Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2-[(2-Aminophenyl)iminomethyl]-4,6-di-*tert*-butylphenol

Liqin Ding,[a]* Xingqiang Lü,[b] Shunsheng Zhao[c] and Yuqin Zhu[a]

[a]School of Chemistry and Chemical Engineering, Xi'an Shiyou University, Xi'an 710065, Shaanxi, People's Republic of China, [b]College of Chemical Engineering, Northwest University, Xi'an 710069, Shaanxi, People's Republic of China, and [c]College of Chemistry and Chemical Engineering, Xi'an University of Science and Technology, Xi'an 710054, Shaanxi, People's Republic of China
Correspondence e-mail: lqding@xsyu.edu.cn

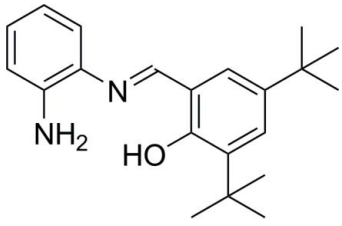
Received 1 August 2012; accepted 8 September 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; R factor = 0.044; wR factor = 0.129; data-to-parameter ratio = 6.2.

In the title compound, $C_{21}H_{28}N_2O$, the dihedral angle between the rings is 35.2 (2)°. A weak intramolecular O—H···N hydrogen bond is observed between the O—H H atom and the imine N atom. In the crystal, molecules are linked by additional intermolecular N—H···O hydrogen bonding, resulting in a wave-like chain along the *b*-axis direction.

Related literature

For related structures, see: Kochem *et al.* (2010); Belmonte *et al.* (2010); Liu *et al.* (2010). Details of the synthesis can be found in Muñoz-Hernández *et al.* (2000).



Experimental

Crystal data

$C_{21}H_{28}N_2O$
M_r = 324.45
Monoclinic, $P2_1$
a = 10.898 (5) Å
b = 6.230 (3) Å
c = 15.095 (8) Å
β = 108.928 (6)°
V = 969.5 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 296 K
0.38 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.979, T_{max} = 0.986
3718 measured reflections
1357 independent reflections
1224 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 22.2°

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.10
1357 reflections
218 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.82	1.87	2.608 (4)	149
N2—H2A···O1^{i}	0.86	2.54	3.342 (4)	155

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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* and local programs.

This project was Supported Scientific Research Program Funded by Shaanxi Provincial Education Department (program Nos. 11 J K0605, 11 J K0588, 11 J K0606) and the Natural Science Basic Research Plan in Shaanxi Province of China (program Nos. 2012JM2011, 2011JQ2011).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2289).

References

Belmonte, M. M., Wezenberg, S. J., Haak, R. M., Anselmo, D., Escudero-Adán, E. C., Benet-Buchholza, J. & Kleij, A. W. (2010). *Dalton Trans.* **39**, 4541–4550.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kochem, A., Orio, M., Jarjayes, O., Neeseb, F. & Thomas, F. (2010). *Chem. Commun.* **46**, 6765–6767.
Liu, P., Feng, X. J. & He, R. (2010). *Tetrahedron*, **66**, 631–636.
Muñoz-Hernández, M. A., Keizer, T. S., Parkin, S., Patrick, B. & Patrick, D. A. (2000). *Organometallics*, **19**, 4416–4421.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2012). E**68**, o2942 [https://doi.org/10.1107/S1600536812038615]

(*E*)-2-[(2-Aminophenyl)iminomethyl]-4,6-di-*tert*-butylphenol

Liqin Ding, Xingqiang Lü, Shunsheng Zhao and Yuqin Zhu

S1. Comment

The title compound is an important synthetic intermediate for design and synthesis of asymmetric Schiff base complexes showing excellent catalytic activity in various reactions. In the structure of the title compound the dihedral angle between the two phenyl rings amount to 35.2 (2) ° and all bond lengths are comparable to those observed in similar compounds (Kochem *et al.*, 2010; Belmonte *et al.*, 2010; Liu *et al.*, 2010) (Fig. 1). An intramolecular O—H···N hydrogen bond between the O-H H atom and the N atom N1 is observed (Table 1). In the crystal structure the molecules are linked into chains along the *b* axis by intermolecular N—H···O hydrogen bonding between the amino group and the hydroxy O atom which act as acceptor (Table 1 and Fig. 2).

S2. Experimental

The title compound was obtained according to the synthetic procedure of Muñoz-Hernández *et al.* (2000). 1,2-diamino-benzene (1.0 g, 9.2 mmol) was added to a solution of 3,5-Di-*tert*-butyl-2- hydroxybenzaldehyde (1.1 g, 4.6 mmol) in absolute ethanol (40 ml) and heated to reflux for 4 h, then concentrated to 20 ml by distillation. An orange solid precipitated from the reaction mixture and collected by filtration and dried open air. The orange solid was recrystalized from ethanol to give an orange crystal, which was collected by filtration and dried under vacuum, yield 71.0%. The single-crystal of the title compound suitble for X-ray diffraction was obtained by slow evaporation of an ethanol solution of the title compound.

S3. Refinement

Hydrogen atoms were positioned with idealized geometry (O-H H atoms allowed to rotate but no to tip) and refined using a riding model with N—H = 0.86 Å, C—H = 0.95–0.99 Å, O—H = 0.82 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C/N), U_{iso}(H) = 1.5 U_{eq}(O). Because no strong anomalous scattering atoms are present the absolute structure cannot be determined. Therefore, Friedel opposites were merged in the refinement.

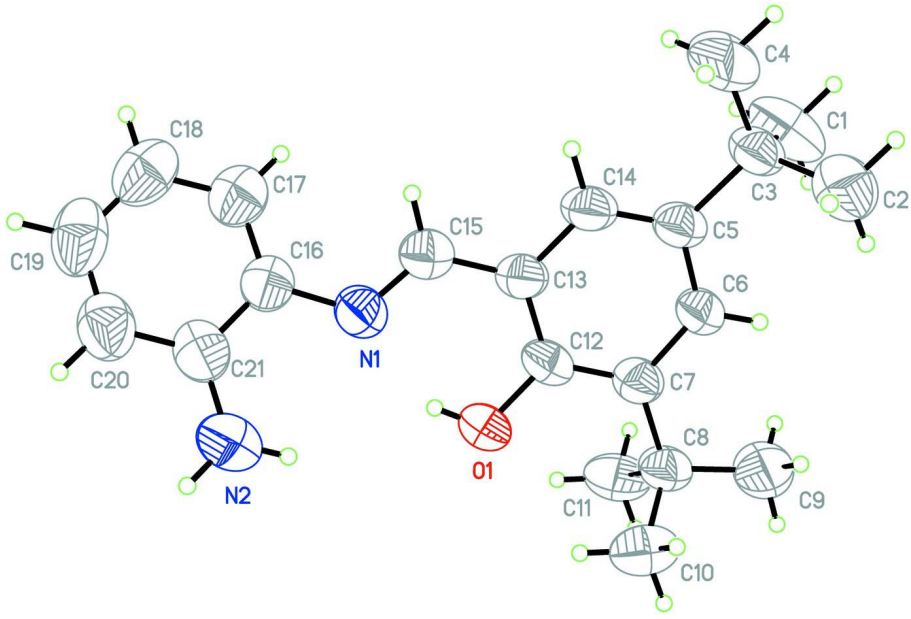


Figure 1

The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids for non-H atoms.

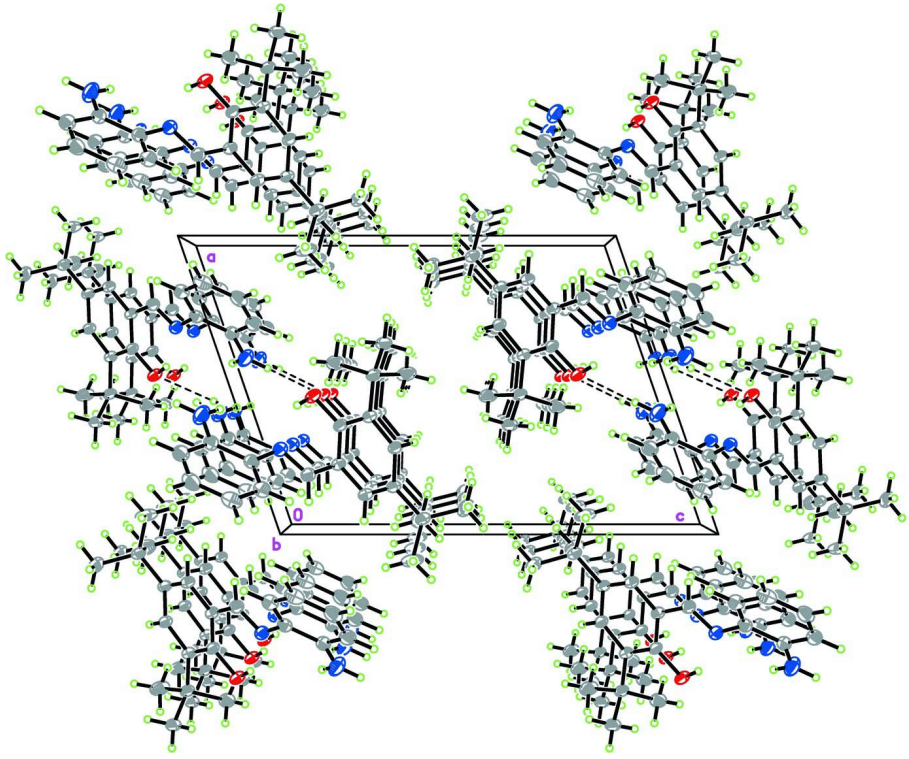


Figure 2

Crystal structure of the title compound with view along the *b* axis. Intermolecular hydrogen bonding is shown as dashed lines.

(*E*)-2-[(2-Aminophenyl)iminomethyl]-4,6-di-*tert*-butylphenol

Crystal data

$C_{21}H_{28}N_2O$
$M_r = 324.45$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 10.898$ (5) Å
$b = 6.230$ (3) Å
$c = 15.095$ (8) Å
$\beta = 108.928$ (6)°
$V = 969.5$ (8) Å^3
$Z = 2$
$F(000) = 352$
$D_x = 1.111$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2451 reflections
$\theta = 1.9$–$26.6°$
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Stick, orange
0.38 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
thin–slice ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.979$, $T_{max} = 0.986$
3718 measured reflections
1357 independent reflections
1224 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 22.2°$, $\theta_{min} = 2.0°$
$h = -11 \rightarrow 11$
$k = -6 \rightarrow 6$
$l = -10 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.129$
$S = 1.10$
1357 reflections
218 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0864P)^2 + 0.0703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.018 (7)

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}
O1	0.5353 (2)	0.2087 (4)	0.80055 (16)	0.0705 (8)
H1A	0.5635	0.3048	0.8389	0.106*
N1	0.6999 (3)	0.4874 (5)	0.9023 (2)	0.0643 (8)

C7	0.6019 (3)	−0.0180 (6)	0.6967 (2)	0.0588 (9)
C6	0.7034 (3)	−0.0771 (7)	0.6648 (2)	0.0603 (9)
H6A	0.6874	−0.1838	0.6193	0.072*
C13	0.7507 (3)	0.2450 (6)	0.7952 (2)	0.0576 (9)
C12	0.6285 (3)	0.1469 (6)	0.7643 (2)	0.0587 (9)
C5	0.8277 (3)	0.0122 (7)	0.6961 (2)	0.0587 (9)
C14	0.8484 (3)	0.1742 (7)	0.7605 (2)	0.0629 (10)
H14A	0.9295	0.2392	0.7818	0.075*
C3	0.9334 (3)	−0.0683 (7)	0.6571 (3)	0.0670 (10)
C15	0.7804 (3)	0.4156 (6)	0.8635 (2)	0.0646 (10)
H15A	0.8626	0.4771	0.8803	0.078*
C8	0.4673 (3)	−0.1218 (7)	0.6579 (2)	0.0669 (11)
C4	1.0650 (4)	0.0253 (13)	0.7108 (4)	0.126 (2)
H4A	1.0608	0.1791	0.7071	0.189*
H4B	1.1284	−0.0257	0.6841	0.189*
H4C	1.0892	−0.0183	0.7752	0.189*
C16	0.7344 (3)	0.6617 (6)	0.9657 (2)	0.0634 (10)
C21	0.6777 (4)	0.6701 (8)	1.0365 (2)	0.0738 (11)
C17	0.8160 (4)	0.8258 (7)	0.9580 (3)	0.0790 (12)
H17A	0.8523	0.8223	0.9102	0.095*
N2	0.5933 (4)	0.5117 (8)	1.0433 (3)	0.1113 (15)
H2A	0.5580	0.5178	1.0866	0.134*
H2B	0.5758	0.4066	1.0043	0.134*
C20	0.7066 (4)	0.8395 (9)	1.0986 (3)	0.0887 (14)
H20A	0.6707	0.8450	1.1466	0.106*
C18	0.8438 (4)	0.9946 (8)	1.0206 (3)	0.0912 (13)
H18A	0.8998	1.1033	1.0157	0.109*
C2	0.9446 (5)	−0.3129 (9)	0.6651 (4)	0.1065 (16)
H2C	0.8623	−0.3766	0.6316	0.160*
H2D	0.9695	−0.3538	0.7298	0.160*
H2E	1.0088	−0.3617	0.6388	0.160*
C9	0.4638 (4)	−0.2935 (10)	0.5856 (4)	0.1117 (19)
H9A	0.5266	−0.4026	0.6137	0.168*
H9B	0.4838	−0.2300	0.5340	0.168*
H9C	0.3789	−0.3564	0.5636	0.168*
C19	0.7882 (4)	1.0003 (10)	1.0899 (3)	0.0961 (15)
H19A	0.8059	1.1147	1.1317	0.115*
C11	0.3664 (3)	0.0476 (9)	0.6095 (3)	0.0846 (14)
H11A	0.3904	0.1140	0.5601	0.127*
H11B	0.3624	0.1547	0.6543	0.127*
H11C	0.2831	−0.0193	0.5838	0.127*
C1	0.8970 (4)	−0.0129 (12)	0.5548 (3)	0.116 (2)
H1C	0.8888	0.1400	0.5473	0.174*
H1D	0.8158	−0.0796	0.5213	0.174*
H1E	0.9631	−0.0636	0.5307	0.174*
C10	0.4275 (4)	−0.2250 (8)	0.7362 (3)	0.0870 (13)
H10A	0.4906	−0.3310	0.7675	0.130*
H10B	0.3443	−0.2921	0.7101	0.130*

H10C	0.4227	−0.1167	0.7802	0.130*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0581 (13)	0.0762 (17)	0.0891 (16)	−0.0028 (14)	0.0405 (13)	−0.0106 (15)
N1	0.0622 (17)	0.067 (2)	0.0671 (17)	0.0028 (16)	0.0262 (14)	−0.0019 (17)
C7	0.0492 (17)	0.062 (2)	0.0684 (19)	0.0013 (17)	0.0239 (15)	0.0010 (19)
C6	0.0470 (18)	0.068 (2)	0.070 (2)	−0.0007 (17)	0.0236 (15)	−0.010 (2)
C13	0.0491 (18)	0.062 (2)	0.0648 (19)	−0.0041 (16)	0.0224 (15)	0.0008 (18)
C12	0.0481 (18)	0.068 (2)	0.0668 (19)	0.0069 (17)	0.0280 (15)	0.004 (2)
C5	0.0445 (17)	0.065 (2)	0.069 (2)	0.0028 (18)	0.0222 (15)	0.003 (2)
C14	0.0468 (17)	0.071 (2)	0.074 (2)	−0.0040 (18)	0.0243 (16)	0.001 (2)
C3	0.050 (2)	0.075 (3)	0.081 (2)	0.0068 (19)	0.0296 (17)	0.001 (2)
C15	0.0577 (19)	0.068 (2)	0.069 (2)	−0.0031 (19)	0.0217 (17)	−0.001 (2)
C8	0.0463 (18)	0.081 (3)	0.076 (2)	−0.0073 (19)	0.0246 (16)	−0.008 (2)
C4	0.051 (2)	0.154 (5)	0.174 (5)	−0.001 (3)	0.039 (3)	−0.047 (5)
C16	0.0574 (19)	0.064 (2)	0.065 (2)	0.005 (2)	0.0137 (16)	−0.001 (2)
C21	0.065 (2)	0.088 (3)	0.065 (2)	0.009 (2)	0.0157 (18)	−0.005 (2)
C17	0.076 (2)	0.075 (3)	0.081 (3)	0.001 (2)	0.018 (2)	0.000 (2)
N2	0.110 (3)	0.137 (4)	0.110 (3)	−0.035 (3)	0.068 (2)	−0.031 (3)
C20	0.075 (3)	0.104 (4)	0.084 (3)	0.010 (3)	0.020 (2)	−0.018 (3)
C18	0.078 (2)	0.074 (3)	0.103 (3)	0.001 (2)	0.004 (2)	−0.002 (3)
C2	0.090 (3)	0.089 (3)	0.151 (4)	0.014 (3)	0.052 (3)	−0.001 (4)
C9	0.068 (2)	0.133 (5)	0.139 (4)	−0.033 (3)	0.039 (3)	−0.061 (4)
C19	0.088 (3)	0.098 (4)	0.083 (3)	0.017 (3)	0.001 (2)	−0.025 (3)
C11	0.0501 (19)	0.115 (4)	0.087 (3)	−0.003 (2)	0.0198 (18)	0.015 (3)
C1	0.096 (3)	0.162 (6)	0.111 (3)	0.032 (4)	0.063 (3)	0.026 (4)
C10	0.069 (2)	0.091 (3)	0.101 (3)	−0.014 (2)	0.027 (2)	0.013 (3)

Geometric parameters (Å, º)

O1—C12	1.357 (4)	C21—N2	1.376 (6)
O1—H1A	0.8200	C21—C20	1.378 (6)
N1—C15	1.284 (4)	C17—C18	1.379 (6)
N1—C16	1.416 (5)	C17—H17A	0.9300
C7—C6	1.393 (4)	N2—H2A	0.8600
C7—C12	1.410 (5)	N2—H2B	0.8600
C7—C8	1.534 (5)	C20—C19	1.373 (7)
C6—C5	1.397 (4)	C20—H20A	0.9300
C6—H6A	0.9300	C18—C19	1.370 (6)
C13—C12	1.400 (5)	C18—H18A	0.9300
C13—C14	1.401 (5)	C2—H2C	0.9600
C13—C15	1.442 (5)	C2—H2D	0.9600
C5—C14	1.368 (5)	C2—H2E	0.9600
C5—C3	1.537 (5)	C9—H9A	0.9600
C14—H14A	0.9300	C9—H9B	0.9600
C3—C1	1.504 (6)	C9—H9C	0.9600

C3—C4	1.517 (6)	C19—H19A	0.9300
C3—C2	1.530 (7)	C11—H11A	0.9600
C15—H15A	0.9300	C11—H11B	0.9600
C8—C9	1.520 (6)	C11—H11C	0.9600
C8—C11	1.528 (6)	C1—H1C	0.9600
C8—C10	1.526 (6)	C1—H1D	0.9600
C4—H4A	0.9600	C1—H1E	0.9600
C4—H4B	0.9600	C10—H10A	0.9600
C4—H4C	0.9600	C10—H10B	0.9600
C16—C17	1.384 (6)	C10—H10C	0.9600
C16—C21	1.399 (5)		

C12—O1—H1A	109.5	C20—C21—C16	119.2 (4)
C15—N1—C16	120.3 (3)	C18—C17—C16	120.7 (4)
C6—C7—C12	116.3 (3)	C18—C17—H17A	119.6
C6—C7—C8	121.6 (3)	C16—C17—H17A	119.6
C12—C7—C8	122.0 (3)	C21—N2—H2A	120.0
C7—C6—C5	124.7 (3)	C21—N2—H2B	120.0
C7—C6—H6A	117.6	H2A—N2—H2B	120.0
C5—C6—H6A	117.6	C19—C20—C21	120.4 (4)
C12—C13—C14	119.6 (3)	C19—C20—H20A	119.8
C12—C13—C15	121.9 (3)	C21—C20—H20A	119.8
C14—C13—C15	118.5 (3)	C19—C18—C17	119.3 (5)
O1—C12—C13	119.8 (3)	C19—C18—H18A	120.4
O1—C12—C7	119.5 (3)	C17—C18—H18A	120.4
C13—C12—C7	120.6 (3)	C3—C2—H2C	109.5
C14—C5—C6	116.9 (3)	C3—C2—H2D	109.5
C14—C5—C3	122.8 (3)	H2C—C2—H2D	109.5
C6—C5—C3	120.3 (3)	C3—C2—H2E	109.5
C5—C14—C13	121.9 (3)	H2C—C2—H2E	109.5
C5—C14—H14A	119.1	H2D—C2—H2E	109.5
C13—C14—H14A	119.1	C8—C9—H9A	109.5
C1—C3—C4	110.4 (4)	C8—C9—H9B	109.5
C1—C3—C2	107.3 (5)	H9A—C9—H9B	109.5
C4—C3—C2	107.6 (4)	C8—C9—H9C	109.5
C1—C3—C5	109.7 (3)	H9A—C9—H9C	109.5
C4—C3—C5	111.5 (3)	H9B—C9—H9C	109.5
C2—C3—C5	110.2 (4)	C18—C19—C20	121.0 (5)
N1—C15—C13	123.7 (3)	C18—C19—H19A	119.5
N1—C15—H15A	118.2	C20—C19—H19A	119.5
C13—C15—H15A	118.2	C8—C11—H11A	109.5
C9—C8—C11	107.2 (3)	C8—C11—H11B	109.5
C9—C8—C10	108.2 (4)	H11A—C11—H11B	109.5
C11—C8—C10	108.6 (3)	C8—C11—H11C	109.5
C9—C8—C7	111.7 (3)	H11A—C11—H11C	109.5
C11—C8—C7	110.0 (4)	H11B—C11—H11C	109.5
C10—C8—C7	111.0 (3)	C3—C1—H1C	109.5
C3—C4—H4A	109.5	C3—C1—H1D	109.5

C3—C4—H4B	109.5	H1C—C1—H1D	109.5
H4A—C4—H4B	109.5	C3—C1—H1E	109.5
C3—C4—H4C	109.5	H1C—C1—H1E	109.5
H4A—C4—H4C	109.5	H1D—C1—H1E	109.5
H4B—C4—H4C	109.5	C8—C10—H10A	109.5
C17—C16—C21	119.4 (4)	C8—C10—H10B	109.5
C17—C16—N1	123.2 (3)	H10A—C10—H10B	109.5
C21—C16—N1	117.3 (4)	C8—C10—H10C	109.5
N2—C21—C20	120.6 (4)	H10A—C10—H10C	109.5
N2—C21—C16	120.2 (4)	H10B—C10—H10C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.82	1.87	2.608 (4)	149
N2—H2*A*···O1^{i}	0.86	2.54	3.342 (4)	155

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+2.