* (1981); Agarwal *et al.* (1983); Varma *et al.* (1986); Singh & Dash (1988); Pandey *et al.* (1999); El-Masry *et al.* (2000); Samadhiya & Halve (2001); Xu *et al.* (1994); Calligaris *et al.* (1972); Cohen *et al.* (1964); Moustakali-Mavridis *et al.* (1978). For related structures: Ersanlı *et al.* (2003); Şahin *et al.* (2005).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2$
 $M_r = 284.35$
 Monoclinic, $P2_1/c$
 $a = 8.2648$ (4) Å

$b = 11.8968$ (4) Å
 $c = 16.6149$ (7) Å
 $\beta = 112.400$ (3)°
 $V = 1510.39$ (11) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹

$T = 296$ K
 $0.54 \times 0.44 \times 0.26$ mm

Data collection

Stoe IPDS 2 diffractometer
 Absorption correction: integration
 ($X\text{-RED32}$; Stoe & Cie, 2002)
 $T_{\text{min}} = 0.957$, $T_{\text{max}} = 0.979$

18967 measured reflections
 3143 independent reflections
 2416 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.189$
 $S = 1.06$
 3143 reflections
 208 parameters
 13 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C8–C13 ring.

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1B}\cdots\text{O2}$ | 0.90 (3) | 1.80 (3) | 2.574 (2) | 142 (2) |
| $\text{O1}-\text{H1A}\cdots\text{O2}^{\text{i}}$ | 0.87 (4) | 1.75 (4) | 2.599 (2) | 165 (4) |
| $\text{C16A}-\text{H16A}\cdots\text{O2}^{\text{ii}}$ | 0.96 | 2.52 | 3.306 (7) | 139 |
| $\text{C5}-\text{H6}\cdots\text{Cg1}^{\text{iii}}$ | 0.93 | 2.93 | 3.799 (3) | 156 |

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

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, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS 2 diffractometer (purchased under grant No. F279 of the University Research Fund).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2307).

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supplementary materials

Acta Cryst. (2010). E66, o3138–o3139 [doi:10.1107/S1600536810045939]

3-Diethylamino-6-[(Z)-(4-hydroxyanilino)methylidene]cyclohexa-2,4-dienone

E. Soydemir, O. Büyükgüngör and Ç. Albayrak

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives are also known to have biological activities such as antimicrobial (El-Masry *et al.*, 2000; Pandey *et al.*, 1999); antifungal (Singh & Dash 1988; Varma *et al.*, 1986); antitumor (Hodnett & Dunn 1970; Misra *et al.*, 1981; Agarwal *et al.*, 1983) and as herbicides (Samadhiya & Halve, 2001). In addition, Schiff bases have been used widely as ligands in the field of coordination chemistry (Calligaris *et al.*, 1972). There are two characteristic properties of Schiff bases, *viz.* photochromism and thermochromism (Cohen *et al.*, 1964; Moustakali-Mavridis *et al.*, 1978). These properties result from proton transfer from the O atom to the imine N atom (Hadjoudis *et al.*, 1987; Xu *et al.*, 1994).

In the molecule of (I), the C13=O2 and N1—C7 bond lengths of 1.301 (2) and 1.307 (3) Å, respectively are in good agreement with those observed in 2-[(2-hydroxy-4-nitrophenyl)aminomethylene]cyclohexa-3,5-dien-1(2H)-one [1.298 (2) and 1.308 (2) Å; Ersanlı *et al.*, 2003] and 2-Hydroxy-6-[(2-methoxyphenyl)aminomethylene]cyclohexa-2,4-dienone [1.2931 (17) and 1.3043 (19) Å; Şahin *et al.*, 2005]. The study of Schiff bases has led to the proposal that molecules exhibiting thermochromism are planar, while those exhibiting photochromism are non-planar. This planarity of the molecule allows the proton to be transferred through the hydrogen bond in the ground state with a small energy requirement (Odabaşoğlu *et al.*, 2004; Hadjoudis *et al.*, 1987). The dihedral angle between benzene rings A(C1—C6) and B(C8—C13) is 6.49 (22)° (Fig.1). These two rings are twisted slightly about the methylene amino group with torsion angles of -0.5 (3)° [C2—C1—N1—C7] and 179.48 (18)° [N1—C7—C8—C9], respectively. C16 was disordered over two positions A and B (C16A and C16B) with the occupancy factors refined to 0.59 (2) and 0.41 (2). Intramolecular N1—H1A···O2 hydrogen bonding contributes to the overall planarity of the molecule. In crystal packing, the weak [C5—H6···Cg1(x, -1/2 - y, -1/2 + z)] interaction, and the hydrogen bonds [C16A—H16A···O2(1 - x, 1/2 + y, 1/2 - z)] and [O1—H1A···O2(1 + x, 1/2 - y, 1/2 + z)] are listed in Table 1 and labelled in Fig.2.

Experimental

The compound (Z)-6-[(4-hydroxyphenylamino)methylene]-3-(diethylamino)cyclohexa-2,4-dienone was prepared by refluxing a mixture of a solution containing 5-(diethylamino)-2-hydroxybenzaldehyde (0.5 g, 2.59 mmol) in 20 ml ethanol and a solution containing 4-hydroxyaniline (0.28 g, 2.59 mmol) in 20 ml ethanol. The reaction mixture was stirred for 1 h under reflux. The crystals of the title compound were obtained by slow evaporation from ethyl alcohol (yield % 73; m.p. 468–470 K).

Refinement

All H atoms were placed in calculated positions except H1A and H1B which were located in a difference Fourier map. All carbon-bound H atoms were refined using a riding model with C—H = 0.93 to 0.97 Å and $U_{iso}(H) = 1.2–1.5 U_{eq}(C)$.

Figures

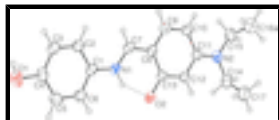


Fig. 1. An *ORTEP* view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. Only the major disorder component C16A are displayed. The dashed line indicates the intramolecular hydrogen bond.

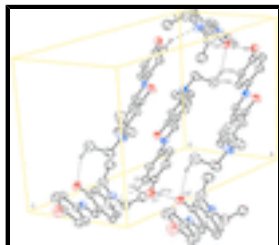


Fig. 2. A packing diagram for (I), hydrogen bonds and C—H... π interaction are drawn as dashed lines. [Symmetry codes: (i) $1 - x, 1/2 + y, 1/2 + z$; (ii) $x + 1, 1/2 - y, 1/2 - z$; (iii) $x, 1/2 - y, 1/2 + z$]

3-Diethylamino-6-[(Z)-(4-hydroxyanilino)methylidene]cyclohexa-2,4- dienone

Crystal data

$C_{17}H_{20}N_2O_2$

$M_r = 284.35$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.2648$ (4) Å

$b = 11.8968$ (4) Å

$c = 16.6149$ (7) Å

$\beta = 112.400$ (3)°

$V = 1510.39$ (11) Å³

$Z = 4$

$F(000) = 608$

$D_x = 1.250$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 18967 reflections

$\theta = 1.7$ – 28.0 °

$\mu = 0.08$ mm⁻¹

$T = 296$ K

Prism, brown

$0.54 \times 0.44 \times 0.26$ mm

Data collection

Stoe IPDS 2
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 6.67 pixels mm⁻¹

rotation method scans

Absorption correction: integration
(*X-RED32*; Stoe & Cie, 2002)

$T_{\min} = 0.957$, $T_{\max} = 0.979$

18967 measured reflections

3143 independent reflections

2416 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.067$

$\theta_{\max} = 26.5$ °, $\theta_{\min} = 2.2$ °

$h = -10 \rightarrow 10$

$k = -14 \rightarrow 14$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.065$$

$$wR(F^2) = 0.189$$

$$S = 1.06$$

3143 reflections

208 parameters

13 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0969P)^2 + 0.3716P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|------------|--------------|--------------|----------------------------------|-----------|
| C1 | 0.8028 (2) | 0.31443 (16) | 0.65717 (13) | 0.0483 (5) | |
| C2 | 0.9660 (3) | 0.35845 (19) | 0.70654 (14) | 0.0549 (5) | |
| H3 | 1.0126 | 0.4163 | 0.6844 | 0.066* | |
| C3 | 1.0587 (3) | 0.31624 (19) | 0.78840 (14) | 0.0581 (5) | |
| H2 | 1.1680 | 0.3463 | 0.8212 | 0.070* | |
| C4 | 0.9927 (3) | 0.22989 (19) | 0.82301 (13) | 0.0550 (5) | |
| C5 | 0.8278 (3) | 0.1876 (2) | 0.77383 (16) | 0.0627 (6) | |
| H6 | 0.7800 | 0.1309 | 0.7963 | 0.075* | |
| C6 | 0.7352 (3) | 0.22939 (19) | 0.69203 (15) | 0.0593 (6) | |
| H5 | 0.6253 | 0.2001 | 0.6595 | 0.071* | |
| C7 | 0.7361 (2) | 0.42738 (17) | 0.52483 (13) | 0.0501 (5) | |
| H7 | 0.8386 | 0.4689 | 0.5495 | 0.060* | |
| C8 | 0.6253 (2) | 0.44989 (16) | 0.43902 (12) | 0.0470 (5) | |
| C9 | 0.6675 (3) | 0.53469 (18) | 0.39085 (14) | 0.0536 (5) | |
| H9 | 0.7688 | 0.5766 | 0.4180 | 0.064* | |
| C10 | 0.5666 (3) | 0.55735 (19) | 0.30686 (14) | 0.0567 (5) | |
| H10 | 0.6000 | 0.6134 | 0.2774 | 0.068* | |
| C11 | 0.4090 (3) | 0.49573 (19) | 0.26311 (13) | 0.0556 (5) | |
| C12 | 0.3648 (3) | 0.41193 (19) | 0.30976 (13) | 0.0552 (5) | |
| H12 | 0.2638 | 0.3701 | 0.2816 | 0.066* | |
| C13 | 0.4654 (2) | 0.38791 (17) | 0.39687 (12) | 0.0473 (5) | |
| C14 | 0.1382 (4) | 0.4570 (2) | 0.13497 (16) | 0.0802 (8) | |
| H15A | 0.0600 | 0.5023 | 0.0875 | 0.096* | |

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| | | | | | |
|------|--------------|--------------|--------------|-------------|----------|
| H15B | 0.0819 | 0.4447 | 0.1759 | 0.096* | |
| C15 | 0.3476 (4) | 0.6042 (3) | 0.12686 (17) | 0.0855 (8) | |
| H14A | 0.4063 | 0.6671 | 0.1634 | 0.103* | |
| H14B | 0.2416 | 0.6319 | 0.0815 | 0.103* | |
| C16A | 0.4682 (17) | 0.5521 (6) | 0.0854 (7) | 0.096 (3) | 0.59 (2) |
| H16A | 0.4960 | 0.6078 | 0.0508 | 0.144* | 0.59 (2) |
| H16B | 0.4097 | 0.4900 | 0.0491 | 0.144* | 0.59 (2) |
| H16C | 0.5741 | 0.5262 | 0.1304 | 0.144* | 0.59 (2) |
| C16B | 0.388 (3) | 0.5630 (9) | 0.0567 (10) | 0.097 (4) | 0.41 (2) |
| H16D | 0.4141 | 0.6249 | 0.0267 | 0.145* | 0.41 (2) |
| H16E | 0.2888 | 0.5224 | 0.0171 | 0.145* | 0.41 (2) |
| H16F | 0.4868 | 0.5137 | 0.0785 | 0.145* | 0.41 (2) |
| C17 | 0.1651 (5) | 0.3472 (3) | 0.1001 (2) | 0.1033 (11) | |
| H17A | 0.0543 | 0.3102 | 0.0725 | 0.155* | |
| H17B | 0.2406 | 0.3013 | 0.1468 | 0.155* | |
| H17C | 0.2177 | 0.3588 | 0.0583 | 0.155* | |
| N1 | 0.7013 (2) | 0.35026 (14) | 0.57193 (11) | 0.0500 (4) | |
| H1B | 0.597 (3) | 0.316 (2) | 0.5437 (17) | 0.066 (7)* | |
| N2 | 0.3028 (3) | 0.51988 (19) | 0.17918 (12) | 0.0712 (6) | |
| O1 | 1.0820 (2) | 0.18478 (17) | 0.90278 (11) | 0.0723 (5) | |
| H1A | 1.193 (5) | 0.197 (3) | 0.919 (3) | 0.131 (14)* | |
| O2 | 0.41712 (18) | 0.31201 (13) | 0.43958 (9) | 0.0573 (4) | |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0409 (9) | 0.0490 (11) | 0.0465 (10) | 0.0055 (8) | 0.0073 (8) | -0.0005 (8) |
| C2 | 0.0445 (10) | 0.0592 (12) | 0.0530 (12) | -0.0014 (9) | 0.0097 (9) | 0.0059 (10) |
| C3 | 0.0429 (10) | 0.0648 (13) | 0.0548 (12) | 0.0007 (9) | 0.0053 (9) | -0.0002 (10) |
| C4 | 0.0488 (10) | 0.0650 (13) | 0.0471 (11) | 0.0128 (9) | 0.0135 (9) | 0.0064 (9) |
| C5 | 0.0546 (11) | 0.0642 (14) | 0.0655 (14) | 0.0015 (10) | 0.0185 (11) | 0.0146 (11) |
| C6 | 0.0444 (10) | 0.0603 (13) | 0.0619 (13) | -0.0036 (9) | 0.0076 (9) | 0.0058 (10) |
| C7 | 0.0429 (9) | 0.0485 (10) | 0.0525 (11) | 0.0005 (8) | 0.0109 (8) | -0.0046 (9) |
| C8 | 0.0449 (9) | 0.0468 (10) | 0.0456 (10) | 0.0028 (8) | 0.0129 (8) | -0.0024 (8) |
| C9 | 0.0497 (10) | 0.0532 (11) | 0.0560 (12) | -0.0039 (9) | 0.0178 (9) | -0.0026 (9) |
| C10 | 0.0634 (12) | 0.0543 (12) | 0.0568 (12) | -0.0037 (10) | 0.0279 (10) | 0.0028 (10) |
| C11 | 0.0632 (12) | 0.0575 (12) | 0.0430 (10) | 0.0009 (10) | 0.0167 (9) | 0.0002 (9) |
| C12 | 0.0554 (11) | 0.0583 (12) | 0.0428 (10) | -0.0078 (9) | 0.0086 (9) | -0.0013 (9) |
| C13 | 0.0471 (10) | 0.0473 (10) | 0.0433 (10) | 0.0010 (8) | 0.0125 (8) | -0.0015 (8) |
| C14 | 0.0868 (16) | 0.0904 (19) | 0.0479 (13) | -0.0054 (12) | 0.0081 (12) | 0.0082 (12) |
| C15 | 0.119 (2) | 0.0810 (18) | 0.0530 (14) | -0.0084 (16) | 0.0290 (15) | 0.0098 (13) |
| C16A | 0.097 (3) | 0.096 (3) | 0.096 (3) | -0.0016 (10) | 0.0374 (14) | 0.0005 (10) |
| C16B | 0.097 (4) | 0.097 (4) | 0.096 (4) | -0.0010 (10) | 0.0381 (18) | 0.0011 (10) |
| C17 | 0.138 (3) | 0.095 (2) | 0.0714 (19) | -0.017 (2) | 0.0339 (19) | -0.0090 (17) |
| N1 | 0.0400 (8) | 0.0524 (10) | 0.0467 (9) | -0.0004 (7) | 0.0044 (7) | -0.0006 (7) |
| N2 | 0.0859 (13) | 0.0756 (13) | 0.0445 (10) | -0.0067 (10) | 0.0161 (9) | 0.0084 (9) |
| O1 | 0.0591 (10) | 0.0981 (14) | 0.0537 (9) | 0.0155 (9) | 0.0147 (8) | 0.0213 (9) |
| O2 | 0.0523 (8) | 0.0624 (9) | 0.0452 (8) | -0.0123 (6) | 0.0050 (6) | 0.0069 (7) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|----------------|-----------|
| C1—C6 | 1.385 (3) | C12—C13 | 1.397 (3) |
| C1—C2 | 1.387 (3) | C12—H12 | 0.9300 |
| C1—N1 | 1.409 (3) | C13—O2 | 1.301 (2) |
| C2—C3 | 1.377 (3) | C14—N2 | 1.479 (3) |
| C2—H3 | 0.9300 | C14—C17 | 1.481 (4) |
| C3—C4 | 1.387 (3) | C14—H15A | 0.9700 |
| C3—H2 | 0.9300 | C14—H15B | 0.9700 |
| C4—O1 | 1.358 (3) | C15—C16B | 1.415 (9) |
| C4—C5 | 1.389 (3) | C15—N2 | 1.465 (3) |
| C5—C6 | 1.374 (3) | C15—C16A | 1.541 (8) |
| C5—H6 | 0.9300 | C15—H14A | 0.9700 |
| C6—H5 | 0.9300 | C15—H14B | 0.9700 |
| C7—N1 | 1.307 (3) | C16A—H16A | 0.9600 |
| C7—C8 | 1.396 (3) | C16A—H16B | 0.9600 |
| C7—H7 | 0.9300 | C16A—H16C | 0.9600 |
| C8—C9 | 1.412 (3) | C16B—H16D | 0.9600 |
| C8—C13 | 1.441 (3) | C16B—H16E | 0.9600 |
| C9—C10 | 1.352 (3) | C16B—H16F | 0.9600 |
| C9—H9 | 0.9300 | C17—H17A | 0.9600 |
| C10—C11 | 1.429 (3) | C17—H17B | 0.9600 |
| C10—H10 | 0.9300 | C17—H17C | 0.9600 |
| C11—N2 | 1.366 (3) | N1—H1B | 0.90 (3) |
| C11—C12 | 1.394 (3) | O1—H1A | 0.87 (4) |
| C6—C1—C2 | 119.00 (19) | N2—C14—H15A | 108.9 |
| C6—C1—N1 | 117.45 (17) | C17—C14—H15A | 108.9 |
| C2—C1—N1 | 123.54 (19) | N2—C14—H15B | 108.9 |
| C3—C2—C1 | 119.8 (2) | C17—C14—H15B | 108.9 |
| C3—C2—H3 | 120.1 | H15A—C14—H15B | 107.8 |
| C1—C2—H3 | 120.1 | C16B—C15—N2 | 116.3 (5) |
| C2—C3—C4 | 121.39 (19) | N2—C15—C16A | 110.1 (4) |
| C2—C3—H2 | 119.3 | C16B—C15—H14A | 124.5 |
| C4—C3—H2 | 119.3 | N2—C15—H14A | 109.6 |
| O1—C4—C3 | 123.0 (2) | C16A—C15—H14A | 109.6 |
| O1—C4—C5 | 118.5 (2) | C16B—C15—H14B | 84.4 |
| C3—C4—C5 | 118.50 (19) | N2—C15—H14B | 109.6 |
| C6—C5—C4 | 120.2 (2) | C16A—C15—H14B | 109.6 |
| C6—C5—H6 | 119.9 | H14A—C15—H14B | 108.2 |
| C4—C5—H6 | 119.9 | C15—C16A—H16A | 109.5 |
| C5—C6—C1 | 121.10 (19) | C15—C16A—H16B | 109.5 |
| C5—C6—H5 | 119.4 | H16A—C16A—H16B | 109.5 |
| C1—C6—H5 | 119.4 | C15—C16A—H16C | 109.5 |
| N1—C7—C8 | 122.57 (18) | H16A—C16A—H16C | 109.5 |
| N1—C7—H7 | 118.7 | H16B—C16A—H16C | 109.5 |
| C8—C7—H7 | 118.7 | C15—C16B—H16D | 109.5 |
| C7—C8—C9 | 120.58 (18) | C15—C16B—H16E | 109.5 |
| C7—C8—C13 | 121.43 (18) | H16D—C16B—H16E | 109.5 |

supplementary materials

| | | | |
|-----------------|--------------|-----------------|--------------|
| C9—C8—C13 | 118.00 (17) | C15—C16B—H16F | 109.5 |
| C10—C9—C8 | 122.60 (19) | H16D—C16B—H16F | 109.5 |
| C10—C9—H9 | 118.7 | H16E—C16B—H16F | 109.5 |
| C8—C9—H9 | 118.7 | C14—C17—H17A | 109.5 |
| C9—C10—C11 | 120.5 (2) | C14—C17—H17B | 109.5 |
| C9—C10—H10 | 119.8 | H17A—C17—H17B | 109.5 |
| C11—C10—H10 | 119.8 | C14—C17—H17C | 109.5 |
| N2—C11—C12 | 121.0 (2) | H17A—C17—H17C | 109.5 |
| N2—C11—C10 | 121.2 (2) | H17B—C17—H17C | 109.5 |
| C12—C11—C10 | 117.73 (19) | C7—N1—C1 | 129.19 (18) |
| C11—C12—C13 | 122.95 (19) | C7—N1—H1B | 112.7 (16) |
| C11—C12—H12 | 118.5 | C1—N1—H1B | 118.1 (16) |
| C13—C12—H12 | 118.5 | C11—N2—C15 | 122.7 (2) |
| O2—C13—C12 | 121.47 (18) | C11—N2—C14 | 120.8 (2) |
| O2—C13—C8 | 120.34 (17) | C15—N2—C14 | 116.4 (2) |
| C12—C13—C8 | 118.20 (19) | C4—O1—H1A | 111 (3) |
| N2—C14—C17 | 113.2 (3) | | |
| C6—C1—C2—C3 | 0.9 (3) | C11—C12—C13—O2 | 177.4 (2) |
| N1—C1—C2—C3 | -178.06 (19) | C11—C12—C13—C8 | -2.5 (3) |
| C1—C2—C3—C4 | 0.1 (3) | C7—C8—C13—O2 | 2.8 (3) |
| C2—C3—C4—O1 | 178.7 (2) | C9—C8—C13—O2 | -177.25 (18) |
| C2—C3—C4—C5 | -1.3 (3) | C7—C8—C13—C12 | -177.39 (19) |
| O1—C4—C5—C6 | -178.5 (2) | C9—C8—C13—C12 | 2.6 (3) |
| C3—C4—C5—C6 | 1.5 (3) | C8—C7—N1—C1 | 176.75 (18) |
| C4—C5—C6—C1 | -0.5 (4) | C6—C1—N1—C7 | -179.4 (2) |
| C2—C1—C6—C5 | -0.7 (3) | C2—C1—N1—C7 | -0.5 (3) |
| N1—C1—C6—C5 | 178.3 (2) | C12—C11—N2—C15 | -177.6 (2) |
| N1—C7—C8—C9 | 179.48 (18) | C10—C11—N2—C15 | 4.1 (4) |
| N1—C7—C8—C13 | -0.5 (3) | C12—C11—N2—C14 | -0.5 (4) |
| C7—C8—C9—C10 | 178.19 (19) | C10—C11—N2—C14 | -178.9 (2) |
| C13—C8—C9—C10 | -1.8 (3) | C16B—C15—N2—C11 | 110.8 (10) |
| C8—C9—C10—C11 | 0.7 (3) | C16A—C15—N2—C11 | 83.7 (6) |
| C9—C10—C11—N2 | 177.9 (2) | C16B—C15—N2—C14 | -66.3 (10) |
| C9—C10—C11—C12 | -0.4 (3) | C16A—C15—N2—C14 | -93.5 (6) |
| N2—C11—C12—C13 | -177.0 (2) | C17—C14—N2—C11 | -81.1 (3) |
| C10—C11—C12—C13 | 1.4 (3) | C17—C14—N2—C15 | 96.1 (3) |

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the C8—C13 ring.

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|----------|-------------|-------------|---------------|
| N1—H1B \cdots O2 | 0.90 (3) | 1.80 (3) | 2.574 (2) | 142 (2) |
| O1—H1A \cdots O2 ⁱ | 0.87 (4) | 1.75 (4) | 2.599 (2) | 165 (4) |
| C16A—H16A \cdots O2 ⁱⁱ | 0.96 | 2.52 | 3.306 (7) | 139 |
| C5—H6 \cdots Cg1 ⁱⁱⁱ | 0.93 | 2.93 | 3.799 (3) | 156 |

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

Fig. 1

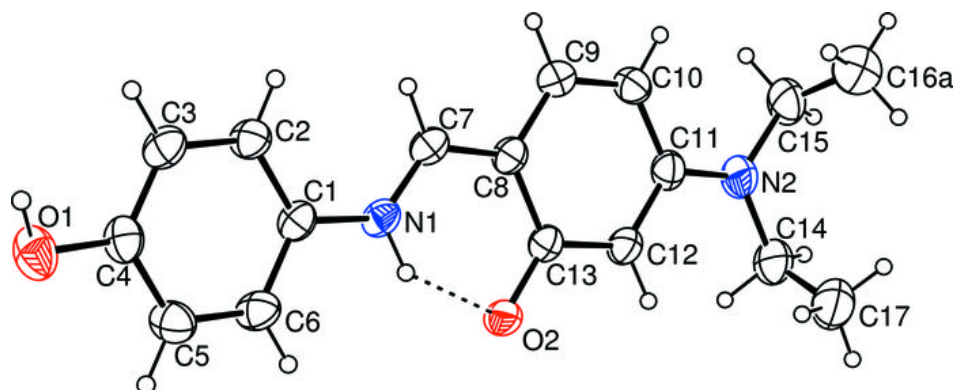


Fig. 2

