Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(Benzylideneacetyl)-2-phenyl-1,2,3,5,10,10a-hexahydro-1*H*-pyrrolo[1,2-*b*]isoquinoline-3-spiro-3′(2′*H*)-1*H*-indol-3′-one

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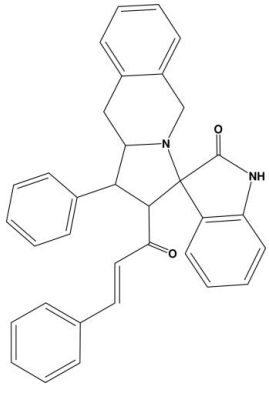
Received 27 March 2007; accepted 9 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.122; data-to-parameter ratio = 9.5.

The pyrrolidine ring in the title compound, $C_{34}H_{28}N_2O_2$, adopts a twist conformation in one of the two independent molecules and an envelope conformation in the other. The oxindole group in both molecules is planar. The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by intermolecular N—H···O interactions.

Related literature

For related literature, see: Allen *et al.* (1987); Amal Raj *et al.* (2003); Cremer & Pople (1975); Nardelli (1983); Stylianakis *et al.* (2003).



Experimental

Crystal data

$C_{34}H_{28}N_2O_2$
M_r = 496.58
Orthorhombic, $Pca2_1$
a = 19.301 (4) Å
b = 14.725 (3) Å
c = 18.583 (4) Å
V = 5281 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.28 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
44375 measured reflections
6482 independent reflections
5365 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.122
S = 1.12
6482 reflections
685 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4^{i}	0.86	2.19	3.023 (3)	164
N4—H4A···O2^{ii}	0.86	2.23	3.047 (3)	159
C2—H2A···O1	0.98	2.57	3.034 (3)	109
C12—H12A···O1	0.97	2.54	3.201 (3)	125
C37—H37···O4	0.98	2.44	2.784 (3)	100
C56—H56···O4	0.93	2.51	2.830 (4)	101

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

DG thanks the CSIR, India, for the award of a Senior Research Fellowship. DV thanks the DST, India, for a Major Research Project. The Department of Science and Technology (DST–FIST) and the University Grants Commission (UGC), Government of India, are acknowledged by DV for providing facilities to the department.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PR2006).

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  doi:10.1107/S160053680703348X

supplementary materials

Acta Cryst. (2007). E**63**, o3490 [**doi:10.1107/S160053680703348X**]

1-(Benzylideneacetyl)-2-phenyl-1,2,3,5,10,10a-hexahydro-1*H*-pyrrolo[1,2-*b*]isoquinoline-3-spiro-3'(2'*H*)-1*H*-indol-3'-one

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Comment

Pyrrolidine occurs widely in nature and is a structural part of porphyrin heme, chlorophyll and vitamin B12. Pyrrolidine compounds have antifungal and antimicrobial activity (Amal Raj *et al.*, 2003). The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids. Synthetic spiro-pyrrolidine derivatives have activity against the aldose reductase enzyme which controls influenza (Stylianakis *et al.*, 2003). As spiropyrrolidine compounds are of great pharmaceutical importance, we have undertaken the three dimensional crystal structure determination of the title compound, C_{34} H_{28} N_2 O_2, by X-ray diffraction (Fig.1).

The bond lengths and angles are comparable with the literature values (Allen *et al.*, 1987). The sum of the bond angles around N1 (339.5 (6)°) and N3 (336.0 (6)°) indicates the sp^3 hybridization. The pyrrolidine ring in molecule A adopts a twisted conformation with a pseudo twofold axis passing through atom C1 and C3—C4 bond. In molecule B, it adopts an envelope conformation with atom C38 deviating by −0.608 (2)Å from the plane of rest of the atoms in the ring. The six membered rings N1/C4—C6/C11/C12 and N3/C38—C40/C45/C46 in molecules A and B, respectively, are non-planar. The oxindole ring in molecules A and B is planar with the dihedral angle between five and six membered rings being 2.3 (1) and 1.8 (1)°, respectively. Oxygen atoms O1 and O3 lie below the plane of the five membered rings in the oxindole moiety in molecule A and B by 0.021 (2) and 0.049 (2) Å, respectively.

The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for pyrrolidine ring N1/C1—C4 in the molecule A are q_2 = 0.392 (3) Å, φ = 310.6 (4)° and $\Delta_2(C_1)$ = 7.5 (2), for pyrrolidine ring N3/C35—C38 in the molecule B are q_2 = 0.400 (3) Å, φ = 152.0 (4)° and $\Delta_s(C_{38})$ = 9.7 (2), for six memebered rings (N1/C4—C6/C11/C12 and N3/C38—C40/C45/C46) in molecule A and B are q2 = 0.394 (3) Å; 0.369 (3) Å, q3 = 0.328 (3) Å; −0.314 (3) Å, Q_T = 0.513 (3) Å; 0.485 (3)Å and θ = 50.2 (3)°; s130.4(3)°, respectively.

The molecule is stabilized by weak C—H···O intramolecular interactions. The crystal packing is stabilized by two N—H···O intermolecular interactions. Atoms N2 and N4 act as donors to O4 (1/2 + *x*,1 − *y*,*z*) and O2 (−1/2 + *x*,-*y*,*z*), generating a chain running along *b* axis.

Experimental

A mixture of isoquinolic aicd (0.186 g, 1 mmol), isatin (0.147 g, 1 mmol), bis-benzylidine acetone (0.224 g, 1 mmol) and triethyl amine (0.252 g, 2.5 mmol) was refluxed in acetonitrile for 12 h. The reaction was monitored by TLC and after completion, the solvent was removed at reduced pressure. The crude product was subjected to column chromatrography (Silica gel, 100–200 mesh) using Hexane/Ethyl acetate (9:1) as eluent. The compound was recrystallized from Hexane/Ethyl acetate (1:1) by slow evaporation.

Refinement

All H-atoms were refined using a riding model with d(C—H) = 0.93 Å, U_{iso} = 1.2U_{eq} (C) for aromatic, 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms and 0.86 Å, U_{iso} = 1.2U_{eq} (N) for the NH group.

Figures

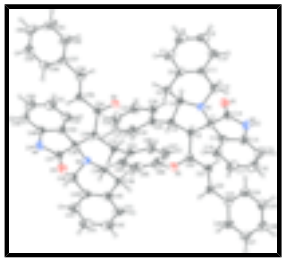

Fig. 1. The molecular structure of title compound, showing 30% probability displacement ellipsoids.

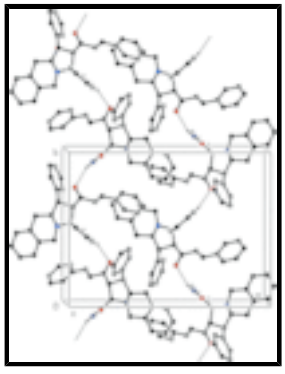

Fig. 2. The molecular packing of (I), viewed down the *c* axis. For clarity, hydrogen atoms which are not involved in hydrogen bonding were omitted.

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Crystal data

$C_{34}H_{28}N_2O_2$	F_{000} = 2096
M_r = 496.58	D_x = 1.249 Mg m^{-3}
Orthorhombic, *Pca*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 3158 reflections
a = 19.301 (4) Å	θ = 1.4–25.0º
b = 14.725 (3) Å	μ = 0.08 mm^{-1}
c = 18.583 (4) Å	*T* = 293 (2) K
V = 5281 (2) Å^3	Block, colourless
Z = 8	0.28 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector	5365 reflections with $I > 2\sigma(I)$

diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
T = 293(2) K
ω scans
Absorption correction: none
44375 measured reflections
6482 independent reflections

R_{int} = 0.030
θ_{max} = 28.1º
θ_{min} = 1.4º
h = −25→25
k = −19→19
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.122
S = 1.12
6482 reflections
685 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
$w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.46079 (10)	0.50133 (13)	0.42242 (11)	0.0422 (4)
N2	0.62533 (12)	0.58129 (15)	0.45090 (14)	0.0587 (6)
H2	0.6587	0.6178	0.4406	0.070*
O1	0.59608 (10)	0.53639 (13)	0.33659 (12)	0.0601 (5)
O2	0.54648 (10)	0.24727 (13)	0.49803 (11)	0.0581 (5)
C1	0.53286 (12)	0.47841 (16)	0.44209 (13)	0.0403 (5)
C2	0.53957 (12)	0.37561 (14)	0.42011 (13)	0.0403 (5)
H2A	0.5671	0.3732	0.3758	0.048*

C3	0.46564 (12)	0.34610 (15)	0.40080 (13)	0.0402 (5)
H3	0.4416	0.3289	0.4452	0.048*
C4	0.43374 (12)	0.43268 (15)	0.37264 (13)	0.0432 (5)
H4	0.4503	0.4449	0.3237	0.052*
C5	0.35535 (13)	0.43692 (18)	0.37494 (16)	0.0526 (6)
H5A	0.3364	0.3977	0.3379	0.063*
H5B	0.3392	0.4150	0.4212	0.063*
C6	0.32979 (13)	0.53207 (18)	0.36345 (14)	0.0512 (6)
C7	0.26194 (15)	0.5487 (2)	0.34094 (17)	0.0632 (7)
H7	0.2320	0.5000	0.3337	0.076*
C8	0.23858 (17)	0.6352 (3)	0.32928 (19)	0.0743 (9)
H8	0.1934	0.6447	0.3136	0.089*
C9	0.28120 (18)	0.7074 (3)	0.3405 (2)	0.0788 (10)
H9	0.2650	0.7662	0.3335	0.095*
C10	0.34873 (17)	0.6929 (2)	0.36238 (18)	0.0659 (8)
H10	0.3781	0.7423	0.3688	0.079*
C11	0.37348 (14)	0.60568 (18)	0.37491 (14)	0.0513 (6)
C12	0.44767 (14)	0.59353 (17)	0.39869 (18)	0.0550 (6)
H12A	0.4784	0.6081	0.3590	0.066*
H12B	0.4576	0.6352	0.4378	0.066*
C13	0.58798 (12)	0.53482 (15)	0.40082 (16)	0.0467 (5)
C14	0.60314 (14)	0.56264 (18)	0.52101 (16)	0.0542 (6)
C15	0.62925 (19)	0.5944 (3)	0.5859 (2)	0.0797 (10)
H15	0.6656	0.6358	0.5873	0.096*
C16	0.5996 (2)	0.5626 (3)	0.6476 (2)	0.0880 (11)
H16	0.6165	0.5830	0.6916	0.106*
C17	0.54584 (18)	0.5018 (3)	0.64710 (18)	0.0746 (9)
H17	0.5273	0.4810	0.6902	0.090*
C18	0.51918 (14)	0.47146 (19)	0.58210 (16)	0.0543 (6)
H18	0.4822	0.4310	0.5811	0.065*
C19	0.54836 (12)	0.50215 (16)	0.51922 (14)	0.0445 (5)
C20	0.57527 (12)	0.31515 (16)	0.47504 (13)	0.0434 (5)
C21	0.64594 (12)	0.33979 (16)	0.49544 (14)	0.0468 (6)
H21	0.6688	0.3841	0.4688	0.056*
C22	0.67879 (13)	0.30178 (18)	0.55021 (15)	0.0494 (6)
H22	0.6552	0.2560	0.5745	0.059*
C23	0.74793 (14)	0.32391 (17)	0.57657 (14)	0.0497 (6)
C24	0.78893 (15)	0.3894 (2)	0.54376 (17)	0.0622 (7)
H24	0.7723	0.4202	0.5037	0.075*
C25	0.85405 (16)	0.4092 (2)	0.56991 (19)	0.0698 (8)
H25	0.8810	0.4531	0.5473	0.084*
C26	0.87937 (17)	0.3645 (2)	0.62915 (19)	0.0689 (8)
H26	0.9231	0.3784	0.6470	0.083*
C27	0.83963 (17)	0.2994 (2)	0.66163 (18)	0.0705 (8)
H27	0.8568	0.2686	0.7015	0.085*
C28	0.77457 (15)	0.2788 (2)	0.63601 (17)	0.0626 (7)
H28	0.7483	0.2342	0.6587	0.075*
C29	0.46236 (12)	0.26695 (15)	0.34907 (13)	0.0419 (5)
C30	0.49026 (15)	0.27250 (18)	0.28099 (14)	0.0513 (6)

H30	0.5118	0.3258	0.2663	0.062*
C31	0.48653 (17)	0.1997 (2)	0.23432 (16)	0.0645 (8)
H31	0.5053	0.2045	0.1884	0.077*
C32	0.45541 (18)	0.1202 (2)	0.2553 (2)	0.0717 (9)
H32	0.4529	0.0712	0.2238	0.086*
C33	0.42797 (19)	0.1138 (2)	0.3231 (2)	0.0755 (9)
H33	0.4072	0.0600	0.3380	0.091*
C34	0.43110 (16)	0.18685 (19)	0.36949 (17)	0.0605 (7)
H34	0.4118	0.1820	0.4152	0.073*
N3	0.30081 (10)	0.01027 (14)	0.48973 (11)	0.0431 (4)
N4	0.13746 (12)	−0.08201 (15)	0.46957 (14)	0.0538 (5)
H4A	0.1044	−0.1181	0.4817	0.065*
O3	0.17250 (10)	−0.03839 (14)	0.58196 (11)	0.0622 (5)
O4	0.22277 (11)	0.26153 (13)	0.42423 (12)	0.0629 (5)
C35	0.22770 (12)	0.02546 (15)	0.47260 (13)	0.0404 (5)
C36	0.21591 (12)	0.12748 (15)	0.49559 (13)	0.0415 (5)
H36	0.1801	0.1271	0.5330	0.050*
C37	0.28381 (12)	0.15756 (16)	0.53213 (13)	0.0419 (5)
H37	0.3125	0.1880	0.4959	0.050*
C38	0.31875 (12)	0.06702 (15)	0.55144 (13)	0.0423 (5)
H38	0.2978	0.0420	0.5952	0.051*
C39	0.39667 (14)	0.07150 (18)	0.56000 (16)	0.0546 (6)
H39A	0.4155	0.1128	0.5243	0.066*
H39B	0.4076	0.0957	0.6072	0.066*
C40	0.43057 (12)	−0.02032 (19)	0.55155 (14)	0.0514 (6)
C41	0.49887 (14)	−0.0325 (2)	0.57275 (16)	0.0615 (7)
H41	0.5236	0.0162	0.5917	0.074*
C42	0.53040 (15)	−0.1157 (3)	0.56606 (17)	0.0717 (9)
H42	0.5763	−0.1229	0.5803	0.086*
C43	0.49443 (17)	−0.1884 (3)	0.53838 (19)	0.0723 (9)
H43	0.5158	−0.2448	0.5344	0.087*
C44	0.42672 (16)	−0.1777 (2)	0.51665 (17)	0.0646 (8)
H44	0.4024	−0.2268	0.4979	0.078*
C45	0.39465 (14)	−0.09327 (19)	0.52270 (14)	0.0517 (6)
C46	0.31998 (14)	−0.08480 (18)	0.49940 (17)	0.0564 (7)
H46A	0.3133	−0.1173	0.4545	0.068*
H46B	0.2902	−0.1121	0.5354	0.068*
C47	0.17671 (12)	−0.03513 (16)	0.51703 (15)	0.0459 (5)
C48	0.15689 (13)	−0.06487 (17)	0.39832 (16)	0.0503 (6)
C49	0.12964 (18)	−0.1014 (2)	0.3361 (2)	0.0708 (9)
H49	0.0932	−0.1426	0.3375	0.085*
C50	0.1583 (2)	−0.0746 (3)	0.2723 (2)	0.0866 (11)
H50	0.1403	−0.0974	0.2295	0.104*
C51	0.2136 (2)	−0.0145 (3)	0.26936 (19)	0.0803 (10)
H51	0.2331	0.0010	0.2253	0.096*
C52	0.23952 (17)	0.02232 (19)	0.33228 (16)	0.0598 (7)
H52	0.2761	0.0635	0.3308	0.072*
C53	0.21077 (13)	−0.00253 (16)	0.39683 (14)	0.0452 (5)
C54	0.19103 (13)	0.19223 (16)	0.43715 (14)	0.0446 (5)

C55	0.12545 (13)	0.17004 (17)	0.40118 (15)	0.0487 (6)
H55	0.0979	0.1239	0.4199	0.058*
C56	0.10429 (14)	0.21369 (19)	0.34293 (16)	0.0547 (6)
H56	0.1323	0.2612	0.3275	0.066*
C57	0.04232 (16)	0.1963 (2)	0.29999 (16)	0.0574 (7)
C58	−0.00864 (18)	0.1359 (2)	0.3203 (2)	0.0738 (9)
H58	−0.0046	0.1045	0.3635	0.089*
C59	−0.0659 (2)	0.1219 (2)	0.2767 (3)	0.0885 (12)
H59	−0.1006	0.0821	0.2913	0.106*
C60	−0.0717 (2)	0.1664 (3)	0.2121 (2)	0.0898 (13)
H60	−0.1091	0.1548	0.1819	0.108*
C61	−0.0228 (3)	0.2269 (4)	0.1927 (2)	0.1200 (18)
H61	−0.0274	0.2585	0.1497	0.144*
C62	0.0339 (2)	0.2423 (3)	0.2362 (2)	0.0962 (13)
H62	0.0670	0.2847	0.2221	0.115*
C63	0.27609 (13)	0.22087 (17)	0.59528 (15)	0.0483 (6)
C64	0.23529 (16)	0.1990 (2)	0.65397 (16)	0.0600 (7)
H64	0.2100	0.1452	0.6535	0.072*
C65	0.2313 (2)	0.2548 (3)	0.71295 (19)	0.0781 (10)
H65	0.2038	0.2385	0.7519	0.094*
C66	0.2676 (2)	0.3339 (3)	0.7146 (2)	0.0886 (12)
H66	0.2651	0.3714	0.7548	0.106*
C67	0.3075 (2)	0.3580 (2)	0.6576 (3)	0.0885 (12)
H67	0.3320	0.4124	0.6587	0.106*
C68	0.31184 (16)	0.3019 (2)	0.5973 (2)	0.0668 (8)
H68	0.3390	0.3192	0.5584	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0385 (10)	0.0402 (10)	0.0480 (11)	0.0028 (8)	−0.0090 (8)	−0.0060 (8)
N2	0.0500 (13)	0.0505 (13)	0.0755 (17)	−0.0118 (10)	−0.0073 (11)	−0.0034 (12)
O1	0.0590 (11)	0.0635 (12)	0.0577 (12)	−0.0005 (9)	0.0036 (9)	0.0076 (9)
O2	0.0575 (11)	0.0499 (10)	0.0669 (12)	−0.0013 (8)	−0.0082 (9)	0.0105 (9)
C1	0.0405 (12)	0.0371 (11)	0.0432 (12)	0.0030 (9)	−0.0041 (9)	−0.0039 (9)
C2	0.0429 (12)	0.0361 (11)	0.0420 (12)	0.0009 (9)	0.0000 (9)	−0.0036 (9)
C3	0.0409 (11)	0.0416 (11)	0.0380 (11)	−0.0013 (9)	0.0004 (9)	−0.0026 (10)
C4	0.0460 (13)	0.0426 (12)	0.0409 (12)	0.0029 (10)	−0.0060 (10)	−0.0043 (10)
C5	0.0439 (13)	0.0541 (14)	0.0599 (16)	−0.0005 (11)	−0.0101 (11)	−0.0085 (12)
C6	0.0465 (13)	0.0630 (15)	0.0440 (13)	0.0062 (12)	−0.0032 (11)	−0.0068 (12)
C7	0.0487 (15)	0.0853 (19)	0.0555 (16)	0.0106 (14)	−0.0086 (13)	−0.0087 (15)
C8	0.0513 (17)	0.103 (3)	0.069 (2)	0.0255 (18)	−0.0135 (15)	0.0011 (19)
C9	0.074 (2)	0.082 (2)	0.080 (2)	0.0339 (19)	−0.0092 (18)	0.0144 (19)
C10	0.0651 (18)	0.0601 (16)	0.072 (2)	0.0130 (14)	−0.0058 (15)	0.0103 (15)
C11	0.0525 (14)	0.0536 (14)	0.0479 (14)	0.0106 (12)	−0.0055 (11)	−0.0009 (11)
C12	0.0515 (14)	0.0408 (12)	0.0729 (17)	0.0055 (11)	−0.0098 (13)	−0.0023 (13)
C13	0.0442 (13)	0.0383 (12)	0.0575 (16)	0.0033 (10)	−0.0041 (11)	0.0019 (11)
C14	0.0520 (14)	0.0497 (14)	0.0609 (16)	0.0032 (11)	−0.0098 (12)	−0.0133 (13)

C15	0.070 (2)	0.081 (2)	0.088 (3)	−0.0124 (17)	−0.0209 (19)	−0.026 (2)
C16	0.087 (2)	0.119 (3)	0.058 (2)	−0.006 (2)	−0.0176 (18)	−0.028 (2)
C17	0.074 (2)	0.100 (3)	0.0504 (18)	0.0078 (19)	−0.0034 (15)	−0.0125 (17)
C18	0.0539 (15)	0.0588 (15)	0.0504 (15)	0.0049 (12)	−0.0047 (12)	−0.0076 (12)
C19	0.0433 (12)	0.0403 (12)	0.0498 (14)	0.0072 (10)	−0.0104 (10)	−0.0093 (11)
C20	0.0469 (13)	0.0387 (12)	0.0447 (13)	0.0053 (10)	−0.0010 (10)	−0.0039 (10)
C21	0.0454 (12)	0.0423 (12)	0.0527 (14)	0.0041 (10)	−0.0005 (11)	0.0055 (11)
C22	0.0484 (13)	0.0522 (14)	0.0476 (13)	−0.0023 (11)	−0.0003 (11)	0.0047 (11)
C23	0.0470 (13)	0.0557 (14)	0.0462 (14)	0.0022 (12)	−0.0014 (11)	0.0016 (11)
C24	0.0584 (16)	0.0696 (17)	0.0585 (17)	−0.0050 (13)	−0.0082 (13)	0.0161 (14)
C25	0.0584 (17)	0.0731 (19)	0.078 (2)	−0.0158 (15)	−0.0037 (15)	0.0087 (16)
C26	0.0549 (16)	0.083 (2)	0.0691 (19)	−0.0074 (15)	−0.0147 (15)	−0.0039 (17)
C27	0.0664 (19)	0.090 (2)	0.0546 (17)	0.0014 (17)	−0.0169 (14)	0.0131 (16)
C28	0.0600 (17)	0.0762 (19)	0.0517 (16)	−0.0079 (14)	−0.0040 (13)	0.0137 (14)
C29	0.0421 (12)	0.0396 (12)	0.0440 (13)	−0.0008 (9)	−0.0014 (10)	−0.0034 (10)
C30	0.0642 (16)	0.0461 (13)	0.0437 (14)	−0.0075 (12)	0.0018 (12)	−0.0009 (11)
C31	0.080 (2)	0.0680 (18)	0.0454 (15)	−0.0018 (16)	0.0021 (14)	−0.0125 (13)
C32	0.084 (2)	0.0561 (17)	0.075 (2)	−0.0078 (15)	−0.0017 (17)	−0.0282 (16)
C33	0.085 (2)	0.0482 (15)	0.093 (3)	−0.0221 (15)	0.0184 (19)	−0.0138 (16)
C34	0.0669 (18)	0.0528 (15)	0.0617 (17)	−0.0094 (13)	0.0158 (14)	−0.0052 (13)
N3	0.0380 (9)	0.0465 (11)	0.0449 (11)	0.0043 (8)	−0.0035 (8)	0.0017 (8)
N4	0.0482 (12)	0.0454 (11)	0.0679 (14)	−0.0106 (9)	0.0023 (10)	0.0045 (10)
O3	0.0646 (12)	0.0704 (13)	0.0515 (12)	−0.0062 (10)	0.0080 (9)	0.0138 (10)
O4	0.0633 (12)	0.0512 (11)	0.0742 (14)	−0.0054 (9)	−0.0162 (10)	0.0170 (10)
C35	0.0390 (12)	0.0408 (12)	0.0415 (12)	−0.0024 (9)	−0.0006 (10)	0.0038 (10)
C36	0.0409 (12)	0.0437 (12)	0.0398 (12)	−0.0020 (9)	0.0014 (9)	0.0026 (9)
C37	0.0416 (12)	0.0439 (12)	0.0402 (12)	−0.0018 (9)	−0.0007 (9)	0.0045 (10)
C38	0.0430 (12)	0.0442 (12)	0.0399 (12)	−0.0008 (9)	−0.0021 (10)	0.0042 (10)
C39	0.0479 (14)	0.0581 (15)	0.0578 (16)	−0.0028 (11)	−0.0100 (12)	0.0070 (13)
C40	0.0419 (13)	0.0687 (16)	0.0436 (13)	0.0046 (11)	0.0018 (10)	0.0126 (12)
C41	0.0417 (13)	0.087 (2)	0.0553 (16)	0.0042 (14)	0.0023 (12)	0.0156 (15)
C42	0.0424 (14)	0.113 (3)	0.0598 (18)	0.0214 (17)	0.0077 (13)	0.0180 (18)
C43	0.0666 (19)	0.090 (2)	0.0602 (18)	0.0373 (18)	0.0085 (15)	0.0073 (16)
C44	0.0651 (17)	0.0672 (18)	0.0616 (17)	0.0219 (14)	0.0024 (14)	−0.0005 (14)
C45	0.0482 (13)	0.0626 (15)	0.0444 (13)	0.0119 (12)	0.0033 (11)	0.0058 (12)
C46	0.0514 (15)	0.0485 (14)	0.0692 (18)	0.0086 (11)	−0.0094 (13)	−0.0040 (13)
C47	0.0406 (12)	0.0412 (12)	0.0558 (15)	0.0031 (10)	0.0012 (11)	0.0086 (11)
C48	0.0468 (13)	0.0425 (12)	0.0617 (15)	−0.0005 (11)	−0.0073 (12)	−0.0001 (12)
C49	0.077 (2)	0.0605 (17)	0.075 (2)	−0.0126 (15)	−0.0192 (18)	−0.0099 (16)
C50	0.110 (3)	0.082 (2)	0.067 (2)	−0.009 (2)	−0.028 (2)	−0.0163 (19)
C51	0.105 (3)	0.085 (2)	0.0504 (18)	−0.005 (2)	−0.0063 (17)	0.0003 (16)
C52	0.0709 (18)	0.0601 (16)	0.0483 (15)	−0.0069 (14)	−0.0005 (13)	0.0013 (13)
C53	0.0452 (12)	0.0418 (12)	0.0485 (13)	0.0039 (10)	−0.0063 (10)	−0.0015 (11)
C54	0.0481 (13)	0.0409 (13)	0.0449 (13)	0.0060 (10)	−0.0016 (10)	0.0010 (10)
C55	0.0444 (13)	0.0466 (13)	0.0551 (14)	0.0035 (10)	−0.0042 (11)	0.0045 (11)
C56	0.0590 (15)	0.0524 (14)	0.0525 (15)	0.0034 (12)	−0.0055 (12)	0.0037 (12)
C57	0.0609 (17)	0.0590 (16)	0.0523 (15)	0.0122 (13)	−0.0124 (12)	−0.0038 (12)
C58	0.078 (2)	0.0532 (16)	0.091 (2)	0.0015 (15)	−0.0312 (18)	0.0114 (16)
C59	0.082 (2)	0.0533 (17)	0.130 (4)	0.0014 (16)	−0.046 (2)	−0.002 (2)

C60	0.097 (3)	0.085 (2)	0.087 (3)	0.018 (2)	−0.050 (2)	−0.016 (2)
C61	0.111 (4)	0.179 (5)	0.071 (3)	−0.002 (4)	−0.042 (3)	0.028 (3)
C62	0.089 (3)	0.133 (4)	0.067 (2)	−0.011 (2)	−0.0248 (19)	0.028 (2)
C63	0.0493 (14)	0.0438 (13)	0.0519 (15)	0.0064 (10)	−0.0107 (11)	0.0011 (11)
C64	0.0723 (19)	0.0588 (16)	0.0488 (15)	0.0069 (14)	−0.0019 (14)	−0.0012 (13)
C65	0.091 (2)	0.089 (2)	0.0540 (18)	0.027 (2)	−0.0041 (16)	−0.0077 (17)
C66	0.099 (3)	0.087 (3)	0.080 (3)	0.021 (2)	−0.017 (2)	−0.036 (2)
C67	0.084 (2)	0.059 (2)	0.123 (4)	−0.0040 (17)	−0.020 (2)	−0.029 (2)
C68	0.0649 (18)	0.0544 (16)	0.081 (2)	−0.0040 (13)	−0.0079 (16)	−0.0097 (15)

Geometric parameters (Å, °)

N1—C12	1.450 (3)	N3—C46	1.459 (3)
N1—C4	1.466 (3)	N3—C38	1.461 (3)
N1—C1	1.477 (3)	N3—C35	1.464 (3)
N2—C13	1.362 (4)	N4—C47	1.352 (4)
N2—C14	1.399 (4)	N4—C48	1.399 (4)
N2—H2	0.8600	N4—H4A	0.8600
O1—C13	1.204 (3)	O3—C47	1.210 (3)
O2—C20	1.221 (3)	O4—C54	1.214 (3)
C1—C19	1.505 (3)	C35—C53	1.503 (4)
C1—C13	1.552 (4)	C35—C47	1.564 (3)
C1—C2	1.573 (3)	C35—C36	1.578 (3)
C2—C20	1.520 (3)	C36—C54	1.523 (3)
C2—C3	1.534 (3)	C36—C37	1.541 (3)
C2—H2A	0.9800	C36—H36	0.9800
C3—C4	1.510 (3)	C37—C63	1.506 (3)
C3—C29	1.512 (3)	C37—C38	1.537 (3)
C3—H3	0.9800	C37—H37	0.9800
C4—C5	1.515 (4)	C38—C39	1.514 (3)
C4—H4	0.9800	C38—H38	0.9800
C5—C6	1.501 (4)	C39—C40	1.510 (4)
C5—H5A	0.9700	C39—H39A	0.9700
C5—H5B	0.9700	C39—H39B	0.9700
C6—C11	1.390 (4)	C40—C45	1.386 (4)
C6—C7	1.397 (4)	C40—C41	1.387 (4)
C7—C8	1.368 (4)	C41—C42	1.374 (5)
C7—H7	0.9300	C41—H41	0.9300
C8—C9	1.361 (5)	C42—C43	1.376 (5)
C8—H8	0.9300	C42—H42	0.9300
C9—C10	1.382 (4)	C43—C44	1.377 (5)
C9—H9	0.9300	C43—H43	0.9300
C10—C11	1.390 (4)	C44—C45	1.393 (4)
C10—H10	0.9300	C44—H44	0.9300
C11—C12	1.509 (4)	C45—C46	1.510 (4)
C12—H12A	0.9700	C46—H46A	0.9700
C12—H12B	0.9700	C46—H46B	0.9700
C14—C19	1.383 (4)	C48—C49	1.380 (4)
C14—C15	1.388 (4)	C48—C53	1.388 (4)

C15—C16	1.364 (6)	C49—C50	1.367 (6)
C15—H15	0.9300	C49—H49	0.9300
C16—C17	1.371 (6)	C50—C51	1.387 (6)
C16—H16	0.9300	C50—H50	0.9300
C17—C18	1.387 (4)	C51—C52	1.383 (5)
C17—H17	0.9300	C51—H51	0.9300
C18—C19	1.374 (4)	C52—C53	1.371 (4)
C18—H18	0.9300	C52—H52	0.9300
C20—C21	1.462 (3)	C54—C55	1.468 (4)
C21—C22	1.323 (4)	C55—C56	1.323 (4)
C21—H21	0.9300	C55—H55	0.9300
C22—C23	1.459 (4)	C56—C57	1.460 (4)
C22—H22	0.9300	C56—H56	0.9300
C23—C28	1.388 (4)	C57—C62	1.375 (5)
C23—C24	1.388 (4)	C57—C58	1.378 (5)
C24—C25	1.379 (4)	C58—C59	1.386 (5)
C24—H24	0.9300	C58—H58	0.9300
C25—C26	1.372 (5)	C59—C60	1.373 (6)
C25—H25	0.9300	C59—H59	0.9300
C26—C27	1.369 (5)	C60—C61	1.346 (7)
C26—H26	0.9300	C60—H60	0.9300
C27—C28	1.377 (4)	C61—C62	1.379 (6)
C27—H27	0.9300	C61—H61	0.9300
C28—H28	0.9300	C62—H62	0.9300
C29—C30	1.377 (4)	C63—C68	1.379 (4)
C29—C34	1.378 (4)	C63—C64	1.383 (4)
C30—C31	1.381 (4)	C64—C65	1.372 (5)
C30—H30	0.9300	C64—H64	0.9300
C31—C32	1.371 (5)	C65—C66	1.359 (6)
C31—H31	0.9300	C65—H65	0.9300
C32—C33	1.371 (5)	C66—C67	1.357 (6)
C32—H32	0.9300	C66—H66	0.9300
C33—C34	1.379 (4)	C67—C68	1.395 (5)
C33—H33	0.9300	C67—H67	0.9300
C34—H34	0.9300	C68—H68	0.9300
C12—N1—C4	113.0 (2)	C46—N3—C38	113.1 (2)
C12—N1—C1	117.0 (2)	C46—N3—C35	114.7 (2)
C4—N1—C1	109.5 (2)	C38—N3—C35	108.2 (2)
C13—N2—C14	112.1 (2)	C47—N4—C48	112.0 (2)
C13—N2—H2	124.0	C47—N4—H4A	124.0
C14—N2—H2	124.0	C48—N4—H4A	124.0
N1—C1—C19	111.70 (19)	N3—C35—C53	111.8 (2)
N1—C1—C13	113.62 (19)	N3—C35—C47	113.88 (19)
C19—C1—C13	102.1 (2)	C53—C35—C47	101.61 (19)
N1—C1—C2	103.47 (17)	N3—C35—C36	103.04 (18)
C19—C1—C2	117.0 (2)	C53—C35—C36	118.90 (19)
C13—C1—C2	109.28 (19)	C47—C35—C36	108.01 (18)
C20—C2—C3	114.39 (19)	C54—C36—C37	113.74 (19)
C20—C2—C1	115.25 (19)	C54—C36—C35	116.6 (2)

C3—C2—C1	104.88 (18)	C37—C36—C35	105.67 (18)
C20—C2—H2A	107.3	C54—C36—H36	106.7
C3—C2—H2A	107.3	C37—C36—H36	106.7
C1—C2—H2A	107.3	C35—C36—H36	106.7
C4—C3—C29	114.4 (2)	C63—C37—C38	113.5 (2)
C4—C3—C2	102.78 (18)	C63—C37—C36	115.9 (2)
C29—C3—C2	113.96 (19)	C38—C37—C36	103.11 (18)
C4—C3—H3	108.5	C63—C37—H37	108.0
C29—C3—H3	108.5	C38—C37—H37	108.0
C2—C3—H3	108.5	C36—C37—H37	108.0
N1—C4—C3	102.61 (18)	N3—C38—C39	110.1 (2)
N1—C4—C5	108.03 (19)	N3—C38—C37	102.06 (18)
C3—C4—C5	115.6 (2)	C39—C38—C37	115.0 (2)
N1—C4—H4	110.1	N3—C38—H38	109.8
C3—C4—H4	110.1	C39—C38—H38	109.8
C5—C4—H4	110.1	C37—C38—H38	109.8
C6—C5—C4	111.3 (2)	C40—C39—C38	112.4 (2)
C6—C5—H5A	109.4	C40—C39—H39A	109.1
C4—C5—H5A	109.4	C38—C39—H39A	109.1
C6—C5—H5B	109.4	C40—C39—H39B	109.1
C4—C5—H5B	109.4	C38—C39—H39B	109.1
H5A—C5—H5B	108.0	H39A—C39—H39B	107.9
C11—C6—C7	118.6 (3)	C45—C40—C41	119.0 (3)
C11—C6—C5	120.5 (2)	C45—C40—C39	121.1 (2)
C7—C6—C5	121.0 (3)	C41—C40—C39	119.8 (3)
C8—C7—C6	121.3 (3)	C42—C41—C40	120.7 (3)
C8—C7—H7	119.3	C42—C41—H41	119.7
C6—C7—H7	119.3	C40—C41—H41	119.7
C9—C8—C7	120.3 (3)	C41—C42—C43	120.3 (3)
C9—C8—H8	119.9	C41—C42—H42	119.9
C7—C8—H8	119.9	C43—C42—H42	119.9
C8—C9—C10	119.6 (3)	C42—C43—C44	119.9 (3)
C8—C9—H9	120.2	C42—C43—H43	120.0
C10—C9—H9	120.2	C44—C43—H43	120.0
C9—C10—C11	121.1 (3)	C43—C44—C45	120.0 (3)
C9—C10—H10	119.4	C43—C44—H44	120.0
C11—C10—H10	119.4	C45—C44—H44	120.0
C10—C11—C6	119.1 (3)	C40—C45—C44	120.0 (3)
C10—C11—C12	119.0 (3)	C40—C45—C46	121.6 (2)
C6—C11—C12	121.9 (2)	C44—C45—C46	118.3 (3)
N1—C12—C11	111.5 (2)	N3—C46—C45	110.9 (2)
N1—C12—H12A	109.3	N3—C46—H46A	109.5
C11—C12—H12A	109.3	C45—C46—H46A	109.5
N1—C12—H12B	109.3	N3—C46—H46B	109.5
C11—C12—H12B	109.3	C45—C46—H46B	109.5
H12A—C12—H12B	108.0	H46A—C46—H46B	108.0
O1—C13—N2	126.8 (3)	O3—C47—N4	126.3 (2)
O1—C13—C1	126.1 (2)	O3—C47—C35	126.2 (2)
N2—C13—C1	107.1 (2)	N4—C47—C35	107.4 (2)

C19—C14—C15	121.0 (3)	C49—C48—C53	121.8 (3)
C19—C14—N2	109.8 (2)	C49—C48—N4	128.4 (3)
C15—C14—N2	129.2 (3)	C53—C48—N4	109.8 (2)
C16—C15—C14	117.6 (3)	C50—C49—C48	117.4 (3)
C16—C15—H15	121.2	C50—C49—H49	121.3
C14—C15—H15	121.2	C48—C49—H49	121.3
C15—C16—C17	122.4 (3)	C49—C50—C51	121.9 (3)
C15—C16—H16	118.8	C49—C50—H50	119.0
C17—C16—H16	118.8	C51—C50—H50	119.0
C16—C17—C18	119.8 (3)	C52—C51—C50	119.7 (3)
C16—C17—H17	120.1	C52—C51—H51	120.1
C18—C17—H17	120.1	C50—C51—H51	120.1
C19—C18—C17	118.8 (3)	C53—C52—C51	119.2 (3)
C19—C18—H18	120.6	C53—C52—H52	120.4
C17—C18—H18	120.6	C51—C52—H52	120.4
C18—C19—C14	120.3 (2)	C52—C53—C48	119.8 (2)
C18—C19—C1	130.7 (2)	C52—C53—C35	131.2 (2)
C14—C19—C1	108.9 (2)	C48—C53—C35	109.0 (2)
O2—C20—C21	122.5 (2)	O4—C54—C55	122.1 (2)
O2—C20—C2	120.6 (2)	O4—C54—C36	120.5 (2)
C21—C20—C2	116.9 (2)	C55—C54—C36	117.2 (2)
C22—C21—C20	122.8 (2)	C56—C55—C54	122.0 (2)
C22—C21—H21	118.6	C56—C55—H55	119.0
C20—C21—H21	118.6	C54—C55—H55	119.0
C21—C22—C23	127.0 (2)	C55—C56—C57	127.9 (3)
C21—C22—H22	116.5	C55—C56—H56	116.0
C23—C22—H22	116.5	C57—C56—H56	116.0
C28—C23—C24	118.1 (3)	C62—C57—C58	118.0 (3)
C28—C23—C22	119.9 (2)	C62—C57—C56	118.8 (3)
C24—C23—C22	122.0 (2)	C58—C57—C56	123.2 (3)
C25—C24—C23	120.8 (3)	C57—C58—C59	120.4 (3)
C25—C24—H24	119.6	C57—C58—H58	119.8
C23—C24—H24	119.6	C59—C58—H58	119.8
C26—C25—C24	120.4 (3)	C60—C59—C58	120.3 (4)
C26—C25—H25	119.8	C60—C59—H59	119.8
C24—C25—H25	119.8	C58—C59—H59	119.8
C27—C26—C25	119.4 (3)	C61—C60—C59	119.5 (4)
C27—C26—H26	120.3	C61—C60—H60	120.3
C25—C26—H26	120.3	C59—C60—H60	120.3
C26—C27—C28	120.9 (3)	C60—C61—C62	120.6 (4)
C26—C27—H27	119.6	C60—C61—H61	119.7
C28—C27—H27	119.6	C62—C61—H61	119.7
C27—C28—C23	120.5 (3)	C57—C62—C61	121.1 (4)
C27—C28—H28	119.8	C57—C62—H62	119.4
C23—C28—H28	119.8	C61—C62—H62	119.4
C30—C29—C34	118.3 (2)	C68—C63—C64	117.7 (3)
C30—C29—C3	121.5 (2)	C68—C63—C37	120.5 (3)
C34—C29—C3	120.2 (2)	C64—C63—C37	121.7 (2)
C29—C30—C31	120.7 (3)	C65—C64—C63	121.4 (3)

C29—C30—H30	119.7	C65—C64—H64	119.3
C31—C30—H30	119.7	C63—C64—H64	119.3
C32—C31—C30	120.4 (3)	C66—C65—C64	120.2 (4)
C32—C31—H31	119.8	C66—C65—H65	119.9
C30—C31—H31	119.8	C64—C65—H65	119.9
C33—C32—C31	119.3 (3)	C67—C66—C65	119.9 (3)
C33—C32—H32	120.3	C67—C66—H66	120.1
C31—C32—H32	120.3	C65—C66—H66	120.1
C32—C33—C34	120.2 (3)	C66—C67—C68	120.4 (3)
C32—C33—H33	119.9	C66—C67—H67	119.8
C34—C33—H33	119.9	C68—C67—H67	119.8
C29—C34—C33	121.0 (3)	C63—C68—C67	120.3 (4)
C29—C34—H34	119.5	C63—C68—H68	119.8
C33—C34—H34	119.5	C67—C68—H68	119.8
C12—N1—C1—C19	85.8 (3)	C46—N3—C35—C53	−72.9 (3)
C4—N1—C1—C19	−143.9 (2)	C38—N3—C35—C53	159.84 (19)
C12—N1—C1—C13	−29.1 (3)	C46—N3—C35—C47	41.5 (3)
C4—N1—C1—C13	101.2 (2)	C38—N3—C35—C47	−85.7 (2)
C12—N1—C1—C2	−147.5 (2)	C46—N3—C35—C36	158.3 (2)
C4—N1—C1—C2	−17.2 (2)	C38—N3—C35—C36	31.0 (2)
N1—C1—C2—C20	−135.5 (2)	N3—C35—C36—C54	121.4 (2)
C19—C1—C2—C20	−12.2 (3)	C53—C35—C36—C54	−2.9 (3)
C13—C1—C2—C20	103.1 (2)	C47—C35—C36—C54	−117.8 (2)
N1—C1—C2—C3	−8.8 (2)	N3—C35—C36—C37	−6.1 (2)
C19—C1—C2—C3	114.5 (2)	C53—C35—C36—C37	−130.4 (2)
C13—C1—C2—C3	−130.2 (2)	C47—C35—C36—C37	114.7 (2)
C20—C2—C3—C4	157.62 (19)	C54—C36—C37—C63	87.3 (2)
C1—C2—C3—C4	30.4 (2)	C35—C36—C37—C63	−143.5 (2)
C20—C2—C3—C29	−78.0 (3)	C54—C36—C37—C38	−148.1 (2)
C1—C2—C3—C29	154.8 (2)	C35—C36—C37—C38	−18.9 (2)
C12—N1—C4—C3	169.2 (2)	C46—N3—C38—C39	65.6 (3)
C1—N1—C4—C3	36.9 (2)	C35—N3—C38—C39	−166.3 (2)
C12—N1—C4—C5	−68.2 (3)	C46—N3—C38—C37	−171.9 (2)
C1—N1—C4—C5	159.5 (2)	C35—N3—C38—C37	−43.7 (2)
C29—C3—C4—N1	−164.70 (19)	C63—C37—C38—N3	163.32 (19)
C2—C3—C4—N1	−40.6 (2)	C36—C37—C38—N3	37.1 (2)
C29—C3—C4—C5	78.0 (3)	C63—C37—C38—C39	−77.6 (3)
C2—C3—C4—C5	−157.9 (2)	C36—C37—C38—C39	156.2 (2)
N1—C4—C5—C6	51.7 (3)	N3—C38—C39—C40	−44.3 (3)
C3—C4—C5—C6	166.0 (2)	C37—C38—C39—C40	−158.9 (2)
C4—C5—C6—C11	−20.3 (4)	C38—C39—C40—C45	14.0 (4)
C4—C5—C6—C7	159.6 (3)	C38—C39—C40—C41	−166.2 (2)
C11—C6—C7—C8	0.9 (5)	C45—C40—C41—C42	−0.7 (4)
C5—C6—C7—C8	−179.0 (3)	C39—C40—C41—C42	179.4 (3)
C6—C7—C8—C9	−0.9 (5)	C40—C41—C42—C43	−0.2 (5)
C7—C8—C9—C10	1.3 (6)	C41—C42—C43—C44	0.6 (5)
C8—C9—C10—C11	−1.6 (5)	C42—C43—C44—C45	0.0 (5)
C9—C10—C11—C6	1.6 (5)	C41—C40—C45—C44	1.3 (4)
C9—C10—C11—C12	180.0 (3)	C39—C40—C45—C44	−178.9 (3)

C7—C6—C11—C10	−1.2 (4)	C41—C40—C45—C46	179.3 (3)
C5—C6—C11—C10	178.7 (3)	C39—C40—C45—C46	−0.9 (4)
C7—C6—C11—C12	−179.5 (3)	C43—C44—C45—C40	−0.9 (4)
C5—C6—C11—C12	0.3 (4)	C43—C44—C45—C46	−179.0 (3)
C4—N1—C12—C11	47.0 (3)	C38—N3—C46—C45	−50.7 (3)
C1—N1—C12—C11	175.6 (2)	C35—N3—C46—C45	−175.4 (2)
C10—C11—C12—N1	169.0 (3)	C40—C45—C46—N3	18.2 (4)
C6—C11—C12—N1	−12.7 (4)	C44—C45—C46—N3	−163.8 (3)
C14—N2—C13—O1	−178.8 (3)	C48—N4—C47—O3	−178.2 (3)
C14—N2—C13—C1	0.8 (3)	C48—N4—C47—C35	2.8 (3)
N1—C1—C13—O1	−60.6 (3)	N3—C35—C47—O3	57.4 (3)
C19—C1—C13—O1	179.0 (2)	C53—C35—C47—O3	177.8 (3)
C2—C1—C13—O1	54.4 (3)	C36—C35—C47—O3	−56.4 (3)
N1—C1—C13—N2	119.8 (2)	N3—C35—C47—N4	−123.6 (2)
C19—C1—C13—N2	−0.6 (2)	C53—C35—C47—N4	−3.2 (2)
C2—C1—C13—N2	−125.2 (2)	C36—C35—C47—N4	122.6 (2)
C13—N2—C14—C19	−0.6 (3)	C47—N4—C48—C49	178.8 (3)
C13—N2—C14—C15	178.0 (3)	C47—N4—C48—C53	−1.1 (3)
C19—C14—C15—C16	1.0 (5)	C53—C48—C49—C50	0.7 (5)
N2—C14—C15—C16	−177.4 (3)	N4—C48—C49—C50	−179.1 (3)
C14—C15—C16—C17	−0.2 (6)	C48—C49—C50—C51	1.2 (6)
C15—C16—C17—C18	−0.8 (6)	C49—C50—C51—C52	−2.2 (6)
C16—C17—C18—C19	1.1 (5)	C50—C51—C52—C53	1.1 (5)
C17—C18—C19—C14	−0.3 (4)	C51—C52—C53—C48	0.8 (4)
C17—C18—C19—C1	177.0 (3)	C51—C52—C53—C35	179.9 (3)
C15—C14—C19—C18	−0.7 (4)	C49—C48—C53—C52	−1.8 (4)
N2—C14—C19—C18	178.0 (2)	N4—C48—C53—C52	178.1 (2)
C15—C14—C19—C1	−178.6 (3)	C49—C48—C53—C35	179.0 (3)
N2—C14—C19—C1	0.1 (3)	N4—C48—C53—C35	−1.2 (3)
N1—C1—C19—C18	61.0 (3)	N3—C35—C53—C52	−54.7 (3)
C13—C1—C19—C18	−177.2 (3)	C47—C35—C53—C52	−176.6 (3)
C2—C1—C19—C18	−58.0 (3)	C36—C35—C53—C52	65.2 (4)
N1—C1—C19—C14	−121.5 (2)	N3—C35—C53—C48	124.4 (2)
C13—C1—C19—C14	0.3 (2)	C47—C35—C53—C48	2.6 (2)
C2—C1—C19—C14	119.6 (2)	C36—C35—C53—C48	−115.7 (2)
C3—C2—C20—O2	2.9 (3)	C37—C36—C54—O4	−2.0 (3)
C1—C2—C20—O2	124.6 (2)	C35—C36—C54—O4	−125.4 (3)
C3—C2—C20—C21	179.9 (2)	C37—C36—C54—C55	−178.1 (2)
C1—C2—C20—C21	−58.4 (3)	C35—C36—C54—C55	58.5 (3)
O2—C20—C21—C22	−13.8 (4)	O4—C54—C55—C56	14.1 (4)
C2—C20—C21—C22	169.3 (2)	C36—C54—C55—C56	−169.8 (2)
C20—C21—C22—C23	−177.4 (2)	C54—C55—C56—C57	177.0 (3)
C21—C22—C23—C28	178.6 (3)	C55—C56—C57—C62	−172.2 (3)
C21—C22—C23—C24	−1.6 (5)	C55—C56—C57—C58	8.0 (5)
C28—C23—C24—C25	−0.4 (5)	C62—C57—C58—C59	0.8 (5)
C22—C23—C24—C25	179.8 (3)	C56—C57—C58—C59	−179.4 (3)
C23—C24—C25—C26	−0.2 (5)	C57—C58—C59—C60	1.5 (6)
C24—C25—C26—C27	0.7 (5)	C58—C59—C60—C61	−2.9 (7)
C25—C26—C27—C28	−0.5 (5)	C59—C60—C61—C62	2.0 (8)

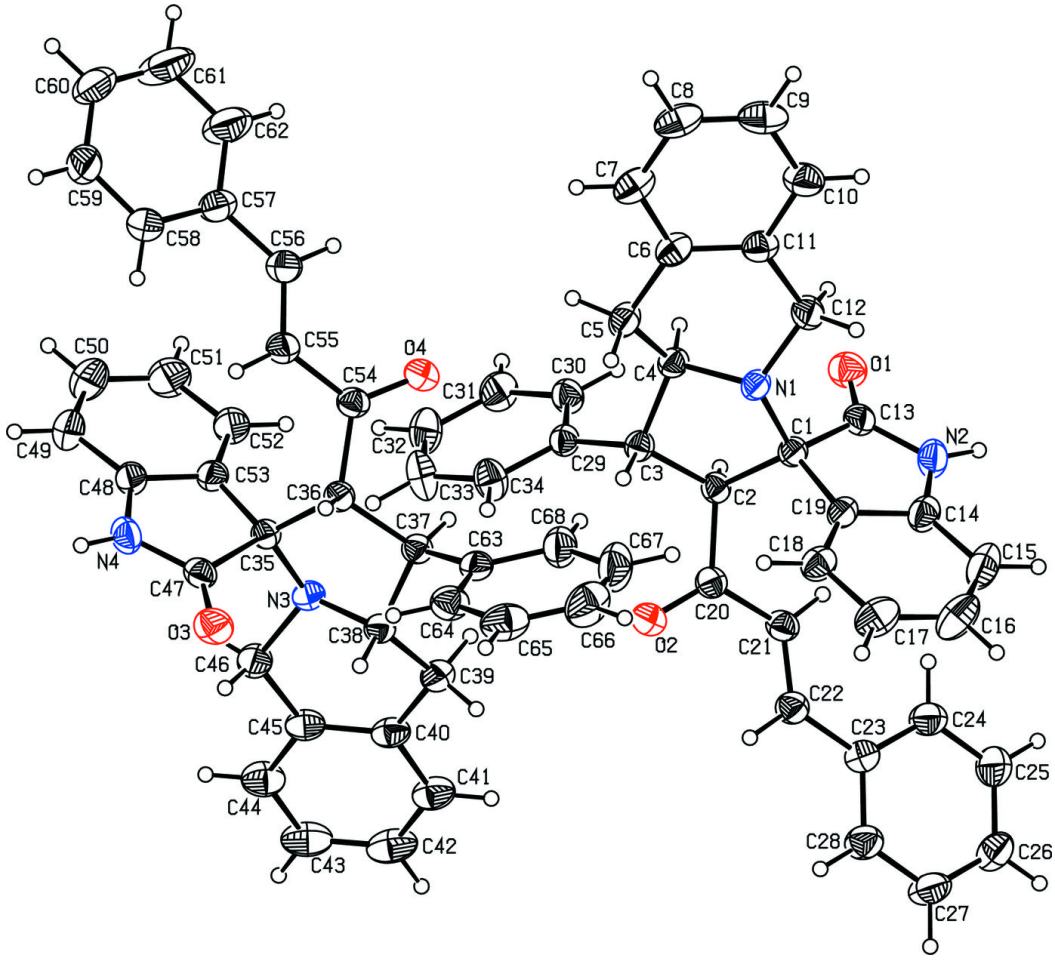
C26—C27—C28—C23	−0.1 (5)	C58—C57—C62—C61	−1.8 (7)
C24—C23—C28—C27	0.5 (5)	C56—C57—C62—C61	178.4 (4)
C22—C23—C28—C27	−179.7 (3)	C60—C61—C62—C57	0.4 (9)
C4—C3—C29—C30	56.8 (3)	C38—C37—C63—C68	114.1 (3)
C2—C3—C29—C30	−61.0 (3)	C36—C37—C63—C68	−126.8 (3)
C4—C3—C29—C34	−123.3 (3)	C38—C37—C63—C64	−63.6 (3)
C2—C3—C29—C34	118.8 (3)	C36—C37—C63—C64	55.4 (3)
C34—C29—C30—C31	0.4 (4)	C68—C63—C64—C65	−1.2 (4)
C3—C29—C30—C31	−179.7 (3)	C37—C63—C64—C65	176.6 (3)
C29—C30—C31—C32	−0.5 (5)	C63—C64—C65—C66	0.4 (5)
C30—C31—C32—C33	0.0 (5)	C64—C65—C66—C67	0.5 (6)
C31—C32—C33—C34	0.6 (6)	C65—C66—C67—C68	−0.4 (6)
C30—C29—C34—C33	0.2 (5)	C64—C63—C68—C67	1.2 (4)
C3—C29—C34—C33	−179.6 (3)	C37—C63—C68—C67	−176.6 (3)
C32—C33—C34—C29	−0.8 (6)	C66—C67—C68—C63	−0.4 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.86	2.19	3.023 (3)	164
N4—H4A···O2^{ii}	0.86	2.23	3.047 (3)	159
C2—H2A···O1	0.98	2.57	3.034 (3)	109
C12—H12A···O1	0.97	2.54	3.201 (3)	125
C37—H37···O4	0.98	2.44	2.784 (3)	100
C56—H56···O4	0.93	2.51	2.830 (4)	101

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

Fig. 1


C60
C61
C62
C59
C57
C58
C56
C55
C50
C51
O4
C54
C31
C30
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C9
C7
C10
C6
C11
C5
C12
C4
N1
O1
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C26
C43
C27

Fig. 2