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4-[(1-Hydroxy-2-naphthyl)methyleneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Qiang Liang* and Qian Wang

Department of Chemistry, Teachers College of Qingdao University, Qingdao, Shandong 266071, People's Republic of China
Correspondence e-mail: qliang59@163.com

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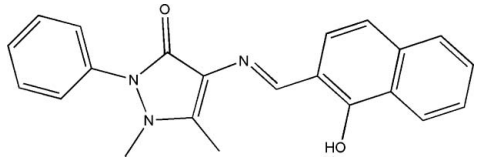
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.060; wR factor = 0.147; data-to-parameter ratio = 15.6.

The title antipyrine derivative, $C_{22}H_{19}N_3O_2$, was synthesized by the reaction of 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one and 1-hydroxynaphthalene-2-carbaldehyde in methanol solution. As expected, the compound adopts a *trans* configuration about the central C=N bond. The N atom is involved in an intramolecular O—H···N bond which stabilizes the molecular configuration. In the crystal structure, adjacent molecules stack with no short contacts.

Related literature

For background to the applications of antipyrine derivatives, see: Bashkatova *et al.* (2005); Bansal *et al.* (2007); Bondock *et al.* (2008); Capel *et al.* (1978); Coolen *et al.* (1999); Collado *et al.* (2000); Cunha *et al.* (2005); Evstropov *et al.* (1992); Khanduja *et al.* (1984); Madiha *et al.* (2007); Plesch *et al.* (1987); Radzikowska *et al.* (1995); Rehim *et al.* (2001); Turan-Zitouni *et al.* (2001); Yadav *et al.* (2003). For some typical structures of antipyrine derivatives, see: Liang *et al.* (2002); Li & Zhang (2004, 2005); Sun, Xie *et al.* (2006); Sun, Zhang, Jin *et al.* (2006); Sun, Zhang, Wang *et al.* (2006); Sun, Hao, Wei *et al.* (2009); Wen *et al.* (2005); You *et al.* (2004, 2006); Zhang & Li *et al.* (2005). For related structures involving Schiff bases, see: Ali *et al.* (2002); Bashkatova *et al.* (2005); Coolen *et al.* (1999); Collado *et al.* (2000); Cukurovali *et al.* (2002); Farag *et al.* (2009); Rehim *et al.* (2001); Sun, Hao, Yu *et al.* (2009); Tarafder *et al.* (2002).



Experimental

Crystal data

$C_{22}H_{19}N_3O_2$
M_r = 357.40
Monoclinic, $P2_1/c$
a = 8.0636 (7) Å
b = 7.4407 (6) Å
c = 30.169 (3) Å
β = 94.329 (2)°
V = 1804.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.23 × 0.10 × 0.02 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.981, T_{max} = 0.998
14746 measured reflections
3942 independent reflections
2403 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.147
S = 1.04
3942 reflections
252 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N3	0.82	1.84	2.569 (2)	148

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2002); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2081).

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supplementary materials

Acta Cryst. (2010). E**66**, o1968-o1969 [**doi:10.1107/S1600536810026450**]

4-[(1-Hydroxy-2-naphthyl)methyleneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

Q. Liang and Q. Wang

Comment

Since antipyrine was first synthesized by Knorr in 1883, the antipyrine and its derivatives exhibit a wide range of biologcial or chemical activities and applications (Capel *et al.*, 1978; Radzikowska *et al.*, 1995; Khanduja *et al.*, 1984; Bondock *et al.*, 2008; Cunha *et al.*, 2005; Plesch *et al.*, 1987; Madiha *et al.*, 2007; Evstropov *et al.*, 1992; Turan-Zitouni *et al.*, 2001; Bansal *et al.*, 2007; Bashkatova *et al.*, 2005; Rehim *et al.*, 2001; Collado *et al.*, 2000; Coolen *et al.*, 1999; Yadav *et al.*, 2003). A few crystal structures of antipyrine derivatives have been investigated (Liang *et al.*, 2002; Li & Zhang, 2004, 2005; Zhang & Li, 2005; You, *et al.*, 2004, 2006; Wen, 2005; Sun, Zhang, Jin *et al.*, 2006, Sun, Zhang, Wang *et al.*, 2006; Sun, Xie *et al.*, 2006 ; Sun, Hao, Wei *et al.* 2009). Schiff bases condensed by aldehydes and amines have demonstrated significant biological, chemical or optical activites, and new examples are being tested for their antitumor, antimicrobial, antiviral, antioxidant, optical and photovoltaic activities (Tarafder *et al.*, 2002; Cukurovali *et al.*, 2002; Ali *et al.*, 2002; Bashkatova *et al.*, 2005; Rehim *et al.*, 2001; Collado *et al.*, 2000; Coolen *et al.*, 1999; Sun, Hao, Yu *et al.*, 2009; Farag *et al.*, 2009). As an extension of these works on the structural characterization of antipyrine derivatives, a new Schiff base compound, (I), is reported here.

As illustrated in Fig. 1, the compound (I) is a neutral 4-((1-hydroxynaphthalen-2-yl)methyleneamino)-1,2-dihydro-1,5-dimethyl- 2-phenylpyrazol-3-one molecule. Selected geometric parameters are listed in Table 1. The N2-N1-C1-C2 and C7-N1-C1-C6 torsion angles are 147.2 (2) and 115.1 (2) °, respectively. Atom O1 deviates from the pyrazoline mean plane by 0.140 (2) Å, whereas atom C10 and C11 deviate from it, on the opposite side, by 0.087 (2) and 0.614 (2) Å, respectively. The dihedral angle between the N1/N2/C7/C8/C9 pyrazoline ring and the C1-C6 benzene ring planes is 50.4 (3) °. The C12═N3 bond length of 1.290 (3) Å confirms to the value for a double bond. As a result of conjugation through the imino double bond, the C12-N3-C8-C9 and C12-N3-C8-C7 torsion angles are 172.8 (2) and -2.5 (3) ° respectively, the pyrazoline and C13-C22 naphthalene rings are nearly coplannar [mean deviation from the overall combined mean plane is 0.084 (3) Å]; the dihedral angle between the pyrazoline ring and C13-C22 naphthalene ring is 11.5 (3) °. As expected, the molecular structure of the Shiff base adopts a *trans* coonfigurations about the central C12═N3 bond as the other similar antipyrine derivatives that have been reported.

In the crystal structure, the molecules stack along the *a* axis with no short contacts except the O—H···N intramolecular hydrogen (Table 2 and Fig. 2).

Experimental

All the chemicals were obtained from commercial sources and used without purification. 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazole-3-one (0.5 mmol, 101.6 mg) and an equimolar quantity of 1-hydroxynaphthalene- 2-carbaldehyde (0.5 mmol, 86.1 mg) were dissolved in methanol (100 ml). The mixture was stirred for 1 h at room temperature to give a clear yellow solution. The resulting solution was kept in air for 8 d after which time yellow plane-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the methanol. (yield 95.2%). Analysis calculated for ($C_{18}H_{16}ClN_3O_2$): C 73.93, H 5.36, N 11.76%; found: C 73.55, H 5.42, N 11.73%.

Refinement

All H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(O) for phenolic H atom, U_{iso}(H) = 1.2 for aromatic H atoms or U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Figures

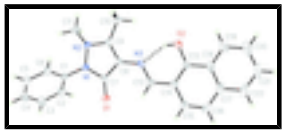

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The O—H···O hydrogen bond is shown as a dashed line.

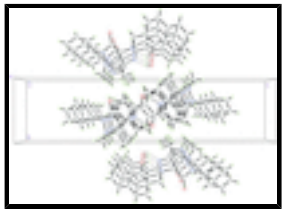

Fig. 2. The crystal packing of (I), viewed down the *a* axis. O—H···N contacts are shown as Hydrogen bonds.

4-[(1-Hydroxy-2-naphthyl)methyleneamino]-1,5-dimethyl-2-phenyl- 1*H*-pyrazol-3(2*H*)-one

Crystal data

$C_{22}H_{19}N_3O_2$	$F(000)$ = 752
M_r = 357.40	D_x = 1.315 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1592 reflections
a = 8.0636 (7) Å	θ = 2.5–25.1°
b = 7.4407 (6) Å	μ = 0.09 mm^{-1}
c = 30.169 (3) Å	T = 295 K
β = 94.329 (2)°	Plane, yellow
V = 1804.9 (3) Å^3	0.23 × 0.10 × 0.02 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	3942 independent reflections
Radiation source: fine-focus sealed tube	2403 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.050
φ and ω scans	θ_{max} = 27.0°, θ_{min} = 1.4°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	h = −10→10
T_{min} = 0.981, T_{max} = 0.998	k = −9→9
14746 measured reflections	l = −37→38

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.060$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.147$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.2473P]$ where $P = (F_o^2 + 2F_c^2)/3$
3942 reflections	$(\Delta/\sigma)_{max} < 0.001$
252 parameters	$\Delta\rho_{max} = 0.14$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

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 $F^2 > 2$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}	Occ. (<1)
O1	0.5537 (2)	0.63818 (19)	0.63468 (5)	0.0518 (4)	
O2	0.2652 (2)	0.2809 (2)	0.48208 (5)	0.0529 (5)	
H2	0.3246	0.2763	0.5053	0.079*	
N1	0.6496 (2)	0.3617 (2)	0.66126 (6)	0.0430 (5)	
N2	0.6208 (2)	0.1816 (2)	0.64902 (6)	0.0440 (5)	
N3	0.4118 (2)	0.3947 (2)	0.55528 (6)	0.0391 (4)	
C1	0.6836 (3)	0.4067 (3)	0.70681 (7)	0.0416 (5)	
C2	0.7888 (3)	0.5480 (3)	0.71763 (8)	0.0546 (6)	
H2A	0.8380	0.6109	0.6954	0.066*	
C3	0.8210 (3)	0.5961 (4)	0.76159 (9)	0.0662 (8)	
H3	0.8910	0.6926	0.7690	0.079*	
C4	0.7503 (4)	0.5023 (4)	0.79433 (9)	0.0699 (8)	
H4	0.7726	0.5351	0.8239	0.084*	
C5	0.6471 (4)	0.3607 (4)	0.78370 (8)	0.0633 (7)	
H5	0.6007	0.2963	0.8061	0.076*	
C6	0.6115 (3)	0.3130 (3)	0.73982 (8)	0.0526 (6)	
H6	0.5393	0.2182	0.7325	0.063*	
C7	0.5673 (3)	0.4746 (3)	0.62969 (7)	0.0383 (5)	

C8	0.5054 (3)	0.3554 (3)	0.59459 (7)	0.0366 (5)	
C9	0.5442 (3)	0.1841 (3)	0.60707 (7)	0.0408 (5)	
C10	0.513 (2)	0.016 (3)	0.5821 (7)	0.0599 (11)	0.68 (4)
H10A	0.4444	0.0406	0.5554	0.090*	0.68 (4)
H10B	0.4572	−0.0679	0.6001	0.090*	0.68 (4)
H10C	0.6168	−0.0343	0.5746	0.090*	0.68 (4)
C10'	0.501 (5)	0.018 (7)	0.5804 (16)	0.0599 (11)	0.32 (4)
H10D	0.3824	0.0105	0.5746	0.090*	0.32 (4)
H10E	0.5402	−0.0861	0.5969	0.090*	0.32 (4)
H10F	0.5525	0.0231	0.5528	0.090*	0.32 (4)
C11	0.7486 (3)	0.0508 (3)	0.66333 (8)	0.0575 (7)	
H11A	0.7126	−0.0673	0.6541	0.086*	
H11B	0.7672	0.0539	0.6951	0.086*	
H11C	0.8501	0.0797	0.6502	0.086*	
C12	0.3597 (3)	0.5544 (3)	0.54508 (7)	0.0412 (5)	
H12	0.3903	0.6499	0.5638	0.049*	
C13	0.2085 (3)	0.4488 (3)	0.47564 (7)	0.0404 (5)	
C14	0.2546 (3)	0.5875 (3)	0.50496 (7)	0.0382 (5)	
C15	0.1916 (3)	0.7620 (3)	0.49565 (8)	0.0496 (6)	
H15	0.2230	0.8555	0.5150	0.059*	
C16	0.0877 (3)	0.7968 (3)	0.45970 (8)	0.0562 (7)	
H16	0.0489	0.9132	0.4546	0.067*	
C17	0.0366 (3)	0.6577 (3)	0.42957 (7)	0.0497 (6)	
C18	0.0988 (3)	0.4819 (3)	0.43735 (7)	0.0444 (6)	
C19	0.0465 (3)	0.3427 (4)	0.40779 (8)	0.0570 (7)	
H19	0.0867	0.2266	0.4126	0.068*	
C20	−0.0626 (4)	0.3776 (5)	0.37217 (9)	0.0740 (9)	
H20	−0.0968	0.2849	0.3529	0.089*	
C21	−0.1237 (3)	0.5509 (5)	0.36419 (9)	0.0755 (9)	
H21	−0.1976	0.5730	0.3396	0.091*	
C22	−0.0758 (3)	0.6875 (4)	0.39216 (8)	0.0660 (8)	
H22	−0.1178	0.8024	0.3866	0.079*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0777 (12)	0.0296 (9)	0.0462 (10)	0.0008 (8)	−0.0081 (8)	−0.0028 (7)
O2	0.0680 (12)	0.0423 (10)	0.0465 (10)	0.0065 (8)	−0.0091 (8)	−0.0060 (8)
N1	0.0618 (12)	0.0285 (10)	0.0368 (10)	−0.0024 (9)	−0.0082 (9)	−0.0010 (8)
N2	0.0610 (13)	0.0258 (10)	0.0432 (11)	0.0017 (9)	−0.0086 (9)	0.0001 (8)
N3	0.0464 (11)	0.0342 (10)	0.0362 (10)	0.0010 (8)	0.0006 (8)	−0.0002 (8)
C1	0.0508 (14)	0.0365 (12)	0.0359 (12)	0.0005 (10)	−0.0071 (10)	0.0004 (10)
C2	0.0617 (16)	0.0486 (15)	0.0519 (15)	−0.0066 (12)	−0.0064 (13)	−0.0003 (12)
C3	0.0732 (19)	0.0597 (18)	0.0620 (18)	−0.0080 (15)	−0.0195 (15)	−0.0113 (15)
C4	0.095 (2)	0.071 (2)	0.0398 (15)	0.0118 (17)	−0.0171 (15)	−0.0065 (14)
C5	0.088 (2)	0.0609 (17)	0.0401 (15)	0.0049 (16)	0.0006 (14)	0.0079 (13)
C6	0.0630 (16)	0.0472 (15)	0.0461 (15)	−0.0066 (12)	−0.0055 (12)	0.0051 (11)
C7	0.0456 (13)	0.0316 (12)	0.0372 (12)	−0.0003 (10)	−0.0001 (10)	0.0023 (9)

C8	0.0416 (12)	0.0305 (11)	0.0372 (12)	−0.0015 (10)	−0.0014 (10)	−0.0018 (9)
C9	0.0476 (13)	0.0349 (12)	0.0395 (13)	−0.0029 (10)	0.0003 (10)	−0.0028 (9)
C10	0.091 (3)	0.0295 (14)	0.057 (2)	0.000 (2)	−0.013 (3)	−0.0056 (15)
C10'	0.091 (3)	0.0295 (14)	0.057 (2)	0.000 (2)	−0.013 (3)	−0.0056 (15)
C11	0.0692 (17)	0.0410 (14)	0.0601 (16)	0.0112 (12)	−0.0110 (13)	0.0041 (12)
C12	0.0460 (13)	0.0374 (13)	0.0401 (13)	−0.0028 (10)	0.0019 (10)	−0.0029 (10)
C13	0.0434 (13)	0.0402 (13)	0.0379 (12)	−0.0006 (10)	0.0048 (10)	0.0023 (10)
C14	0.0410 (12)	0.0358 (12)	0.0377 (12)	−0.0015 (10)	0.0031 (10)	0.0029 (9)
C15	0.0563 (15)	0.0399 (13)	0.0519 (15)	0.0023 (11)	−0.0003 (12)	0.0023 (11)
C16	0.0575 (16)	0.0499 (15)	0.0604 (17)	0.0077 (13)	0.0001 (13)	0.0140 (13)
C17	0.0417 (14)	0.0658 (17)	0.0417 (14)	0.0009 (12)	0.0027 (11)	0.0149 (12)
C18	0.0413 (13)	0.0564 (15)	0.0357 (12)	−0.0070 (11)	0.0043 (10)	0.0045 (11)
C19	0.0570 (16)	0.0698 (18)	0.0435 (14)	−0.0126 (13)	−0.0011 (12)	−0.0044 (13)
C20	0.0669 (19)	0.105 (3)	0.0485 (17)	−0.0213 (18)	−0.0042 (14)	−0.0058 (17)
C21	0.0522 (17)	0.131 (3)	0.0420 (16)	−0.0102 (19)	−0.0091 (13)	0.0173 (18)
C22	0.0512 (16)	0.093 (2)	0.0533 (17)	0.0027 (15)	−0.0003 (13)	0.0269 (16)

Geometric parameters (Å, °)

O1—C7	1.232 (2)	C10—H10C	0.9600
O2—C13	1.340 (2)	C10'—H10D	0.9600
O2—H2	0.8200	C10'—H10E	0.9600
N1—C7	1.399 (3)	C10'—H10F	0.9600
N1—N2	1.405 (2)	C11—H11A	0.9600
N1—C1	1.421 (3)	C11—H11B	0.9600
N2—C9	1.366 (3)	C11—H11C	0.9600
N2—C11	1.459 (3)	C12—C14	1.445 (3)
N3—C12	1.290 (3)	C12—H12	0.9300
N3—C8	1.388 (3)	C13—C14	1.391 (3)
C1—C2	1.375 (3)	C13—C18	1.422 (3)
C1—C6	1.380 (3)	C14—C15	1.415 (3)
C2—C3	1.379 (3)	C15—C16	1.345 (3)
C2—H2A	0.9300	C15—H15	0.9300
C3—C4	1.369 (4)	C16—C17	1.418 (3)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.365 (4)	C17—C22	1.411 (3)
C4—H4	0.9300	C17—C18	1.414 (3)
C5—C6	1.380 (3)	C18—C19	1.410 (3)
C5—H5	0.9300	C19—C20	1.362 (3)
C6—H6	0.9300	C19—H19	0.9300
C7—C8	1.441 (3)	C20—C21	1.395 (4)
C8—C9	1.359 (3)	C20—H20	0.9300
C9—C10	1.47 (3)	C21—C22	1.358 (4)
C9—C10'	1.50 (6)	C21—H21	0.9300
C10—H10A	0.9600	C22—H22	0.9300
C10—H10B	0.9600		
C13—O2—H2	109.5	H10D—C10'—H10E	109.5
C7—N1—N2	109.49 (16)	C9—C10'—H10F	109.5
C7—N1—C1	124.31 (17)	H10D—C10'—H10F	109.5

N2—N1—C1	119.69 (16)	H10E—C10'—H10F	109.5
C9—N2—N1	106.54 (16)	N2—C11—H11A	109.5
C9—N2—C11	122.92 (18)	N2—C11—H11B	109.5
N1—N2—C11	117.36 (18)	H11A—C11—H11B	109.5
C12—N3—C8	123.00 (18)	N2—C11—H11C	109.5
C2—C1—C6	120.1 (2)	H11A—C11—H11C	109.5
C2—C1—N1	118.7 (2)	H11B—C11—H11C	109.5
C6—C1—N1	121.2 (2)	N3—C12—C14	121.1 (2)
C1—C2—C3	119.7 (2)	N3—C12—H12	119.4
C1—C2—H2A	120.2	C14—C12—H12	119.4
C3—C2—H2A	120.2	O2—C13—C14	121.88 (19)
C4—C3—C2	120.2 (3)	O2—C13—C18	117.6 (2)
C4—C3—H3	119.9	C14—C13—C18	120.5 (2)
C2—C3—H3	119.9	C13—C14—C15	118.7 (2)
C5—C4—C3	120.2 (2)	C13—C14—C12	121.15 (19)
C5—C4—H4	119.9	C15—C14—C12	120.1 (2)
C3—C4—H4	119.9	C16—C15—C14	122.0 (2)
C4—C5—C6	120.2 (3)	C16—C15—H15	119.0
C4—C5—H5	119.9	C14—C15—H15	119.0
C6—C5—H5	119.9	C15—C16—C17	120.7 (2)
C5—C6—C1	119.6 (2)	C15—C16—H16	119.7
C5—C6—H6	120.2	C17—C16—H16	119.7
C1—C6—H6	120.2	C22—C17—C18	118.5 (2)
O1—C7—N1	123.52 (19)	C22—C17—C16	122.4 (2)
O1—C7—C8	131.94 (19)	C18—C17—C16	119.1 (2)
N1—C7—C8	104.51 (17)	C19—C18—C17	119.3 (2)
C9—C8—N3	122.23 (19)	C19—C18—C13	121.6 (2)
C9—C8—C7	108.31 (19)	C17—C18—C13	119.1 (2)
N3—C8—C7	129.33 (18)	C20—C19—C18	120.2 (3)
C8—C9—N2	110.37 (18)	C20—C19—H19	119.9
C8—C9—C10	128.9 (9)	C18—C19—H19	119.9
N2—C9—C10	120.7 (9)	C19—C20—C21	120.8 (3)
C8—C9—C10'	125.8 (18)	C19—C20—H20	119.6
N2—C9—C10'	123.7 (18)	C21—C20—H20	119.6
C10—C9—C10'	4(2)	C22—C21—C20	120.3 (3)
C9—C10—H10A	109.5	C22—C21—H21	119.9
C9—C10—H10B	109.5	C20—C21—H21	119.9
C9—C10—H10C	109.5	C21—C22—C17	121.0 (3)
C9—C10'—H10D	109.5	C21—C22—H22	119.5
C9—C10'—H10E	109.5	C17—C22—H22	119.5
C7—N1—N2—C9	9.2 (2)	C11—N2—C9—C8	−147.1 (2)
C1—N1—N2—C9	162.14 (19)	N1—N2—C9—C10	173.1 (9)
C7—N1—N2—C11	151.62 (19)	C11—N2—C9—C10	33.4 (9)
C1—N1—N2—C11	−55.5 (3)	N1—N2—C9—C10'	176.1 (18)
C7—N1—C1—C2	−64.1 (3)	C11—N2—C9—C10'	36.4 (18)
N2—N1—C1—C2	147.2 (2)	C8—N3—C12—C14	−176.44 (19)
C7—N1—C1—C6	115.0 (2)	O2—C13—C14—C15	179.1 (2)
N2—N1—C1—C6	−33.6 (3)	C18—C13—C14—C15	−0.7 (3)
C6—C1—C2—C3	−0.4 (4)	O2—C13—C14—C12	−3.1 (3)

N1—C1—C2—C3	178.8 (2)	C18—C13—C14—C12	177.09 (19)
C1—C2—C3—C4	0.8 (4)	N3—C12—C14—C13	−1.2 (3)
C2—C3—C4—C5	−0.1 (4)	N3—C12—C14—C15	176.6 (2)
C3—C4—C5—C6	−1.0 (4)	C13—C14—C15—C16	0.7 (3)
C4—C5—C6—C1	1.4 (4)	C12—C14—C15—C16	−177.1 (2)
C2—C1—C6—C5	−0.7 (4)	C14—C15—C16—C17	0.2 (4)
N1—C1—C6—C5	−179.9 (2)	C15—C16—C17—C22	178.2 (2)
N2—N1—C7—O1	170.7 (2)	C15—C16—C17—C18	−1.1 (3)
C1—N1—C7—O1	19.4 (3)	C22—C17—C18—C19	−0.2 (3)
N2—N1—C7—C8	−7.4 (2)	C16—C17—C18—C19	179.1 (2)
C1—N1—C7—C8	−158.8 (2)	C22—C17—C18—C13	−178.2 (2)
C12—N3—C8—C9	172.8 (2)	C16—C17—C18—C13	1.1 (3)
C12—N3—C8—C7	−2.5 (3)	O2—C13—C18—C19	2.0 (3)
O1—C7—C8—C9	−175.0 (2)	C14—C13—C18—C19	−178.2 (2)
N1—C7—C8—C9	2.9 (2)	O2—C13—C18—C17	179.96 (19)
O1—C7—C8—N3	0.8 (4)	C14—C13—C18—C17	−0.2 (3)
N1—C7—C8—N3	178.7 (2)	C17—C18—C19—C20	0.0 (3)
N3—C8—C9—N2	−173.34 (19)	C13—C18—C19—C20	178.0 (2)
C7—C8—C9—N2	2.8 (3)	C18—C19—C20—C21	0.3 (4)
N3—C8—C9—C10	6.1 (10)	C19—C20—C21—C22	−0.5 (4)
C7—C8—C9—C10	−177.7 (9)	C20—C21—C22—C17	0.3 (4)
N3—C8—C9—C10'	3.1 (18)	C18—C17—C22—C21	0.1 (4)
C7—C8—C9—C10'	179.3 (18)	C16—C17—C22—C21	−179.2 (2)
N1—N2—C9—C8	−7.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N3	0.82	1.84	2.569 (2)	148.

Fig. 1

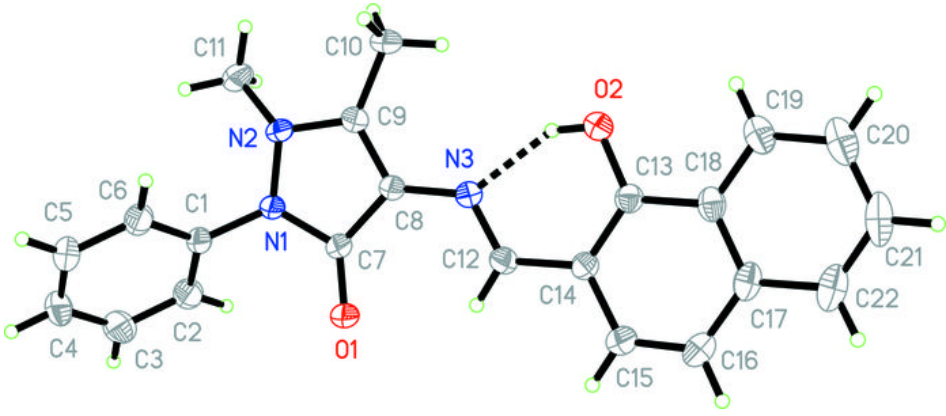
C11
C10
O2
C19
C9
N2
N3
C20
C13
C18
C6
C1
C8
C5
N1
C7
C12
C21
C14
C22
C17
C2
C4
C3
O1
C16
C15

Fig. 2