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1-[(2-Hydroxyphenyl)(pyrrolidin-1-yl)-methyl]naphthalen-2-ol *N*,*N*-dimethylformamide monosolvate

Wen-xiang Wang

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 211189, People's Republic of China
Correspondence e-mail: wxwang@seu.edu.cn

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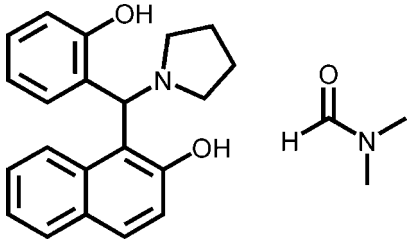
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.066; wR factor = 0.184; data-to-parameter ratio = 18.6.

The title compound, $C_{21}H_{21}NO_2\cdot C_3H_7NO$, was synthesized by solvent-free one-pot three-component reaction of naphthalen-2-ol, 2-hydroxybenzaldehyde and pyrrolidine. The dihedral angle between the naphthalene ring system and the benzene ring is 77.74 (6)°. The pyrrolidine ring assumes an envelope conformation. An intramolecular O—H···N and an intermolecular O—H···O hydrogen bond are observed.

Related literature

For background to Betti-type reactions, see: Pu & Yu (2001); Yuan (2005). For ring puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{21}H_{21}NO_2\cdot C_3H_7NO$
M_r = 392.48
Monoclinic, $P2_1/n$
a = 13.675 (3) Å
b = 9.6518 (19) Å
c = 16.505 (3) Å
β = 102.84 (3)°
V = 2123.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.25 × 0.22 × 0.20 mm

Data collection

Rigaku Mercury2 (2 × 2 bin mode) diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.963, T_{max} = 0.989
21445 measured reflections
4874 independent reflections
2223 reflections with $I > 2\sigma(I)$
R_{int} = 0.100

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.184
S = 1.03
4874 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 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—H2A···N1	0.82	1.88	2.565 (3)	140
O3—H3A···O1	0.82	1.86	2.678 (3)	173

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97*.

This work was supported by Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2712).

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Pu, L. & Yu, H. B. (2001). *Chem. Rev.* **101**, 757–824.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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Yuan, C.-L. (2005). *Acta Cryst.* E**61**, o1182–o1183.

supplementary materials

Acta Cryst. (2012). E**68**, o884 [doi:10.1107/S1600536812007787]

1-[(2-Hydroxyphenyl)(pyrrolidin-1-yl)methyl]naphthalen-2-ol *N*,*N*-dimethylformamide monosolvate

Wen-xiang Wang

Comment

The so-called Betti base derivatives, which can be synthesized by many ways (Pu & Yu, 2001; Yuan, 2005), have been of great interest in coordination chemistry. Herein the crystal structure of one such compounds obtained by solvent free, one-pot, three-component domino reaction of naphthalen-2-ol, 2-hydroxybenzaldehyde and pyrrolidine is reported.

In the molecule of the title compound (Fig. 1) the bond lengths and angles are well within the expected range. The dihedral angle between the naphthalene ring system and the benzene ring is 77.74 (6)°. The pyrrolidine ring adopts an envelope conformation, with puckering parameters (Cremer & Pople, 1975) Q = 0.408 (3) Å and φ = 170.7 (5)°. An intramolecular O—H···N hydrogen bond (Table 1) stabilizes the molecular conformation. In the crystal structure, the molecules interact *via* an intermolecular O—H···O hydrogen bond (Table 1).

Experimental

A dry 50 ml flask was charged with 2-hydroxybenzaldehyde (10 mmol), naphthalen-2-ol (10 mmol) and pyrrolidine (10 mmol). The mixture was stirred at 100°C for 5 h, then ethanol (15 ml) was added. After refluxing for 30 minutes, the precipitate was filtrated out, washed with ethanol for 3 times and purified by recrystallization from a mixed solution of dichloromethane, methanol and DMF (30:8:10 *v*/*v*/*v*) to give crystals of the title compound suitable for X-ray analysis.

Refinement

All H atoms were calculated geometrically and refined using a riding model, with C—H = 0.93–0.98 Å, O—H = 0.82 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C, O) for methyl and hydroxy H atoms.

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); 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: *SHELXL97* (Sheldrick, 2008).

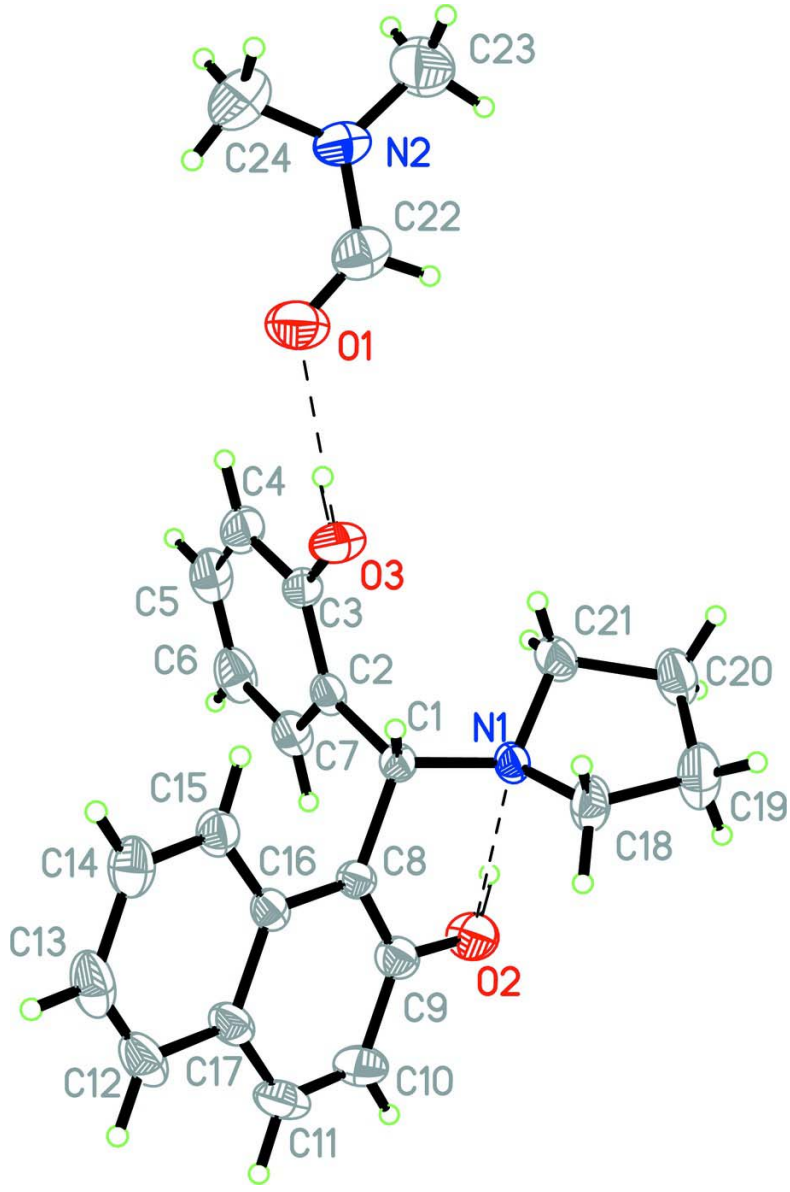

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Intra- and intermolecular hydrogen bonds are shown as dashed lines.

1-[(2-Hydroxyphenyl)(pyrrolidin-1-yl)methyl]naphthalen-2-ol *N*,*N*-dimethylformamide monosolvate

Crystal data

$C_{21}H_{21}NO_2{\cdot}C_3H_7NO$
M_r = 392.48
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 13.675 (3) Å
b = 9.6518 (19) Å
c = 16.505 (3) Å
β = 102.84 (3)°
V = 2123.9 (7) Å^3
Z = 4
F(000) = 840
D_x = 1.227 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3860 reflections
θ = 2.3–27.5°
μ = 0.08 mm^{-1}

T = 293 K
Prism, colourless

0.25 × 0.22 × 0.20 mm

Data collection

Rigaku Mercury2 (2x2 bin mode) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD Profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.963, T_{max} = 0.989

21445 measured reflections
4874 independent reflections
2223 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −17→17
k = −12→12
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.184
S = 1.03
4874 reflections
262 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.0577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O2	0.86622 (14)	0.7318 (2)	1.01567 (12)	0.0696 (6)
H2A	0.8484	0.7762	1.0522	0.104*
O3	0.59379 (14)	1.15741 (19)	1.04615 (12)	0.0620 (6)
H3A	0.5723	1.2281	1.0636	0.093*
N1	0.73008 (15)	0.8368 (2)	1.08164 (11)	0.0421 (5)
C8	0.72196 (18)	0.8476 (3)	0.93131 (15)	0.0396 (6)
C16	0.66100 (19)	0.8697 (2)	0.84998 (15)	0.0414 (6)
C15	0.5776 (2)	0.9595 (3)	0.83292 (16)	0.0508 (7)
H15A	0.5614	1.0099	0.8761	0.061*
C1	0.69749 (19)	0.9227 (2)	1.00552 (14)	0.0397 (6)
H1A	0.6245	0.9335	0.9952	0.048*
C9	0.8026 (2)	0.7581 (3)	0.94091 (17)	0.0508 (7)
C17	0.6846 (2)	0.7971 (3)	0.78094 (16)	0.0518 (7)
C7	0.8420 (2)	1.0914 (3)	1.00816 (15)	0.0540 (7)

H7A	0.8790	1.0181	0.9940	0.065*
C2	0.7445 (2)	1.0671 (3)	1.01694 (14)	0.0422 (6)
C3	0.6901 (2)	1.1791 (3)	1.03819 (15)	0.0485 (7)
C10	0.8248 (2)	0.6866 (3)	0.8726 (2)	0.0648 (9)
H10A	0.8794	0.6267	0.8806	0.078*
C14	0.5201 (2)	0.9739 (3)	0.75428 (19)	0.0639 (8)
H14A	0.4650	1.0330	0.7451	0.077*
C5	0.8301 (3)	1.3300 (3)	1.04007 (18)	0.0706 (10)
H5A	0.8585	1.4179	1.0477	0.085*
C12	0.6236 (3)	0.8158 (4)	0.70070 (17)	0.0679 (9)
H12A	0.6392	0.7685	0.6561	0.082*
C13	0.5429 (3)	0.9011 (4)	0.68713 (19)	0.0734 (10)
H13A	0.5030	0.9113	0.6340	0.088*
C18	0.6664 (2)	0.7135 (3)	1.08117 (17)	0.0628 (9)
H18A	0.6778	0.6458	1.0409	0.075*
H18B	0.5958	0.7382	1.0690	0.075*
C11	0.7670 (2)	0.7048 (3)	0.7955 (2)	0.0651 (9)
H11A	0.7817	0.6557	0.7512	0.078*
C21	0.7284 (2)	0.9051 (3)	1.16070 (15)	0.0596 (8)
H21A	0.6674	0.9587	1.1564	0.072*
H21B	0.7858	0.9658	1.1778	0.072*
C4	0.7332 (3)	1.3106 (3)	1.04918 (17)	0.0636 (9)
H4A	0.6967	1.3851	1.0626	0.076*
C20	0.7325 (3)	0.7847 (3)	1.22155 (18)	0.0734 (10)
H20A	0.8000	0.7729	1.2547	0.088*
H20B	0.6876	0.8013	1.2585	0.088*
C19	0.7004 (3)	0.6593 (4)	1.16925 (18)	0.0850 (11)
H19A	0.6458	0.6123	1.1867	0.102*
H19B	0.7558	0.5951	1.1732	0.102*
C6	0.8852 (2)	1.2218 (4)	1.01990 (18)	0.0667 (9)
H6A	0.9507	1.2357	1.0142	0.080*
N2	0.45935 (18)	1.5046 (3)	1.20142 (17)	0.0669 (7)
O1	0.50827 (19)	1.3762 (2)	1.10246 (16)	0.0883 (8)
C22	0.5038 (2)	1.3964 (4)	1.1737 (3)	0.0729 (9)
H22A	0.5340	1.3312	1.2128	0.087*
C23	0.4627 (3)	1.5224 (4)	1.2890 (2)	0.1042 (13)
H23A	0.5010	1.4487	1.3198	0.156*
H23C	0.3957	1.5206	1.2979	0.156*
H23D	0.4934	1.6096	1.3073	0.156*
C24	0.4053 (3)	1.6046 (4)	1.1449 (3)	0.1163 (15)
H24C	0.4091	1.5807	1.0892	0.174*
H24D	0.4342	1.6946	1.1585	0.174*
H24A	0.3364	1.6056	1.1491	0.174*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0655 (13)	0.0786 (15)	0.0595 (14)	0.0240 (11)	0.0028 (11)	−0.0003 (11)
O3	0.0592 (13)	0.0468 (12)	0.0864 (15)	−0.0006 (10)	0.0300 (11)	−0.0142 (10)
N1	0.0557 (13)	0.0384 (12)	0.0315 (11)	−0.0083 (11)	0.0085 (10)	0.0001 (9)

C8	0.0430 (15)	0.0391 (15)	0.0381 (15)	−0.0027 (12)	0.0116 (12)	−0.0032 (11)
C16	0.0480 (16)	0.0391 (15)	0.0385 (15)	−0.0134 (13)	0.0125 (12)	−0.0023 (11)
C15	0.0569 (18)	0.0499 (18)	0.0439 (16)	−0.0025 (14)	0.0077 (14)	0.0028 (13)
C1	0.0474 (15)	0.0372 (15)	0.0346 (14)	−0.0019 (12)	0.0090 (11)	−0.0002 (11)
C9	0.0529 (17)	0.0527 (18)	0.0465 (17)	0.0006 (15)	0.0105 (14)	−0.0028 (14)
C17	0.0662 (19)	0.0539 (18)	0.0368 (16)	−0.0154 (15)	0.0146 (14)	−0.0106 (13)
C7	0.0608 (19)	0.0584 (19)	0.0433 (16)	−0.0102 (15)	0.0130 (14)	0.0009 (14)
C2	0.0505 (16)	0.0446 (17)	0.0309 (14)	−0.0086 (13)	0.0079 (12)	0.0009 (11)
C3	0.0684 (19)	0.0371 (16)	0.0394 (15)	−0.0053 (14)	0.0108 (14)	−0.0048 (12)
C10	0.064 (2)	0.067 (2)	0.069 (2)	0.0125 (16)	0.0265 (18)	−0.0124 (17)
C14	0.0599 (19)	0.069 (2)	0.056 (2)	−0.0037 (16)	−0.0010 (16)	0.0133 (16)
C5	0.102 (3)	0.054 (2)	0.0509 (19)	−0.035 (2)	0.0074 (19)	−0.0035 (15)
C12	0.093 (3)	0.075 (2)	0.0352 (18)	−0.031 (2)	0.0135 (17)	−0.0097 (15)
C13	0.087 (3)	0.084 (3)	0.0406 (19)	−0.026 (2)	−0.0045 (17)	0.0112 (17)
C18	0.084 (2)	0.0501 (18)	0.0505 (18)	−0.0236 (16)	0.0062 (15)	0.0073 (14)
C11	0.075 (2)	0.066 (2)	0.064 (2)	−0.0113 (18)	0.0362 (18)	−0.0245 (16)
C21	0.085 (2)	0.0585 (19)	0.0348 (15)	−0.0073 (17)	0.0131 (15)	−0.0049 (14)
C4	0.093 (3)	0.0411 (18)	0.0552 (19)	−0.0131 (17)	0.0138 (17)	−0.0047 (14)
C20	0.094 (2)	0.080 (2)	0.0452 (18)	−0.0161 (19)	0.0134 (17)	0.0097 (17)
C19	0.114 (3)	0.077 (2)	0.058 (2)	−0.030 (2)	0.006 (2)	0.0151 (19)
C6	0.071 (2)	0.077 (2)	0.0511 (19)	−0.030 (2)	0.0121 (16)	0.0032 (17)
N2	0.0663 (17)	0.0551 (17)	0.082 (2)	0.0039 (14)	0.0228 (15)	−0.0131 (15)
O1	0.1021 (19)	0.0734 (17)	0.101 (2)	−0.0031 (13)	0.0463 (16)	−0.0201 (15)
C22	0.065 (2)	0.056 (2)	0.097 (3)	−0.0124 (17)	0.018 (2)	−0.010 (2)
C23	0.136 (4)	0.094 (3)	0.084 (3)	−0.007 (3)	0.028 (2)	−0.020 (2)
C24	0.130 (4)	0.099 (3)	0.119 (3)	0.048 (3)	0.027 (3)	0.023 (3)

Geometric parameters (Å, º)

O2—C9	1.366 (3)	C5—H5A	0.9300
O2—H2A	0.8200	C12—C13	1.356 (4)
O3—C3	1.368 (3)	C12—H12A	0.9300
O3—H3A	0.8200	C13—H13A	0.9300
N1—C21	1.467 (3)	C18—C19	1.518 (4)
N1—C18	1.473 (3)	C18—H18A	0.9700
N1—C1	1.489 (3)	C18—H18B	0.9700
C8—C9	1.382 (3)	C11—H11A	0.9300
C8—C16	1.430 (3)	C21—C20	1.529 (4)
C8—C1	1.523 (3)	C21—H21A	0.9700
C16—C15	1.410 (4)	C21—H21B	0.9700
C16—C17	1.434 (3)	C4—H4A	0.9300
C15—C14	1.367 (4)	C20—C19	1.495 (4)
C15—H15A	0.9300	C20—H20A	0.9700
C1—C2	1.529 (3)	C20—H20B	0.9700
C1—H1A	0.9800	C19—H19A	0.9700
C9—C10	1.411 (4)	C19—H19B	0.9700
C17—C12	1.411 (4)	C6—H6A	0.9300
C17—C11	1.415 (4)	N2—C22	1.339 (4)
C7—C6	1.386 (4)	N2—C24	1.428 (4)
C7—C2	1.393 (4)	N2—C23	1.446 (4)

C7—H7A	0.9300	O1—C22	1.207 (4)
C2—C3	1.400 (4)	C22—H22A	0.9300
C3—C4	1.394 (4)	C23—H23A	0.9600
C10—C11	1.351 (4)	C23—H23C	0.9600
C10—H10A	0.9300	C23—H23D	0.9600
C14—C13	1.404 (4)	C24—H24C	0.9600
C14—H14A	0.9300	C24—H24D	0.9600
C5—C6	1.372 (4)	C24—H24A	0.9600
C5—C4	1.379 (4)		

C9—O2—H2A	109.5	N1—C18—H18A	111.2
C3—O3—H3A	109.5	C19—C18—H18A	111.2
C21—N1—C18	104.0 (2)	N1—C18—H18B	111.2
C21—N1—C1	116.0 (2)	C19—C18—H18B	111.2
C18—N1—C1	112.29 (19)	H18A—C18—H18B	109.2
C9—C8—C16	119.0 (2)	C10—C11—C17	121.3 (3)
C9—C8—C1	121.2 (2)	C10—C11—H11A	119.4
C16—C8—C1	119.8 (2)	C17—C11—H11A	119.4
C15—C16—C8	123.8 (2)	N1—C21—C20	103.7 (2)
C15—C16—C17	117.1 (2)	N1—C21—H21A	111.0
C8—C16—C17	119.1 (2)	C20—C21—H21A	111.0
C14—C15—C16	121.4 (3)	N1—C21—H21B	111.0
C14—C15—H15A	119.3	C20—C21—H21B	111.0
C16—C15—H15A	119.3	H21A—C21—H21B	109.0
N1—C1—C8	109.61 (19)	C5—C4—C3	119.9 (3)
N1—C1—C2	111.41 (19)	C5—C4—H4A	120.0
C8—C1—C2	111.8 (2)	C3—C4—H4A	120.0
N1—C1—H1A	107.9	C19—C20—C21	105.9 (2)
C8—C1—H1A	107.9	C19—C20—H20A	110.6
C2—C1—H1A	107.9	C21—C20—H20A	110.6
O2—C9—C8	123.5 (2)	C19—C20—H20B	110.6
O2—C9—C10	115.1 (3)	C21—C20—H20B	110.6
C8—C9—C10	121.5 (3)	H20A—C20—H20B	108.7
C12—C17—C11	121.5 (3)	C20—C19—C18	105.2 (3)
C12—C17—C16	119.6 (3)	C20—C19—H19A	110.7
C11—C17—C16	118.9 (3)	C18—C19—H19A	110.7
C6—C7—C2	121.8 (3)	C20—C19—H19B	110.7
C6—C7—H7A	119.1	C18—C19—H19B	110.7
C2—C7—H7A	119.1	H19A—C19—H19B	108.8
C7—C2—C3	118.1 (2)	C5—C6—C7	119.0 (3)
C7—C2—C1	121.7 (2)	C5—C6—H6A	120.5
C3—C2—C1	120.2 (2)	C7—C6—H6A	120.5
O3—C3—C4	121.0 (3)	C22—N2—C24	120.8 (3)
O3—C3—C2	118.8 (2)	C22—N2—C23	121.2 (3)
C4—C3—C2	120.1 (3)	C24—N2—C23	118.0 (3)
C11—C10—C9	120.3 (3)	O1—C22—N2	125.8 (4)
C11—C10—H10A	119.9	O1—C22—H22A	117.1
C9—C10—H10A	119.9	N2—C22—H22A	117.1
C15—C14—C13	121.1 (3)	N2—C23—H23A	109.5

C15—C14—H14A	119.4	N2—C23—H23C	109.5
C13—C14—H14A	119.4	H23A—C23—H23C	109.5
C6—C5—C4	121.1 (3)	N2—C23—H23D	109.5
C6—C5—H5A	119.5	H23A—C23—H23D	109.5
C4—C5—H5A	119.5	H23C—C23—H23D	109.5
C13—C12—C17	121.5 (3)	N2—C24—H24C	109.5
C13—C12—H12A	119.3	N2—C24—H24D	109.5
C17—C12—H12A	119.3	H24C—C24—H24D	109.5
C12—C13—C14	119.3 (3)	N2—C24—H24A	109.5
C12—C13—H13A	120.4	H24C—C24—H24A	109.5
C14—C13—H13A	120.4	H24D—C24—H24A	109.5
N1—C18—C19	102.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1	0.82	1.88	2.565 (3)	140
O3—H3*A*···O1	0.82	1.86	2.678 (3)	173