(2E)-3-(2-Chlorophenyl)-1-[1'-(2-chlorophenyl)indoline-3-spiro-3'-perhydropyrrolo[1,2-a]indol-2'-yl]prop-2-en-1-one

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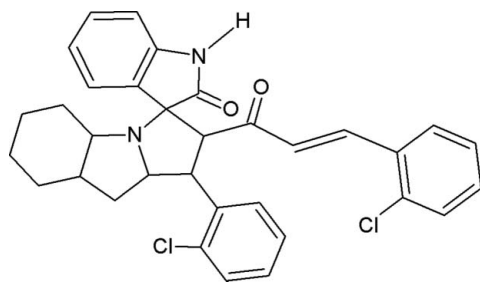Correspondence e-mail: a_spandian@yahoo.com

In the title compound, $C_{33}H_{30}Cl_2N_2O_2$, both five-membered rings in the indolizidine system adopt envelope conformations. The oxindole ring system is essentially planar. The crystal packing is characterized by N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Beddoes *et al.* (1986); Bowman *et al.* (1996); Cremer & Pople (1975); Esker & Newcomb (1993); Fallis & Brinza (1997); Guindon *et al.* (2001); Jeyabharathi *et al.* (2001); Nardelli (1983); Rajeswaran *et al.* (1999); Seshadri *et al.* (2003).

Experimental

Crystal data

$C_{33}H_{30}Cl_2N_2O_2$	c = 24.0094 (17) Å
M_r = 557.49	β = 100.912 (3)°
Monoclinic, $C2/c$	V = 5642.1 (6) Å^3
a = 24.8293 (16) Å	Z = 8
b = 9.6387 (6) Å	Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}	0.24 × 0.20 × 0.18 mm
T = 293 (2) K	

Data collection

Bruker APEXII CCD area-detector diffractometer	35182 measured reflections
	8131 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	5246 reflections with $I > 2\sigma(I)$
T_{min} = 0.939, T_{max} = 0.954	R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052	472 parameters
$wR(F^2)$ = 0.164	All H-atom parameters refined
S = 1.02	$\Delta\rho_{max}$ = 0.65 e Å^{-3}
8131 reflections	$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···Cl2	0.968 (18)	2.515 (18)	3.0895 (16)	118.0 (13)
N19—H19···O1[i]	0.86 (2)	2.54 (2)	3.222 (2)	136.0 (18)
C17—H17···O1[i]	0.92 (3)	2.50 (3)	3.253 (3)	140 (2)
C22—H22···O2[ii]	0.97 (2)	2.57 (2)	3.522 (2)	166.3 (17)
C25—H25···O2[ii]	0.95 (3)	2.43 (3)	3.366 (3)	171 (2)

Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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supplementary materials

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P. Ramesh, S. Murugavel, A. S. Pandi, R. Murugan and S. S. Narayanan

Comment

Oxindole derivatives are found to be potent aldose reductase inhibitors (ARIs), which help to treat and prevent diabetic complications arising from elevated levels of sorbitol (Rajeswaran *et al.*, 1999). Pyrrolidine derivatives are present in a large number of biologically active natural products and numerous therapeutic agents. Radical cyclizations have emerged as a useful synthetic tool and have been reported in the synthesis of alkaloids and related pyrrolidinic compounds *via* the generation and trapping of nitrogen-centred radicals *e.g.* aminyl, iminyl, amidyl radicals *etc*. (Esker & Newcomb, 1993; Fallis & Brinza, 1997; Bowman *et al.*, 1996; Guindon *et al.*, 2001).

The bond lengths in the indolizidine moiety are slightly longer than the values reported for similar structures (Seshadri *et al.*, 2003; Jeyabharathi *et al.*, 2001). This may be due to steric forces caused by the bulky substituents on the indolizidine moiety. The sum of angle at N1 of the indolizidine ring (335.3°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986), and the sum of angles at N19 of the oxindole moiety (359.8°) is in accordance with sp^2 hybridization.

Both the five-membered rings of the indolizidine moiety adopt envelope conformations, with the the puckering parameters q_2 and φ (Cremer & Pople, 1975) and the smallest displacement asymmetric parameters, Δ, (Nardelli *et al.*, 1983) as follows: for ring A are q_2=0.463 (2) Å, φ=68.4 (2)°, and Δ_s(C3)=4.6 (2); for ring B are q_2=0.454 (2) Å, φ=280.8 (2)°, Δ_s()=6.0 (2). Oxindole ring systems are essentially planar, with atoms O1 displaced by 0.067 (1) Å.

The molecule is stabilized by intra molecular C—H···Cl interactions and the crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds. The molecule at (*x*, *y*, *z*) and (1/2 − *x*, 3/2 − *y*, *z*) are linked by C22—H22···O2 hydrogen bonds into centrosymmetric R_2^2(8) dimers. Atom C25 of the molecule at (*x*, *y*, *z*) donate one proton to atom O2 molecule at (1/2 − *x*, 3/2 − *y*, *z*), forming a R_2^2(14) dimers. These two dimers R_2^2(8) and R_2^2(14) are cross-linked along [0 1 0] direction through C—H···O and N—H···O intermolecular hydrogen bonds.

Experimental

To solution of (1E, 6E)-4-(2-chloro phenylidine)-1, 7-bis (2-cholorophenyl) hepta-1, 6diene- 3, 5dione (1 mmol), isatin (1 mmol) and octa hydro 1*H*-indole-2-carboxylate and in aqeous methanol (20 ml) was refluxed until the disappearence of starting material as evidenced by TLC. The final product was recrystallized in ethanol and choloroform(2:8)

Refinement

All H atoms were freely refined.
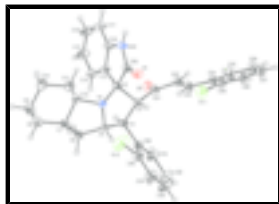
supplementary materials

Figures

Fig. 1. The molecular configuration and atom numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitory radii.

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Crystal data

$C_{33}H_{30}Cl_2N_2O_2$	$Z = 8$
$M_r = 557.49$	$F_{000} = 2336$
Monoclinic, $C2/c$	$D_x = 1.313$ Mg m^{-3}
Hall symbol: -C 2yc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 24.8293$ (16) Å	Cell parameters from 10569 reflections
$b = 9.6387$ (6) Å	$\mu = 0.26$ mm^{-1}
$c = 24.0094$ (17) Å	$T = 293$ (2) K
$\beta = 100.912$ (3)º	Block, colourless
$V = 5642.1$ (6) Å^3	$0.24 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	8131 independent reflections
Radiation source: fine-focus sealed tube	5246 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 293(2)$ K	$\theta_{max} = 29.9$º
ω scans	$\theta_{min} = 1.7$º
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -33 \rightarrow 34$
$T_{min} = 0.939$, $T_{max} = 0.954$	$k = -13 \rightarrow 13$
35182 measured reflections	$l = -33 \rightarrow 30$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	All H-atom parameters refined
$wR(F^2) = 0.164$	$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 3.0845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.016$

8131 reflections

472 parameters

Primary atom site location: structure-invariant direct methods

$\Delta\rho_{max} = 0.65$ e Å^{-3}

$\Delta\rho_{min} = -0.60$ 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
Cl1	0.08524 (3)	0.95549 (7)	0.15832 (3)	0.06593 (18)
Cl2	0.40447 (3)	1.11218 (10)	0.05770 (4)	0.0987 (3)
O1	0.22538 (6)	0.81919 (14)	0.23356 (6)	0.0487 (3)
O2	0.30791 (6)	0.82469 (18)	0.06638 (6)	0.0594 (4)
N1	0.33152 (6)	0.96102 (12)	0.24171 (5)	0.0296 (3)
C2	0.31207 (6)	0.85115 (15)	0.20058 (6)	0.0289 (3)
C3	0.28838 (7)	0.93942 (16)	0.14758 (6)	0.0301 (3)
H3	0.2558 (7)	0.9847 (18)	0.1567 (7)	0.029 (4)*
C4	0.33421 (7)	1.04669 (15)	0.14776 (7)	0.0309 (3)
H4	0.3649 (7)	1.0012 (19)	0.1358 (7)	0.030 (4)*
C5	0.34852 (7)	1.08439 (15)	0.21083 (7)	0.0313 (3)
H5	0.3254 (7)	1.1636 (18)	0.2160 (7)	0.028 (4)*
C6	0.40806 (8)	1.1131 (2)	0.23944 (8)	0.0429 (4)
H6A	0.4345 (8)	1.054 (2)	0.2238 (8)	0.040 (5)*
H6B	0.4189 (9)	1.213 (2)	0.2369 (9)	0.054 (6)*
C7	0.40712 (8)	1.06498 (19)	0.29989 (8)	0.0401 (4)
H7	0.3823 (8)	1.128 (2)	0.3149 (8)	0.036 (5)*
C8	0.45964 (10)	1.0451 (3)	0.34284 (10)	0.0556 (5)
H8A	0.4788 (11)	1.133 (3)	0.3502 (12)	0.075 (8)*
H8B	0.4822 (9)	0.977 (2)	0.3284 (9)	0.054 (6)*
C9	0.44547 (12)	0.9898 (3)	0.39755 (10)	0.0644 (6)
H9A	0.4801 (13)	0.973 (3)	0.4237 (13)	0.092 (9)*
H9B	0.4235 (12)	1.061 (3)	0.4127 (12)	0.084 (9)*
C10	0.41086 (13)	0.8586 (3)	0.38859 (11)	0.0681 (7)
H10A	0.4327 (10)	0.783 (3)	0.3784 (10)	0.065 (7)*
H10B	0.4016 (11)	0.824 (3)	0.4235 (13)	0.078 (8)*
C11	0.35993 (11)	0.8740 (3)	0.34230 (9)	0.0572 (6)
H11A	0.3420 (11)	0.784 (3)	0.3330 (11)	0.070 (7)*

H11B	0.3351 (11)	0.942 (3)	0.3543 (11)	0.075 (8)*
C12	0.37641 (7)	0.92858 (17)	0.28916 (7)	0.0357 (4)
H12	0.4018 (8)	0.861 (2)	0.2767 (8)	0.037 (5)*
C13	0.35180 (7)	0.73744 (16)	0.19144 (7)	0.0329 (3)
C14	0.40243 (8)	0.74020 (19)	0.17579 (9)	0.0441 (4)
H14	0.4169 (8)	0.823 (2)	0.1667 (9)	0.046 (5)*
C15	0.43018 (10)	0.6167 (2)	0.17180 (10)	0.0572 (6)
H15	0.4648 (11)	0.619 (2)	0.1589 (10)	0.064 (7)*
C16	0.40719 (11)	0.4924 (2)	0.18328 (11)	0.0636 (7)
H16	0.4268 (11)	0.410 (3)	0.1833 (11)	0.075 (8)*
C17	0.35686 (10)	0.4872 (2)	0.19983 (10)	0.0549 (5)
H17	0.3410 (10)	0.406 (3)	0.2082 (11)	0.067 (7)*
C18	0.32983 (8)	0.61061 (16)	0.20366 (7)	0.0374 (4)
N19	0.27867 (7)	0.63167 (15)	0.21980 (7)	0.0405 (4)
H19	0.2591 (9)	0.568 (2)	0.2314 (9)	0.046 (6)*
C20	0.26575 (7)	0.76780 (16)	0.22028 (7)	0.0334 (3)
C21	0.27287 (7)	0.85917 (18)	0.09258 (7)	0.0368 (4)
C22	0.21529 (8)	0.8265 (2)	0.06962 (8)	0.0427 (4)
H22	0.2098 (8)	0.770 (2)	0.0357 (9)	0.052 (6)*
C23	0.17201 (8)	0.8731 (2)	0.08916 (8)	0.0442 (4)
H23	0.1781 (8)	0.923 (2)	0.1225 (10)	0.050 (6)*
C24	0.11472 (8)	0.8535 (2)	0.06237 (8)	0.0446 (4)
C25	0.10014 (10)	0.8012 (3)	0.00719 (10)	0.0582 (6)
H25	0.1293 (11)	0.769 (3)	−0.0099 (11)	0.078 (8)*
C26	0.04625 (10)	0.7835 (3)	−0.01841 (11)	0.0657 (6)
H26	0.0361 (11)	0.749 (3)	−0.0580 (12)	0.076 (8)*
C27	0.00531 (11)	0.8181 (3)	0.00991 (11)	0.0657 (7)
H27	−0.0299 (13)	0.807 (3)	−0.0057 (13)	0.093 (10)*
C28	0.01747 (9)	0.8710 (3)	0.06405 (11)	0.0587 (6)
H28	−0.0107 (10)	0.894 (2)	0.0856 (10)	0.057 (6)*
C29	0.07196 (8)	0.8891 (2)	0.08982 (9)	0.0466 (4)
C30	0.31806 (7)	1.16913 (17)	0.10870 (7)	0.0370 (4)
C31	0.27304 (10)	1.2499 (2)	0.11304 (10)	0.0568 (6)
H31	0.2548 (10)	1.227 (3)	0.1423 (11)	0.076 (8)*
C32	0.25854 (14)	1.3647 (3)	0.07904 (13)	0.0795 (9)
H32	0.2294 (12)	1.415 (3)	0.0855 (12)	0.080 (9)*
C33	0.28963 (14)	1.4019 (3)	0.04007 (13)	0.0836 (9)
H33	0.2787 (14)	1.478 (4)	0.0178 (15)	0.111 (11)*
C34	0.33413 (13)	1.3252 (3)	0.03449 (12)	0.0757 (8)
H34	0.3565 (13)	1.349 (3)	0.0109 (14)	0.100 (10)*
C35	0.34795 (9)	1.2089 (2)	0.06815 (9)	0.0523 (5)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0649 (4)	0.0765 (4)	0.0587 (3)	0.0066 (3)	0.0176 (3)	−0.0165 (3)
Cl2	0.0773 (5)	0.1266 (7)	0.1101 (6)	0.0276 (4)	0.0634 (4)	0.0608 (5)
O1	0.0472 (8)	0.0453 (7)	0.0614 (9)	−0.0026 (6)	0.0300 (7)	0.0027 (6)

O2	0.0513 (8)	0.0846 (11)	0.0458 (8)	−0.0024 (7)	0.0181 (7)	−0.0272 (7)
N1	0.0393 (7)	0.0239 (6)	0.0263 (6)	−0.0026 (5)	0.0081 (5)	0.0006 (5)
C2	0.0352 (8)	0.0245 (7)	0.0296 (8)	−0.0024 (6)	0.0123 (6)	−0.0002 (5)
C3	0.0352 (8)	0.0289 (7)	0.0278 (8)	−0.0014 (6)	0.0102 (6)	−0.0005 (6)
C4	0.0381 (9)	0.0270 (7)	0.0298 (8)	−0.0008 (6)	0.0121 (7)	0.0034 (6)
C5	0.0415 (9)	0.0214 (7)	0.0325 (8)	−0.0019 (6)	0.0110 (7)	0.0021 (5)
C6	0.0479 (11)	0.0362 (9)	0.0440 (10)	−0.0123 (8)	0.0072 (8)	0.0020 (7)
C7	0.0455 (10)	0.0351 (8)	0.0382 (9)	−0.0029 (7)	0.0043 (8)	−0.0028 (7)
C8	0.0527 (13)	0.0540 (12)	0.0541 (13)	−0.0059 (10)	−0.0051 (10)	−0.0042 (10)
C9	0.0747 (17)	0.0651 (14)	0.0440 (12)	0.0007 (12)	−0.0131 (12)	0.0005 (10)
C10	0.0857 (19)	0.0690 (16)	0.0419 (12)	−0.0066 (14)	−0.0074 (12)	0.0171 (11)
C11	0.0666 (15)	0.0648 (14)	0.0371 (11)	−0.0145 (12)	0.0024 (10)	0.0153 (10)
C12	0.0423 (10)	0.0312 (8)	0.0323 (8)	−0.0002 (7)	0.0037 (7)	0.0014 (6)
C13	0.0425 (9)	0.0249 (7)	0.0333 (8)	0.0010 (6)	0.0125 (7)	−0.0001 (6)
C14	0.0493 (11)	0.0319 (9)	0.0566 (12)	0.0031 (7)	0.0244 (9)	0.0042 (8)
C15	0.0636 (14)	0.0463 (11)	0.0713 (15)	0.0165 (10)	0.0374 (12)	0.0082 (10)
C16	0.0875 (17)	0.0362 (10)	0.0772 (16)	0.0214 (11)	0.0414 (14)	0.0095 (10)
C17	0.0811 (15)	0.0266 (9)	0.0635 (13)	0.0026 (9)	0.0305 (12)	0.0037 (8)
C18	0.0527 (10)	0.0270 (8)	0.0346 (9)	−0.0027 (7)	0.0138 (8)	−0.0004 (6)
N19	0.0509 (9)	0.0278 (7)	0.0466 (9)	−0.0101 (6)	0.0189 (7)	0.0019 (6)
C20	0.0409 (9)	0.0311 (8)	0.0306 (8)	−0.0066 (6)	0.0126 (7)	−0.0006 (6)
C21	0.0426 (10)	0.0386 (8)	0.0306 (8)	−0.0029 (7)	0.0103 (7)	−0.0031 (6)
C22	0.0482 (11)	0.0452 (10)	0.0343 (9)	−0.0074 (8)	0.0070 (8)	−0.0067 (7)
C23	0.0465 (11)	0.0517 (11)	0.0345 (9)	−0.0094 (8)	0.0083 (8)	−0.0053 (8)
C24	0.0434 (10)	0.0489 (10)	0.0410 (10)	−0.0082 (8)	0.0066 (8)	0.0012 (8)
C25	0.0507 (13)	0.0787 (16)	0.0454 (12)	−0.0132 (11)	0.0094 (10)	−0.0086 (11)
C26	0.0565 (14)	0.0903 (18)	0.0471 (13)	−0.0210 (12)	0.0015 (11)	−0.0015 (12)
C27	0.0457 (13)	0.0854 (17)	0.0610 (15)	−0.0144 (12)	−0.0027 (12)	0.0109 (13)
C28	0.0433 (12)	0.0678 (14)	0.0661 (15)	0.0004 (10)	0.0134 (11)	0.0110 (11)
C29	0.0477 (11)	0.0452 (10)	0.0470 (11)	−0.0017 (8)	0.0097 (9)	0.0036 (8)
C30	0.0451 (10)	0.0345 (8)	0.0319 (8)	−0.0037 (7)	0.0086 (7)	0.0055 (6)
C31	0.0698 (15)	0.0514 (12)	0.0550 (13)	0.0172 (10)	0.0266 (11)	0.0201 (10)
C32	0.099 (2)	0.0674 (16)	0.0767 (18)	0.0364 (15)	0.0275 (16)	0.0332 (13)
C33	0.110 (2)	0.0707 (17)	0.0716 (18)	0.0181 (16)	0.0212 (16)	0.0450 (14)
C34	0.088 (2)	0.0803 (18)	0.0648 (16)	−0.0002 (15)	0.0285 (15)	0.0406 (14)
C35	0.0520 (12)	0.0597 (12)	0.0474 (11)	−0.0043 (9)	0.0148 (9)	0.0191 (9)

Geometric parameters (Å, °)

Cl1—C29	1.737 (2)	C13—C18	1.392 (2)
Cl2—C35	1.742 (2)	C14—C15	1.388 (3)
O1—C20	1.214 (2)	C14—H14	0.92 (2)
O2—C21	1.213 (2)	C15—C16	1.378 (3)
N1—C2	1.4659 (19)	C15—H15	0.97 (3)
N1—C12	1.469 (2)	C16—C17	1.382 (3)
N1—C5	1.5035 (19)	C16—H16	0.93 (3)
C2—C13	1.518 (2)	C17—C18	1.378 (3)
C2—C20	1.549 (2)	C17—H17	0.92 (3)
C2—C3	1.551 (2)	C18—N19	1.411 (2)

C3—C21	1.516 (2)	N19—C20	1.351 (2)
C3—C4	1.537 (2)	N19—H19	0.86 (2)
C3—H3	0.980 (18)	C21—C22	1.466 (3)
C4—C30	1.514 (2)	C22—C23	1.329 (3)
C4—C5	1.532 (2)	C22—H22	0.97 (2)
C4—H4	0.968 (18)	C23—C24	1.458 (3)
C5—C6	1.533 (3)	C23—H23	0.92 (2)
C5—H5	0.977 (18)	C24—C29	1.394 (3)
C6—C7	1.528 (3)	C24—C25	1.400 (3)
C6—H6A	1.00 (2)	C25—C26	1.373 (3)
C6—H6B	1.00 (2)	C25—H25	0.95 (3)
C7—C8	1.514 (3)	C26—C27	1.367 (4)
C7—C12	1.517 (2)	C26—H26	0.99 (3)
C7—H7	0.978 (19)	C27—C28	1.375 (4)
C8—C9	1.519 (4)	C27—H27	0.89 (3)
C8—H8A	0.97 (3)	C28—C29	1.388 (3)
C8—H8B	0.97 (2)	C28—H28	0.97 (2)
C9—C10	1.522 (4)	C30—C31	1.382 (3)
C9—H9A	0.98 (3)	C30—C35	1.385 (2)
C9—H9B	0.99 (3)	C31—C32	1.382 (3)
C10—C11	1.525 (3)	C31—H31	0.93 (3)
C10—H10A	0.96 (3)	C32—C33	1.369 (4)
C10—H10B	0.97 (3)	C32—H32	0.91 (3)
C11—C12	1.507 (3)	C33—C34	1.357 (4)
C11—H11A	0.98 (3)	C33—H33	0.92 (4)
C11—H11B	0.98 (3)	C34—C35	1.387 (3)
C12—H12	0.993 (19)	C34—H34	0.90 (3)
C13—C14	1.379 (2)	C35—Cl2	1.742 (2)
C2—N1—C12	118.26 (12)	C14—C13—C2	132.56 (14)
C2—N1—C5	109.06 (12)	C18—C13—C2	108.19 (14)
C12—N1—C5	107.95 (12)	C13—C14—C15	119.51 (18)
N1—C2—C13	118.61 (13)	C13—C14—H14	119.9 (13)
N1—C2—C20	109.76 (12)	C15—C14—H14	120.6 (13)
C13—C2—C20	102.13 (12)	C16—C15—C14	120.2 (2)
N1—C2—C3	100.47 (11)	C16—C15—H15	120.6 (14)
C13—C2—C3	115.67 (12)	C14—C15—H15	119.1 (14)
C20—C2—C3	110.30 (13)	C15—C16—C17	121.36 (19)
C21—C3—C4	114.31 (13)	C15—C16—H16	120.5 (17)
C21—C3—C2	115.41 (13)	C17—C16—H16	117.9 (17)
C4—C3—C2	101.60 (12)	C18—C17—C16	117.80 (19)
C21—C3—H3	109.7 (10)	C18—C17—H17	119.4 (16)
C4—C3—H3	110.0 (10)	C16—C17—H17	122.8 (16)
C2—C3—H3	105.2 (10)	C17—C18—C13	121.94 (18)
C30—C4—C5	114.34 (13)	C17—C18—N19	128.15 (16)
C30—C4—C3	114.43 (14)	C13—C18—N19	109.91 (14)
C5—C4—C3	101.26 (12)	C20—N19—C18	111.69 (14)
C30—C4—H4	107.5 (11)	C20—N19—H19	122.8 (14)
C5—C4—H4	110.7 (10)	C18—N19—H19	125.3 (14)
C3—C4—H4	108.5 (11)	O1—C20—N19	127.43 (15)

N1—C5—C4	105.40 (12)	O1—C20—C2	124.52 (14)
N1—C5—C6	104.99 (13)	N19—C20—C2	108.05 (14)
C4—C5—C6	120.61 (15)	O2—C21—C22	119.38 (16)
N1—C5—H5	109.2 (10)	O2—C21—C3	120.18 (16)
C4—C5—H5	106.2 (10)	C22—C21—C3	120.41 (15)
C6—C5—H5	110.0 (10)	C23—C22—C21	126.20 (17)
C7—C6—C5	100.60 (14)	C23—C22—H22	119.5 (12)
C7—C6—H6A	108.3 (12)	C21—C22—H22	114.2 (12)
C5—C6—H6A	112.1 (11)	C22—C23—C24	125.92 (18)
C7—C6—H6B	113.7 (13)	C22—C23—H23	118.3 (13)
C5—C6—H6B	113.1 (12)	C24—C23—H23	115.8 (13)
H6A—C6—H6B	108.8 (17)	C29—C24—C25	116.90 (18)
C8—C7—C12	110.28 (16)	C29—C24—C23	121.74 (18)
C8—C7—C6	121.29 (18)	C25—C24—C23	121.34 (19)
C12—C7—C6	101.50 (14)	C26—C25—C24	121.6 (2)
C8—C7—H7	110.4 (11)	C26—C25—H25	121.6 (16)
C12—C7—H7	105.5 (11)	C24—C25—H25	116.5 (16)
C6—C7—H7	106.6 (11)	C27—C26—C25	120.0 (2)
C7—C8—C9	108.8 (2)	C27—C26—H26	118.6 (15)
C7—C8—H8A	110.0 (16)	C25—C26—H26	121.3 (15)
C9—C8—H8A	109.6 (17)	C26—C27—C28	120.7 (2)
C7—C8—H8B	108.9 (14)	C26—C27—H27	122 (2)
C9—C8—H8B	108.6 (14)	C28—C27—H27	118 (2)
H8A—C8—H8B	111 (2)	C27—C28—C29	119.3 (2)
C8—C9—C10	112.7 (2)	C27—C28—H28	122.5 (14)
C8—C9—H9A	107.0 (18)	C29—C28—H28	118.2 (14)
C10—C9—H9A	111.1 (18)	C28—C29—C24	121.6 (2)
C8—C9—H9B	108.0 (17)	C28—C29—Cl1	117.56 (17)
C10—C9—H9B	107.1 (17)	C24—C29—Cl1	120.87 (15)
H9A—C9—H9B	111 (2)	C31—C30—C35	116.47 (17)
C9—C10—C11	113.0 (2)	C31—C30—C4	121.03 (16)
C9—C10—H10A	109.3 (16)	C35—C30—C4	122.47 (17)
C11—C10—H10A	108.5 (15)	C32—C31—C30	122.0 (2)
C9—C10—H10B	112.7 (17)	C32—C31—H31	121.6 (17)
C11—C10—H10B	111.4 (16)	C30—C31—H31	116.2 (16)
H10A—C10—H10B	101 (2)	C33—C32—C31	119.7 (3)
C12—C11—C10	109.3 (2)	C33—C32—H32	123.2 (19)
C12—C11—H11A	107.4 (15)	C31—C32—H32	117.0 (19)
C10—C11—H11A	111.0 (15)	C34—C33—C32	120.0 (2)
C12—C11—H11B	107.6 (16)	C34—C33—H33	122 (2)
C10—C11—H11B	109.4 (16)	C32—C33—H33	118 (2)
H11A—C11—H11B	112 (2)	C33—C34—C35	119.9 (2)
N1—C12—C11	116.32 (16)	C33—C34—H34	122 (2)
N1—C12—C7	103.32 (13)	C35—C34—H34	118 (2)
C11—C12—C7	111.52 (16)	C30—C35—C34	121.8 (2)
N1—C12—H12	109.7 (11)	C30—C35—Cl2	120.44 (15)
C11—C12—H12	108.4 (11)	C34—C35—Cl2	117.75 (18)
C7—C12—H12	107.1 (11)	C30—C35—Cl2	120.44 (15)
C14—C13—C18	119.22 (15)	C34—C35—Cl2	117.75 (18)

C12—N1—C2—C13	−27.37 (19)	C16—C17—C18—N19	−179.6 (2)
C5—N1—C2—C13	96.41 (15)	C14—C13—C18—C17	−0.9 (3)
C12—N1—C2—C20	89.38 (16)	C2—C13—C18—C17	−179.32 (18)
C5—N1—C2—C20	−146.84 (13)	C14—C13—C18—N19	178.74 (16)
C12—N1—C2—C3	−154.43 (13)	C2—C13—C18—N19	0.34 (19)
C5—N1—C2—C3	−30.65 (15)	C17—C18—N19—C20	178.2 (2)
N1—C2—C3—C21	169.87 (13)	C13—C18—N19—C20	−1.4 (2)
C13—C2—C3—C21	40.89 (19)	C18—N19—C20—O1	−178.11 (17)
C20—C2—C3—C21	−74.35 (17)	C18—N19—C20—C2	1.9 (2)
N1—C2—C3—C4	45.62 (14)	N1—C2—C20—O1	51.7 (2)
C13—C2—C3—C4	−83.36 (15)	C13—C2—C20—O1	178.44 (17)
C20—C2—C3—C4	161.40 (12)	C3—C2—C20—O1	−58.1 (2)
C21—C3—C4—C30	68.36 (18)	N1—C2—C20—N19	−128.25 (14)
C2—C3—C4—C30	−166.65 (13)	C13—C2—C20—N19	−1.55 (17)
C21—C3—C4—C5	−168.14 (14)	C3—C2—C20—N19	121.95 (15)
C2—C3—C4—C5	−43.14 (14)	C4—C3—C21—O2	37.5 (2)
C2—N1—C5—C4	4.06 (16)	C2—C3—C21—O2	−79.8 (2)
C12—N1—C5—C4	133.74 (13)	C4—C3—C21—C22	−140.45 (16)
C2—N1—C5—C6	−124.30 (14)	C2—C3—C21—C22	102.23 (18)
C12—N1—C5—C6	5.38 (17)	O2—C21—C22—C23	−170.3 (2)
C30—C4—C5—N1	148.21 (14)	C3—C21—C22—C23	7.7 (3)
C3—C4—C5—N1	24.64 (15)	C21—C22—C23—C24	172.42 (18)
C30—C4—C5—C6	−93.44 (18)	C22—C23—C24—C29	170.9 (2)
C3—C4—C5—C6	142.99 (15)	C22—C23—C24—C25	−10.8 (3)
N1—C5—C6—C7	−31.18 (17)	C29—C24—C25—C26	−1.1 (3)
C4—C5—C6—C7	−149.73 (15)	C23—C24—C25—C26	−179.5 (2)
C5—C6—C7—C8	167.58 (17)	C24—C25—C26—C27	0.4 (4)
C5—C6—C7—C12	45.07 (17)	C25—C26—C27—C28	0.1 (4)
C12—C7—C8—C9	−58.8 (2)	C26—C27—C28—C29	0.1 (4)
C6—C7—C8—C9	−177.02 (19)	C27—C28—C29—C24	−0.8 (3)
C7—C8—C9—C10	54.5 (3)	C27—C28—C29—Cl1	−179.73 (19)
C8—C9—C10—C11	−52.3 (4)	C25—C24—C29—C28	1.2 (3)
C9—C10—C11—C12	51.9 (3)	C23—C24—C29—C28	179.63 (19)
C2—N1—C12—C11	−90.1 (2)	C25—C24—C29—Cl1	−179.81 (17)
C5—N1—C12—C11	145.60 (17)	C23—C24—C29—Cl1	−1.4 (3)
C2—N1—C12—C7	147.41 (14)	C5—C4—C30—C31	−60.8 (2)
C5—N1—C12—C7	23.08 (17)	C3—C4—C30—C31	55.4 (2)
C10—C11—C12—N1	−174.89 (18)	C5—C4—C30—C35	117.3 (2)
C10—C11—C12—C7	−56.8 (3)	C3—C4—C30—C35	−126.51 (19)
C8—C7—C12—N1	−172.44 (16)	C35—C30—C31—C32	−0.3 (4)
C6—C7—C12—N1	−42.64 (18)	C4—C30—C31—C32	177.9 (2)
C8—C7—C12—C11	61.9 (2)	C30—C31—C32—C33	−0.7 (5)
C6—C7—C12—C11	−168.31 (18)	C31—C32—C33—C34	0.7 (5)
N1—C2—C13—C14	−56.7 (3)	C32—C33—C34—C35	0.3 (5)
C20—C2—C13—C14	−177.40 (19)	C31—C30—C35—C34	1.3 (3)
C3—C2—C13—C14	62.8 (3)	C4—C30—C35—C34	−176.9 (2)
N1—C2—C13—C18	121.44 (15)	C31—C30—C35—Cl2	−178.35 (18)
C20—C2—C13—C18	0.70 (17)	C4—C30—C35—Cl2	3.4 (3)
C3—C2—C13—C18	−119.10 (15)	C31—C30—C35—Cl2	−178.35 (18)

C18—C13—C14—C15	0.8 (3)	C4—C30—C35—Cl2	3.4 (3)
C2—C13—C14—C15	178.76 (19)	C33—C34—C35—C30	−1.3 (4)
C13—C14—C15—C16	0.2 (4)	C33—C34—C35—Cl2	178.4 (3)
C14—C15—C16—C17	−1.1 (4)	C33—C34—C35—Cl2	178.4 (3)
C15—C16—C17—C18	1.0 (4)	Cl2—Cl2—C35—C30	0.00 (4)
C16—C17—C18—C13	0.0 (3)	Cl2—Cl2—C35—C34	0.00 (2)

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C4—H4⋯Cl2	0.968 (18)	2.515 (18)	3.0895 (16)	118.0 (13)
N19—H19⋯O1[i]	0.86 (2)	2.54 (2)	3.222 (2)	136.0 (18)
C17—H17⋯O1[i]	0.92 (3)	2.50 (3)	3.253 (3)	140 (2)
C22—H22⋯O2[ii]	0.97 (2)	2.57 (2)	3.522 (2)	166.3 (17)
C25—H25⋯O2[ii]	0.95 (3)	2.43 (3)	3.366 (3)	171 (2)

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, −*y*+3/2, −*z*.

Fig. 1