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5-Diethylamino-2-[(*Z*)-(4-methyl-3-nitrophenyl)iminomethyl]phenol

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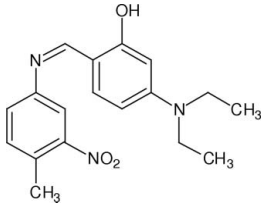
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.066; wR factor = 0.192; data-to-parameter ratio = 15.8.

In the crystal structure of the title compound, $C_{18}H_{21}N_3O_3$, the molecular conformation is stabilized by an intramolecular O—H···N hydrogen bond. The dihedral angle between the two aromatic rings is 3.97 (12)°. The nitro group is almost coplanar with the aromatic ring to which it is attached [dihedral angle = 6.3 (4)°].

Related literature

For related literature, see: Agarwal *et al.* (1983); Butcher *et al.* (2007); Büyükgüngör *et al.* (2007); El-Masry *et al.* (2000); Hodnett & Dunn (1970); Misra *et al.* (1981); Narayana *et al.* (2007); Odabaşoğlu *et al.* (2007); Pandey *et al.* (1999); Samadhiya & Halve (2001); Sarojini *et al.* (2007); Siddiqui *et al.* (2006); Singh & Dash (1988); Varma *et al.* (1986); Yathirajan *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{21}N_3O_3$
M_r = 327.38
Monoclinic, $P2_1/n$
a = 7.3157 (4) Å
b = 22.3708 (10) Å
c = 10.3849 (6) Å
β = 103.571 (5)°
V = 1652.12 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.41 × 0.37 × 0.33 mm

Data collection

STOE IPDS II two-circle-diffractometer
Absorption correction: none
34345 measured reflections
3823 independent reflections
3334 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.192
S = 0.97
3823 reflections
242 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.89 (3)	1.80 (3)	2.6010 (19)	147 (3)

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2072).

References

Agarwal, R., Chaudhary, K. C. & Misra, V. S. (1983). *Indian J. Chem.* **22B**, 308–310.
Butcher, R. J., Jasinski, J. P., Yathirajan, H. S., Vijesh, A. M. & Narayana, B. (2007). *Acta Cryst.* E**63**, o3748.
Büyükgüngör, O., Odabaşoğlu, M., Narayana, B., Vijesh, A. M. & Yathirajan, H. S. (2007). *Acta Cryst.* E**63**, o1996–o1998.
El-Masry, A. H., Fahmy, H. H. & Abdelwahed, S. H. A. (2000). *Molecules.* **5**, 1429–1438.
Hodnett, E. M. & Dunn, W. J. (1970). *J. Med. Chem.* **13**, 768–770.
Misra, V. S., Singh, S., Agarwal, R. & Chaudhary, K. C. (1981). *J. Chem. Soc. Pak.* **3**, 209–213.
Narayana, B., Sunil, K., Yathirajan, H. S., Sarojini, B. K. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2948.
Odabaşoğlu, M., Büyükgüngör, O., Narayana, B., Vijesh, A. M. & Yathirajan, H. S. (2007). *Acta Cryst.* E**63**, o1916–o1918.
Pandey, S. N., Sriram, D., Nath, G. & De Clercq, E. (1999). *Farmaco*, **54**, 624–628.
Samadhiya, S. & Halve, A. (2001). *Orient. J. Chem.* **17**, 119–122.
Sarojini, B. K., Yathirajan, H. S., Sunil, K., Narayana, B. & Bolte, M. (2007). *Acta Cryst.* E**63**, o3487.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Siddiqui, H. L., Iqbal, A., Ahmad, S. & Weaver, G. W. (2006). *Molecules.* **11**, 2006–2011.
Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Varma, R. S., Prakash, R., Khan, M. M. & Ali, A. (1986). *Indian Drugs*, **23**, 345–349.
Yathirajan, H. S., Vijesh, A. M., Narayana, B., Sarojini, B. K. & Bolte, M. (2007). *Acta Cryst.* E**63**, o936–o938.

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supplementary materials

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5-Diethylamino-2-[(*Z*)-(4-methyl-3-nitrophenyl)iminomethyl]phenol

B. K. Sarojini, B. Narayana, K. Mustafa, H. S. Yathirajan and M. Bolte

Comment

Schiff bases are used as substrates in the preparation of a number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. They are also used as substrates in the preparation of number of biologically active compounds (Siddiqui *et al.*, 2006). Some Schiff base derivatives are also known to have 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). The crystal structures of (*E*)-2-hydroxy-5-methyl-3-[(4-methyl-2 pyridyl)iminomethyl] benzaldehyde (Büyükgüngör *et al.*, 2007); (*E*)-2-hydroxy-5-methyl-3-[(2-pyridylimino) methyl]benzaldehyde Odabaşoğlu *et al.* (2007).

1-(4-{[(*E*)-(4-diethylamino-2-hydroxy phenyl)methylene]amino}phenyl)ethanone (Yathirajan *et al.* (2007), 2-{(*E*)-[(2-chloro-5-nitrophenyl)imino]methyl}-5-(diethylamino)phenol (Butcher *et al.*, 2007), 2-bromo-*N*'-[(*E*)-(4-fluorophenyl)methylene]-5-methoxybenzohydrazide monohydrate (Narayana *et al.*, 2007), 2-bromo-*N*'-isopropylidene-5-methoxybenzohydrazide (Sarojini *et al.*, 2007) have been reported. As a part of our ongoing investigations on the synthesis and structure of Schiff bases, we have determined the structure of the title compound (I).

In the structure of the title compound the oxazine ring adopts a half-chair conformation. The molecular conformation is stabilized by an intramolecular O—H···N hydroggen bond. The dihedral angle between the two aromatic rings is 3.97 (12) Å. The nitro groups is almost co planar with the aromatic ring to which it is attached [dihedral angle 6.3 (4)°].

Experimental

A mixture of 4-methyl-3-nitroaniline (1.52 g, 0.01 mol) and 4-(diethylamino)-2-hydroxybenzaldehyde (1.93 g, 0.01 mol) in 35 ml of ethanol containing 2 drops of 4 *M* sulfuric acid was refluxed for about 9 h. On cooling, a solid separated. It was filtered off and recrystallized from acetone (m.p.: 403–407 K). Analysis found: C 65.98, H 6.41, N 12.78%; $C_{18}H_{21}N_3O_3$ requires: C 66.04, H 6.47, N 12.84%.

Refinement

H atoms were found in a difference map, but those bonded to C were geometrically positioned and refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C) or U(H) = 1.5 U_{eq}(C_{methyl})] using a riding model with C—H ranging from 0.95Å to 0.99 Å. The methyl group bonded to the aromatic ring was allowed to rotate but not to tip. The hydroxyl H atom was freely refined. One of the ethyl chains is disordered over two sites with a site occupation factor of 0.602 (7) for the major occupied site. The bond lengths and angles of the disordered groups were restrained to have the same values as the non-disordered one.

Figures

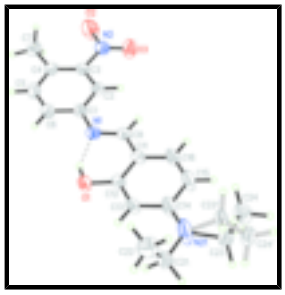


Fig. 1. Perspective view of the title compound with the atom numbering scheme and displacement ellipsoids drawn at the 50% probability level. The minor occupied site of the disordered ethyl chain is drawn with open bonds. The intramolecular hydrogen bond is shown as a dashed line.

5-Diethylamino-2-[(*Z*)-(4-methyl-3-nitrophenyl)iminomethyl]phenol

Crystal data

$C_{18}H_{21}N_3O_3$	F_{000} = 696
M_r = 327.38	D_x = 1.316 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.3157 (4) Å	Cell parameters from 32963 reflections
b = 22.3708 (10) Å	θ = 2.8–27.5º
c = 10.3849 (6) Å	μ = 0.09 mm^{-1}
β = 103.571 (5)º	T = 173 (2) K
V = 1652.12 (15) Å^3	Block, orange
Z = 4	0.41 × 0.37 × 0.33 mm

Data collection

STOE IPDS II two-circle-diffractometer	3334 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.032
Monochromator: graphite	θ_{max} = 27.6º
T = 173(2) K	θ_{min} = 2.7º
ω scans	h = −9→9
Absorption correction: none	k = −29→28
34345 measured reflections	l = −13→12
3823 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.066	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.192	$w = 1/[\sigma^2(F_o^2) + (0.1026P)^2 + 1.2402P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 0.97	$(\Delta/\sigma)_{max} < 0.001$
3823 reflections	$\Delta\rho_{max}$ = 0.84 e Å^{-3}
242 parameters	$\Delta\rho_{min}$ = −0.65 e Å^{-3}
9 restraints	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.055 (7)

Special details

Experimental. ;

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}	Occ. (<1)
O1	0.38176 (19)	0.59715 (6)	0.59143 (14)	0.0370 (3)	
H1	0.322 (5)	0.5734 (15)	0.526 (3)	0.081 (10)*	
O2	0.0769 (3)	0.39185 (11)	−0.12486 (19)	0.0797 (7)	
O3	0.3429 (2)	0.43512 (10)	−0.05554 (17)	0.0634 (5)	
N1	0.3258 (2)	0.53706 (7)	0.37122 (14)	0.0319 (3)	
N2	0.1885 (2)	0.41980 (8)	−0.04052 (16)	0.0422 (4)	
C1	0.2115 (2)	0.49854 (8)	0.27839 (17)	0.0313 (4)	
C2	0.2568 (2)	0.47627 (8)	0.16421 (17)	0.0319 (4)	
H2	0.3738	0.4860	0.1448	0.038*	
C3	0.1293 (2)	0.43970 (8)	0.07904 (17)	0.0331 (4)	
C4	−0.0456 (3)	0.42242 (9)	0.10075 (18)	0.0363 (4)	
C5	−0.0846 (3)	0.44508 (10)	0.2170 (2)	0.0429 (5)	
H5	−0.2005	0.4348	0.2377	0.051*	
C6	0.0388 (3)	0.48186 (9)	0.30300 (19)	0.0400 (4)	
H6	0.0054	0.4961	0.3806	0.048*	
C7	−0.1915 (3)	0.38345 (10)	0.0125 (2)	0.0460 (5)	
H7A	−0.2978	0.3776	0.0536	0.069*	
H7B	−0.1358	0.3446	0.0005	0.069*	
H7C	−0.2355	0.4029	−0.0738	0.069*	
C8	0.4853 (2)	0.55697 (8)	0.35579 (16)	0.0314 (4)	
H8	0.5288	0.5445	0.2808	0.038*	
C11	0.5982 (2)	0.59736 (7)	0.44885 (16)	0.0303 (4)	
C12	0.5429 (2)	0.61720 (7)	0.56401 (16)	0.0289 (4)	
C13	0.6516 (3)	0.65743 (8)	0.65132 (18)	0.0336 (4)	

H13	0.6094	0.6708	0.7262	0.040*	
C14	0.8232 (3)	0.67875 (10)	0.6305 (2)	0.0485 (6)	
C15	0.8784 (3)	0.65861 (11)	0.5147 (2)	0.0545 (6)	
H15	0.9933	0.6723	0.4976	0.065*	
C16	0.7681 (3)	0.61986 (9)	0.42803 (19)	0.0413 (5)	
H16	0.8078	0.6078	0.3512	0.050*	
N21	0.9350 (3)	0.71679 (12)	0.7186 (2)	0.0826 (9)	
C21	0.8793 (3)	0.74053 (10)	0.8340 (2)	0.0443 (5)	
H21A	0.9936	0.7539	0.8991	0.053*	
H21B	0.8223	0.7078	0.8756	0.053*	
C22	0.7429 (4)	0.79165 (10)	0.8061 (3)	0.0570 (6)	
H22A	0.7130	0.8047	0.8889	0.086*	
H22B	0.6275	0.7787	0.7437	0.086*	
H22C	0.7992	0.8249	0.7676	0.086*	
C23	1.1389 (6)	0.72798 (15)	0.7141 (3)	0.0403 (10)	0.602 (7)
H23A	1.2166	0.7372	0.8035	0.048*	0.602 (7)
H23B	1.1925	0.6927	0.6787	0.048*	0.602 (7)
C24	1.1300 (7)	0.78111 (17)	0.6227 (4)	0.0508 (11)	0.602 (7)
H24A	1.2571	0.7911	0.6143	0.076*	0.602 (7)
H24B	1.0761	0.8154	0.6594	0.076*	0.602 (7)
H24C	1.0511	0.7712	0.5353	0.076*	0.602 (7)
C23'	1.0482 (7)	0.7645 (2)	0.6538 (5)	0.0332 (13)	0.398 (7)
H23C	1.0022	0.7664	0.5563	0.040*	0.398 (7)
H23D	1.0447	0.8048	0.6922	0.040*	0.398 (7)
C24'	1.2430 (8)	0.7363 (3)	0.6948 (6)	0.0449 (15)	0.398 (7)
H24D	1.3333	0.7610	0.6621	0.067*	0.398 (7)
H24E	1.2396	0.6961	0.6569	0.067*	0.398 (7)
H24F	1.2813	0.7338	0.7916	0.067*	0.398 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0384 (7)	0.0395 (7)	0.0379 (7)	−0.0090 (5)	0.0184 (6)	−0.0085 (6)
O2	0.0791 (13)	0.1144 (17)	0.0515 (10)	−0.0434 (12)	0.0274 (9)	−0.0426 (11)
O3	0.0452 (9)	0.0981 (14)	0.0518 (9)	−0.0122 (9)	0.0212 (7)	−0.0298 (9)
N1	0.0352 (8)	0.0312 (7)	0.0286 (7)	0.0030 (6)	0.0062 (6)	−0.0011 (6)
N2	0.0437 (9)	0.0513 (10)	0.0318 (8)	−0.0039 (7)	0.0089 (7)	−0.0086 (7)
C1	0.0333 (8)	0.0316 (8)	0.0277 (8)	0.0048 (6)	0.0048 (6)	0.0006 (6)
C2	0.0306 (8)	0.0351 (9)	0.0296 (8)	0.0027 (7)	0.0064 (6)	0.0007 (7)
C3	0.0353 (9)	0.0373 (9)	0.0256 (8)	0.0035 (7)	0.0051 (7)	−0.0004 (7)
C4	0.0340 (9)	0.0396 (10)	0.0331 (9)	−0.0006 (7)	0.0034 (7)	0.0012 (7)
C5	0.0342 (9)	0.0546 (12)	0.0416 (10)	−0.0046 (8)	0.0125 (8)	−0.0033 (9)
C6	0.0387 (10)	0.0495 (11)	0.0341 (9)	0.0009 (8)	0.0130 (7)	−0.0055 (8)
C7	0.0405 (10)	0.0505 (12)	0.0428 (11)	−0.0081 (9)	0.0016 (8)	−0.0032 (9)
C8	0.0389 (9)	0.0300 (8)	0.0258 (8)	0.0041 (7)	0.0085 (7)	0.0008 (6)
C11	0.0374 (9)	0.0279 (8)	0.0271 (8)	0.0014 (6)	0.0107 (7)	0.0010 (6)
C12	0.0334 (8)	0.0265 (8)	0.0292 (8)	0.0007 (6)	0.0118 (6)	0.0028 (6)
C13	0.0409 (9)	0.0336 (9)	0.0311 (8)	−0.0050 (7)	0.0178 (7)	−0.0050 (7)

C14	0.0539 (12)	0.0533 (12)	0.0470 (11)	−0.0231 (10)	0.0295 (10)	−0.0202 (9)
C15	0.0573 (13)	0.0635 (14)	0.0552 (13)	−0.0289 (11)	0.0382 (11)	−0.0256 (11)
C16	0.0515 (11)	0.0436 (10)	0.0362 (9)	−0.0083 (8)	0.0248 (8)	−0.0083 (8)
N21	0.0802 (15)	0.1071 (19)	0.0811 (16)	−0.0645 (14)	0.0604 (13)	−0.0646 (15)
C21	0.0471 (11)	0.0501 (11)	0.0397 (10)	−0.0118 (9)	0.0181 (8)	−0.0151 (9)
C22	0.0653 (15)	0.0449 (12)	0.0582 (14)	−0.0102 (10)	0.0090 (11)	0.0005 (10)
C23	0.036 (2)	0.0433 (18)	0.0420 (17)	−0.0064 (14)	0.0097 (14)	−0.0062 (14)
C24	0.060 (3)	0.045 (2)	0.053 (2)	−0.0138 (18)	0.0234 (19)	−0.0036 (16)
C23'	0.032 (2)	0.028 (2)	0.043 (3)	−0.0066 (18)	0.017 (2)	−0.0013 (19)
C24'	0.029 (3)	0.052 (3)	0.055 (3)	−0.003 (2)	0.014 (2)	−0.007 (2)

Geometric parameters (Å, °)

O1—C12	1.353 (2)	C14—N21	1.370 (3)
O1—H1	0.89 (3)	C14—C15	1.429 (3)
O2—N2	1.220 (2)	C15—C16	1.368 (3)
O3—N2	1.225 (2)	C15—H15	0.9500
N1—C8	1.294 (2)	C16—H16	0.9500
N1—C1	1.411 (2)	N21—C21	1.454 (3)
N2—C3	1.476 (2)	N21—C23	1.524 (4)
C1—C2	1.396 (2)	N21—C23'	1.593 (6)
C1—C6	1.397 (3)	C21—C22	1.501 (3)
C2—C3	1.391 (2)	C21—H21A	0.9900
C2—H2	0.9500	C21—H21B	0.9900
C3—C4	1.404 (3)	C22—H22A	0.9800
C4—C5	1.399 (3)	C22—H22B	0.9800
C4—C7	1.510 (3)	C22—H22C	0.9800
C5—C6	1.382 (3)	C23—C24	1.513 (5)
C5—H5	0.9500	C23—H23A	0.9900
C6—H6	0.9500	C23—H23B	0.9900
C7—H7A	0.9800	C24—H24A	0.9800
C7—H7B	0.9800	C24—H24B	0.9800
C7—H7C	0.9800	C24—H24C	0.9800
C8—C11	1.435 (2)	C23'—C24'	1.524 (7)
C8—H8	0.9500	C23'—H23C	0.9900
C11—C16	1.404 (3)	C23'—H23D	0.9900
C11—C12	1.420 (2)	C24'—H24D	0.9800
C12—C13	1.388 (2)	C24'—H24E	0.9800
C13—C14	1.406 (3)	C24'—H24F	0.9800
C13—H13	0.9500		
C12—O1—H1	109 (2)	C14—C15—H15	119.5
C8—N1—C1	122.18 (15)	C15—C16—C11	122.20 (17)
O2—N2—O3	122.25 (18)	C15—C16—H16	118.9
O2—N2—C3	118.63 (17)	C11—C16—H16	118.9
O3—N2—C3	118.95 (16)	C14—N21—C21	122.56 (18)
C2—C1—C6	117.71 (16)	C14—N21—C23	121.1 (2)
C2—C1—N1	125.28 (16)	C21—N21—C23	115.5 (2)
C6—C1—N1	117.01 (15)	C14—N21—C23'	115.0 (3)
C3—C2—C1	119.52 (16)	C21—N21—C23'	112.2 (2)

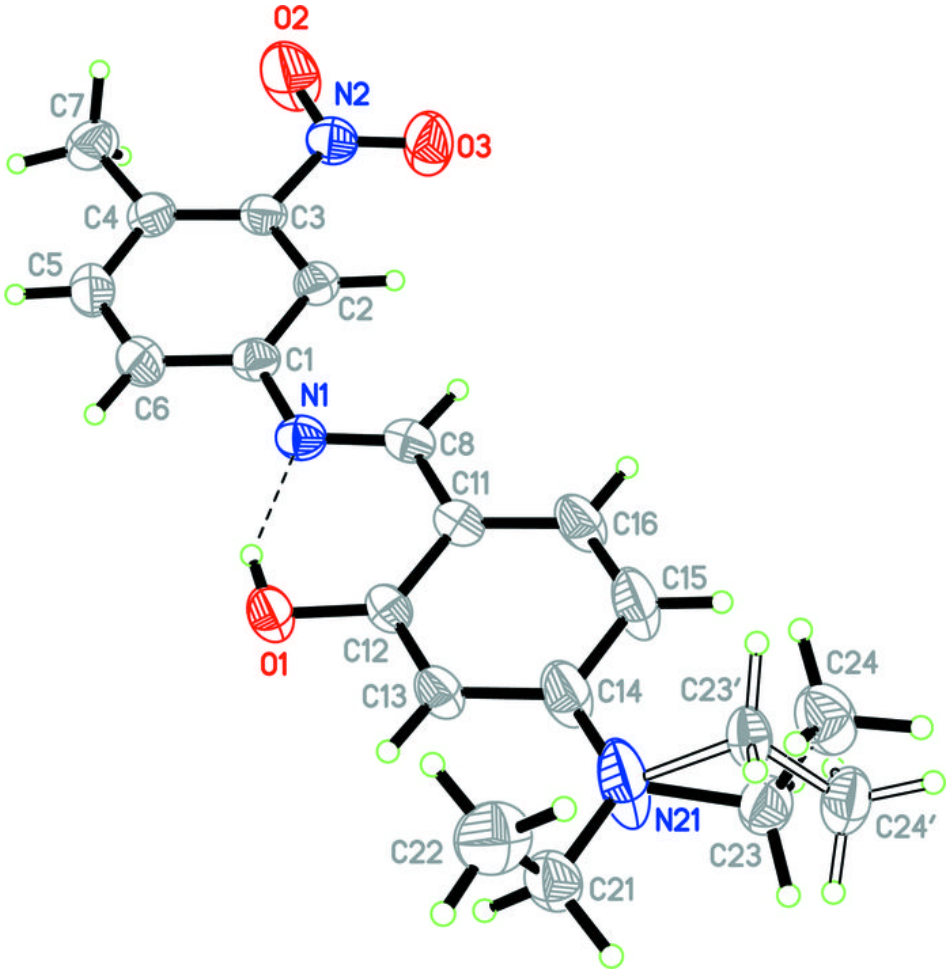
C3—C2—H2	120.2	N21—C21—C22	114.9 (2)
C1—C2—H2	120.2	N21—C21—H21A	108.5
C2—C3—C4	124.06 (16)	C22—C21—H21A	108.5
C2—C3—N2	115.27 (16)	N21—C21—H21B	108.5
C4—C3—N2	120.67 (16)	C22—C21—H21B	108.5
C5—C4—C3	114.64 (17)	H21A—C21—H21B	107.5
C5—C4—C7	118.31 (18)	C21—C22—H22A	109.5
C3—C4—C7	127.04 (17)	C21—C22—H22B	109.5
C6—C5—C4	122.57 (18)	H22A—C22—H22B	109.5
C6—C5—H5	118.7	C21—C22—H22C	109.5
C4—C5—H5	118.7	H22A—C22—H22C	109.5
C5—C6—C1	121.49 (17)	H22B—C22—H22C	109.5
C5—C6—H6	119.3	C24—C23—N21	104.5 (3)
C1—C6—H6	119.3	C24—C23—H23A	110.9
C4—C7—H7A	109.5	N21—C23—H23A	110.9
C4—C7—H7B	109.5	C24—C23—H23B	110.9
H7A—C7—H7B	109.5	N21—C23—H23B	110.9
C4—C7—H7C	109.5	H23A—C23—H23B	108.9
H7A—C7—H7C	109.5	C23—C24—H24A	109.5
H7B—C7—H7C	109.5	C23—C24—H24B	109.5
N1—C8—C11	121.82 (16)	H24A—C24—H24B	109.5
N1—C8—H8	119.1	C23—C24—H24C	109.5
C11—C8—H8	119.1	H24A—C24—H24C	109.5
C16—C11—C12	117.06 (16)	H24B—C24—H24C	109.5
C16—C11—C8	120.85 (16)	C24'—C23'—N21	98.7 (4)
C12—C11—C8	122.08 (16)	C24'—C23'—H23C	112.0
O1—C12—C13	118.13 (15)	N21—C23'—H23C	112.0
O1—C12—C11	120.53 (15)	C24'—C23'—H23D	112.0
C13—C12—C11	121.34 (16)	N21—C23'—H23D	112.0
C12—C13—C14	120.99 (16)	H23C—C23'—H23D	109.7
C12—C13—H13	119.5	C23'—C24'—H24D	109.5
C14—C13—H13	119.5	C23'—C24'—H24E	109.5
N21—C14—C13	121.52 (18)	H24D—C24'—H24E	109.5
N21—C14—C15	121.00 (18)	C23'—C24'—H24F	109.5
C13—C14—C15	117.47 (18)	H24D—C24'—H24F	109.5
C16—C15—C14	120.91 (18)	H24E—C24'—H24F	109.5
C16—C15—H15	119.5		
C8—N1—C1—C2	2.0 (3)	C8—C11—C12—C13	−178.38 (16)
C8—N1—C1—C6	−177.52 (16)	O1—C12—C13—C14	178.26 (19)
C6—C1—C2—C3	1.1 (3)	C11—C12—C13—C14	−1.7 (3)
N1—C1—C2—C3	−178.46 (16)	C12—C13—C14—N21	−177.6 (2)
C1—C2—C3—C4	−0.8 (3)	C12—C13—C14—C15	1.6 (3)
C1—C2—C3—N2	178.49 (16)	N21—C14—C15—C16	178.9 (3)
O2—N2—C3—C2	−173.9 (2)	C13—C14—C15—C16	−0.3 (4)
O3—N2—C3—C2	1.5 (3)	C14—C15—C16—C11	−0.9 (4)
O2—N2—C3—C4	5.5 (3)	C12—C11—C16—C15	0.8 (3)
O3—N2—C3—C4	−179.13 (19)	C8—C11—C16—C15	179.7 (2)
C2—C3—C4—C5	0.1 (3)	C13—C14—N21—C21	−3.9 (5)
N2—C3—C4—C5	−179.22 (17)	C15—C14—N21—C21	176.9 (3)

C2—C3—C4—C7	179.40 (18)	C13—C14—N21—C23	165.2 (3)
N2—C3—C4—C7	0.1 (3)	C15—C14—N21—C23	−13.9 (5)
C3—C4—C5—C6	0.4 (3)	C13—C14—N21—C23'	−146.3 (3)
C7—C4—C5—C6	−178.99 (19)	C15—C14—N21—C23'	34.6 (4)
C4—C5—C6—C1	−0.1 (3)	C14—N21—C21—C22	−78.8 (3)
C2—C1—C6—C5	−0.6 (3)	C23—N21—C21—C22	111.4 (3)
N1—C1—C6—C5	178.94 (18)	C23'—N21—C21—C22	64.4 (3)
C1—N1—C8—C11	178.33 (15)	C14—N21—C23—C24	90.6 (4)
N1—C8—C11—C16	−178.02 (17)	C21—N21—C23—C24	−99.5 (3)
N1—C8—C11—C12	0.8 (3)	C23'—N21—C23—C24	−3.6 (3)
C16—C11—C12—O1	−179.47 (16)	C14—N21—C23'—C24'	−105.5 (4)
C8—C11—C12—O1	1.6 (3)	C21—N21—C23'—C24'	108.3 (3)
C16—C11—C12—C13	0.5 (3)	C23—N21—C23'—C24'	4.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.89 (3)	1.80 (3)	2.6010 (19)	147 (3)

Fig. 1


O2
N2
O3
C7
C4
C3
C5
C2
C1
C6
N1
C8
C11
C16
C15
O1
C12
C13
C14
C23′
C24
N21
C24′
C22
C23
C21