IUCrData

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6b-Hydroxy-17-methyl-15-(3-nitrophenyl)-6b,7,16,17-tetrahydro-7,14a-methanonaphtho-[1′,8′:1,2,3]pyrrolo[3′,2′:8,8a]azuleno[5,6-*b*]-quinolin-14(15*H*)-one dichloromethane hemisolvate

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Structural data: full structural data are available from iucrdata.iucr.org

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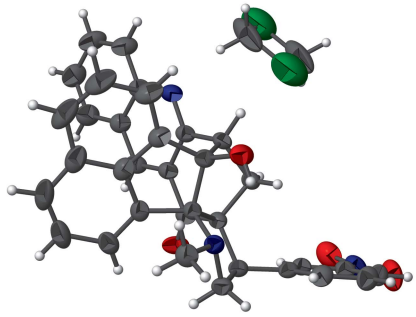
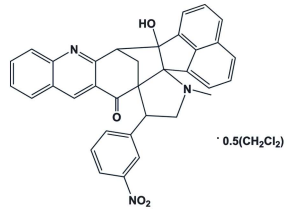
In the title compound, $C_{34}H_{25}N_3O_4\cdot0.5CH_2Cl_2$, which crystallized as a dichloromethane hemisolvate, the central 1-methylpyrrolidine ring adopts an envelope conformation with the N atom as the flap. The cyclopentane ring adopts a twist conformation on the CH—CH_2 bond and the cyclohexane ring has an envelope conformation with the CH_2 atom as the flap. The pyrrolidine ring mean plane makes dihedral angles of 40.09 (11), 69.21 (10) and 80.88 (8)° with the mean planes of the cyclopentane, cyclohexane and acenaphthylene rings, respectively. The 3-nitrobenzene ring is inclined to the acenaphthylene and quinoline ring systems by 69.32 (8) and 82.07 (7)°, respectively. There is an intramolecular O—H···N hydrogen bond present forming an $S(5)$ ring motif. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming sheets lying parallel to the *ab* plane, which enclose $R_2^2(18)$, $R_2^2(14)$, $R_2^2(12)$ and $R_2^2(16)$ ring motifs.

3D view



Chemical scheme



Structure description

Highly functionalized pyrrolidines have gained much interest in the past few years as they constitute the main structural element of many natural and synthetic pharmacologically active compounds (Waldmann, 1995). Pyrrolidine derivatives are widely used as organic catalysts and serve as important structural units in biologically active molecules (Pinna *et al.*, 2002). Optically active pyrrolidines have been used as intermediates, chiral ligands or auxiliaries in controlled asymmetric synthesis (Savithri *et al.*, 2014). The spiro-pyrrolidine

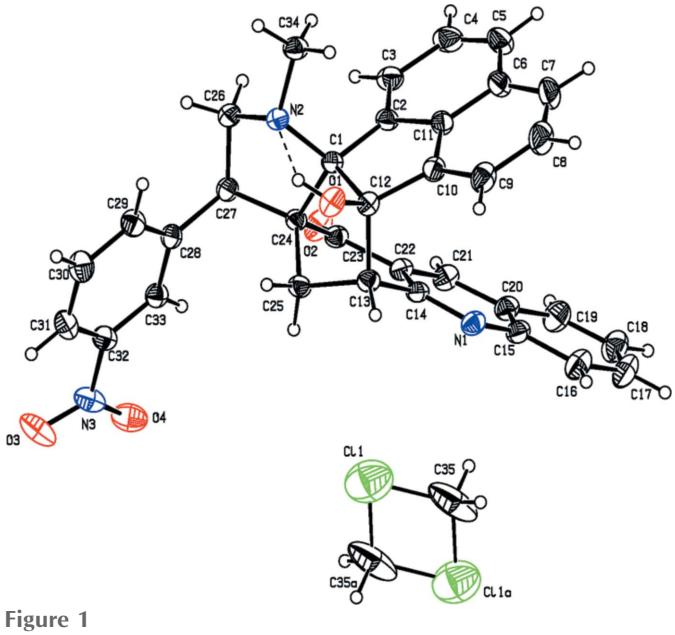


Figure 1
The molecular structure of the title compound, showing the atomic numbering and displacement ellipsoids drawn at 30% probability level. The intramolecular O—H···N hydrogen bond is shown as a dashed line.

ring system is associated with antitumor activity (Araki *et al.*, 2002). Pyrrolidine compounds are reported to exhibit antimicrobial, antifungal, anti-influenza virus A, anti-inflammatory (Mathusalini *et al.*, 2015), antitumor, antidepressant, antibiotic and anticonvulsant (Joseph *et al.*, 2015) activity and to act as sphingosine-1-phosphate (S1P) receptor agonists, malic enzyme inhibitors, ketoamide-based cathepsin K inhibitors and human melanocortin-4 receptor agonists (Babu *et al.*, 2012).

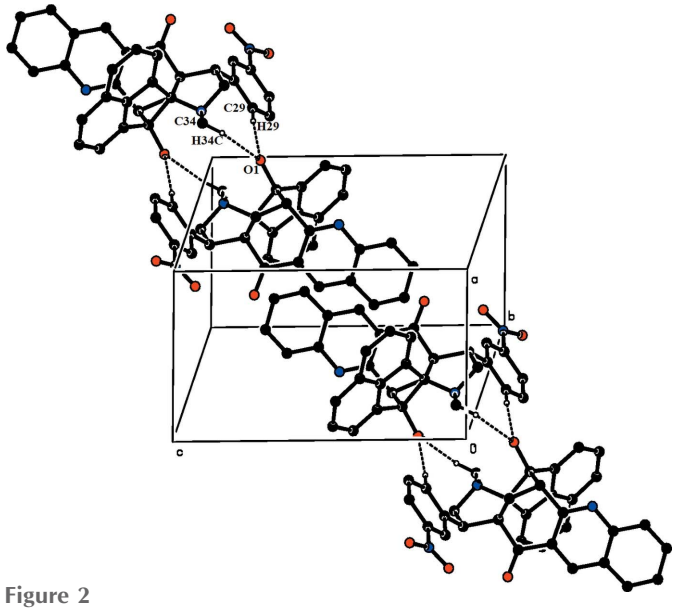


Figure 2
A partial crystal packing diagram of the title compound, viewed approximately along the *b* axis, showing the $R_2^2(12)$ and $R_2^2(16)$ ring motifs formed by C29—H29···O1 and C34—H34*C*···O1 hydrogen bonds (dashed lines; see Table 1). H atoms not involved in the hydrogen bonds have been excluded for clarity.

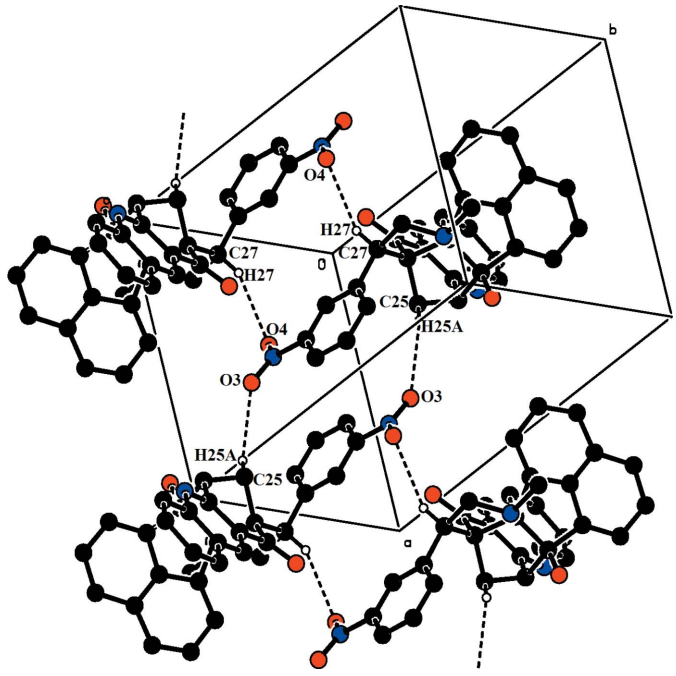


Figure 3
A partial crystal packing diagram of the title compound, illustrating the formation of the hydrogen bonded chains (dashed lines; see Table 1) running along [100], and enclosing $R_2^2(18)$ and $R_2^2(14)$ ring motifs. H atoms not involved in the hydrogen bonds have been omitted for clarity.

The molecular structure of the title compound is shown in Fig. 1. There is an intramolecular O—H···N hydrogen bond present forming an *S*(5) ring motif (Table 1 and Fig. 1). The central 1-methyl pyrrolidine ring (N2/C1/C24/C27/C26) adopts an envelope conformation with atom N2 as the flap; it is displaced by 0.561 (1) Å from the mean plane through the other four atoms. The cyclohexane (C13/C14/C22–C25) ring adopts an envelope conformation with atom C25 as the flap, displaced by 0.863 (1) Å from the mean plane through the

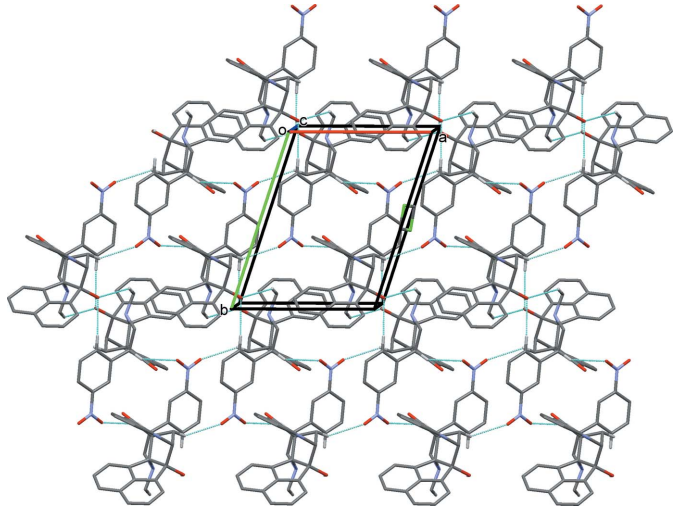


Figure 4
A view along the *c* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1), and H atoms not involved in the hydrogen bonds have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2	0.82	2.12	2.669 (2)	124
C25—H25A···O3^{i}	0.97	2.57	3.289 (3)	131
C27—H27···O4^{ii}	0.98	2.54	3.384 (3)	145
C29—H29···O1^{iii}	0.93	2.58	3.502 (2)	174
C34—H34C···O1^{iii}	0.96	2.56	3.274 (2)	131

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $-x+1, -y+1, -z+2$; (iii) $-x+2, -y+2, -z+2$.

other five atoms. The cyclopentane ring (C1/C12/C13/C25/C24) adopts a twist conformation on the C13—C25 bond. The quinoline ring system (C14/N2/C15–C22) and the acenaphthylene ring system (C1–C12) adopt almost planar conformations, with the maximum deviation being 0.041 (1) Å for atom C14 in the quinoline ring system and 0.143 (1) Å for atom C1 in the acenaphthylene ring system. The mean plane of the pyrrolidine ring makes dihedral angles of 40.09 (11), 69.21 (10), 80.88 (8) and 62.38 (10)° with the mean planes of the cyclopentane, cyclohexane, acenaphthylene and the 3-nitrobenzene rings, respectively. The methyl group atom C34 is displaced by 0.238 (1) Å from the pyrrolidine ring to which it is attached and atom O2 is displaced from the cyclohexane ring mean plane by 0.332 (1) Å. The nitro group lies almost in the plane of the attached benzene ring (C28–C33), making a dihedral angle of 11.8 (3)°.

In the crystal, hydrogen bonds C29—H29···O1 and C34—H34C···O1 involving the same acceptor atom link molecules into inversion dimers enclosing $R_2^2(12)$ and $R_2^2(16)$ ring motifs (Table 1 and Fig. 2). The dimers are linked by C25—H25A···O3 and C27—H27···O4 hydrogen bonds, enclosing $R_2^2(18)$ and $R_2^2(14)$ ring motifs (Table 1 and Fig. 3), forming sheets lying parallel to the *ab* plane (Table 1 and Fig. 4).

Synthesis and crystallization

A mixture of (*E*)-2-(3-nitrobenzylidene)-3,4-dihydroacridin-1(2H)-one (1 mmol), acenaphthoquinone (1 mmol) and sarcosine (1.5 mmol) was heated to reflux in toluene (3 ml) for 10 h. After completion of the reaction as evident from TLC, the reaction mixture was extracted with ethyl acetate (2 × 20 ml), washed with water (2 × 10 ml), dried over anhydrous Na_2SO_4. It was then concentrated under reduced pressure, and subjected to column chromatography using petroleum ether–AcOEt (5:1 *v*/*v*) as eluent to obtain the title compound as a pure product. Slow evaporation of a solution of the title compound in dichloromethane at room temperature gave colourless block-like crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{34}H_{25}N_3O_4{\cdot}0.5CH_2Cl_2$
M_r	582.03
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	9.3566 (3), 12.1189 (4), 12.7055 (4)
α, β, γ (°)	94.490 (2), 93.300 (2), 107.554 (1)
V (Å^3)	1364.39 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.19
Crystal size (mm)	0.24 × 0.18 × 0.11
Data collection	
Diffractometer	Bruker *SMART* APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.746, 0.845
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20602, 5597, 4781
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.151, 1.03
No. of reflections	5597
No. of parameters	391
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.71, −0.81

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

Acknowledgements

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection.

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full crystallographic data

IUCrData (2016). **1**, x160828 [doi:10.1107/S2414314616008282]

6b-Hydroxy-17-methyl-15-(3-nitrophenyl)-6b,7,16,17-tetrahydro-7,14a-methanonaphtho[1′,8′:1,2,3]pyrrolo[3′,2′:8,8a]azuleno[5,6-*b*]quinolin-14(15*H*)-one dichloromethane hemisolvate

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6b-Hydroxy-17-methyl-15-(3-nitrophenyl)-6b,7,16,17-tetrahydro-7,14a-methanonaphtho[1′,8′:1,2,3]pyrrolo[3′,2′:8,8a]azuleno[5,6-*b*]quinolin-14(15*H*)-one dichloromethane hemisolvate

Crystal data

$C_{34}H_{25}N_3O_4·0.5CH_2Cl_2$
M_r = 582.03
Triclinic, $P\overline{1}$
a = 9.3566 (3) Å
b = 12.1189 (4) Å
c = 12.7055 (4) Å
α = 94.490 (2)°
β = 93.300 (2)°
γ = 107.554 (1)°
V = 1364.39 (8) Å^3
Z = 2
$F(000)$ = 606
D_x = 1.417 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5597 reflections
θ = 1.6–26.5°
μ = 0.19 mm^{-1}
T = 293 K
Block, colourless
0.24 × 0.18 × 0.11 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.746, T_{max} = 0.845
20602 measured reflections
5597 independent reflections
4781 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.5°, θ_{min} = 1.6°
h = −11→11
k = −15→15
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.151
S = 1.03
5597 reflections
391 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
$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.7773P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.81 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (2)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C35	0.9773 (8)	0.5614 (7)	0.4596 (8)	0.122 (3)	0.5
H35A	0.8820	0.5779	0.4543	0.146*	0.5
H35B	1.0486	0.6182	0.4233	0.146*	0.5
Cl1	1.04569 (14)	0.57364 (13)	0.59887 (11)	0.1339 (5)	
C1	0.72811 (18)	0.87889 (14)	0.84196 (13)	0.0306 (4)	
C2	0.6091 (2)	0.91913 (14)	0.78307 (14)	0.0343 (4)	
C3	0.4613 (2)	0.90609 (18)	0.79707 (16)	0.0445 (4)	
H3	0.4119	0.8591	0.8466	0.053*	
C4	0.3853 (3)	0.9663 (2)	0.73399 (19)	0.0588 (6)	
H4	0.2847	0.9578	0.7430	0.071*	
C5	0.4541 (3)	1.0362 (2)	0.66084 (19)	0.0638 (7)	
H5	0.4007	1.0753	0.6223	0.077*	
C6	0.6059 (3)	1.04980 (18)	0.64289 (16)	0.0516 (5)	
C7	0.6941 (4)	1.1164 (2)	0.56918 (18)	0.0675 (7)	
H7	0.6521	1.1594	0.5263	0.081*	
C8	0.8393 (4)	1.1184 (2)	0.56030 (19)	0.0658 (7)	
H8	0.8944	1.1634	0.5116	0.079*	
C9	0.9087 (3)	1.05452 (18)	0.62237 (16)	0.0514 (5)	
H9	1.0073	1.0558	0.6141	0.062*	
C10	0.8275 (2)	0.99051 (15)	0.69512 (14)	0.0379 (4)	
C11	0.6784 (2)	0.98855 (15)	0.70502 (14)	0.0386 (4)	
C12	0.86515 (19)	0.91164 (15)	0.77043 (13)	0.0330 (4)	
C13	0.86429 (19)	0.79050 (15)	0.71955 (14)	0.0339 (4)	
H13	0.9619	0.7940	0.6933	0.041*	
C14	0.73823 (19)	0.73871 (14)	0.63388 (13)	0.0329 (4)	
C15	0.6529 (2)	0.69894 (16)	0.45693 (14)	0.0393 (4)	
C16	0.6839 (3)	0.7051 (2)	0.34972 (16)	0.0538 (5)	
H16	0.7825	0.7363	0.3329	0.065*	
C17	0.5707 (3)	0.6658 (2)	0.27066 (17)	0.0618 (6)	
H17	0.5930	0.6713	0.2005	0.074*	
C18	0.4214 (3)	0.6173 (2)	0.29334 (17)	0.0591 (6)	
H18	0.3451	0.5922	0.2386	0.071*	
C19	0.3883 (3)	0.6071 (2)	0.39542 (17)	0.0544 (5)	
H19	0.2892	0.5731	0.4100	0.065*	
C20	0.5024 (2)	0.64736 (16)	0.47987 (15)	0.0408 (4)	
C21	0.4750 (2)	0.63939 (17)	0.58695 (15)	0.0416 (4)	
H21	0.3783	0.6038	0.6054	0.050*	
C22	0.5905 (2)	0.68392 (15)	0.66374 (14)	0.0345 (4)	
C23	0.5629 (2)	0.67639 (14)	0.77782 (14)	0.0345 (4)	

C24	0.69506 (19)	0.74606 (14)	0.85484 (13)	0.0312 (4)
C25	0.8359 (2)	0.71954 (16)	0.81484 (14)	0.0361 (4)
H25A	0.9205	0.7455	0.8683	0.043*
H25B	0.8154	0.6372	0.7939	0.043*
C26	0.6759 (2)	0.85428 (16)	1.01904 (14)	0.0395 (4)
H26A	0.5768	0.8643	1.0171	0.047*
H26B	0.7194	0.8689	1.0919	0.047*
C27	0.6661 (2)	0.73169 (15)	0.97300 (13)	0.0351 (4)
H27	0.5620	0.6824	0.9750	0.042*
C28	0.7674 (2)	0.67420 (15)	1.02952 (13)	0.0342 (4)
C29	0.8895 (2)	0.73465 (17)	1.10098 (15)	0.0432 (4)
H29	0.9135	0.8150	1.1141	0.052*
C30	0.9760 (2)	0.67755 (19)	1.15298 (17)	0.0501 (5)
H30	1.0570	0.7199	1.2004	0.060*
C31	0.9431 (2)	0.55854 (18)	1.13504 (17)	0.0466 (5)
H31	1.0002	0.5195	1.1701	0.056*
C32	0.8232 (2)	0.49940 (16)	1.06359 (15)	0.0393 (4)
C33	0.7352 (2)	0.55443 (16)	1.01075 (14)	0.0362 (4)
H33	0.6550	0.5115	0.9630	0.043*
C34	0.7644 (2)	1.05220 (16)	0.97148 (15)	0.0410 (4)
H34A	0.6616	1.0512	0.9610	0.061*
H34B	0.8232	1.1004	0.9231	0.061*
H34C	0.8031	1.0828	1.0430	0.061*
N1	0.76883 (18)	0.74405 (13)	0.53420 (12)	0.0388 (4)
N2	0.77263 (16)	0.93343 (12)	0.95215 (11)	0.0339 (3)
N3	0.7856 (2)	0.37245 (15)	1.04308 (16)	0.0505 (4)
O1	1.00338 (14)	0.96413 (12)	0.83179 (10)	0.0437 (3)
H1	0.9886	0.9675	0.8948	0.066*
O2	0.44393 (16)	0.61858 (13)	0.80500 (11)	0.0509 (4)
O3	0.8470 (2)	0.32270 (15)	1.10228 (18)	0.0773 (6)
O4	0.6955 (2)	0.32303 (14)	0.96856 (16)	0.0652 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C35	0.079 (4)	0.094 (5)	0.231 (10)	0.058 (4)	0.071 (6)	0.085 (6)
Cl1	0.1021 (8)	0.1563 (11)	0.1467 (11)	0.0483 (7)	0.0245 (7)	−0.0078 (8)
C1	0.0313 (8)	0.0289 (8)	0.0325 (8)	0.0101 (6)	0.0049 (6)	0.0028 (6)
C2	0.0379 (9)	0.0295 (8)	0.0364 (9)	0.0132 (7)	−0.0003 (7)	−0.0008 (7)
C3	0.0410 (10)	0.0481 (11)	0.0466 (10)	0.0195 (9)	0.0019 (8)	−0.0041 (8)
C4	0.0514 (13)	0.0725 (15)	0.0597 (13)	0.0374 (12)	−0.0102 (10)	−0.0127 (12)
C5	0.0824 (17)	0.0676 (15)	0.0548 (13)	0.0494 (14)	−0.0160 (12)	−0.0021 (11)
C6	0.0748 (15)	0.0421 (11)	0.0428 (11)	0.0284 (10)	−0.0076 (10)	0.0006 (8)
C7	0.117 (2)	0.0481 (13)	0.0448 (12)	0.0355 (14)	−0.0031 (13)	0.0144 (10)
C8	0.103 (2)	0.0458 (12)	0.0481 (12)	0.0167 (13)	0.0157 (13)	0.0187 (10)
C9	0.0654 (14)	0.0391 (10)	0.0430 (10)	0.0039 (9)	0.0114 (9)	0.0077 (8)
C10	0.0469 (10)	0.0282 (8)	0.0350 (9)	0.0066 (7)	0.0036 (7)	0.0023 (7)
C11	0.0509 (11)	0.0301 (8)	0.0344 (9)	0.0136 (8)	−0.0026 (8)	0.0004 (7)

C12	0.0301 (8)	0.0334 (9)	0.0333 (8)	0.0061 (7)	0.0050 (6)	0.0030 (7)
C13	0.0293 (8)	0.0372 (9)	0.0377 (9)	0.0123 (7)	0.0103 (7)	0.0053 (7)
C14	0.0366 (9)	0.0279 (8)	0.0359 (9)	0.0117 (7)	0.0098 (7)	0.0023 (6)
C15	0.0474 (10)	0.0335 (9)	0.0372 (9)	0.0125 (8)	0.0090 (8)	0.0006 (7)
C16	0.0609 (13)	0.0550 (12)	0.0408 (11)	0.0097 (10)	0.0136 (9)	0.0015 (9)
C17	0.0798 (17)	0.0641 (14)	0.0339 (10)	0.0114 (12)	0.0096 (10)	−0.0011 (9)
C18	0.0681 (15)	0.0605 (14)	0.0413 (11)	0.0133 (11)	−0.0053 (10)	−0.0062 (10)
C19	0.0505 (12)	0.0597 (13)	0.0463 (11)	0.0096 (10)	0.0011 (9)	−0.0034 (10)
C20	0.0452 (10)	0.0367 (9)	0.0393 (9)	0.0118 (8)	0.0046 (8)	−0.0012 (7)
C21	0.0374 (10)	0.0408 (10)	0.0433 (10)	0.0068 (8)	0.0090 (8)	0.0014 (8)
C22	0.0366 (9)	0.0310 (8)	0.0362 (9)	0.0101 (7)	0.0087 (7)	0.0017 (7)
C23	0.0367 (9)	0.0289 (8)	0.0385 (9)	0.0095 (7)	0.0104 (7)	0.0037 (7)
C24	0.0337 (9)	0.0295 (8)	0.0334 (8)	0.0123 (7)	0.0087 (7)	0.0066 (6)
C25	0.0366 (9)	0.0381 (9)	0.0392 (9)	0.0178 (7)	0.0096 (7)	0.0071 (7)
C26	0.0464 (10)	0.0428 (10)	0.0353 (9)	0.0198 (8)	0.0130 (8)	0.0069 (7)
C27	0.0359 (9)	0.0370 (9)	0.0344 (9)	0.0121 (7)	0.0095 (7)	0.0075 (7)
C28	0.0362 (9)	0.0352 (9)	0.0326 (8)	0.0099 (7)	0.0110 (7)	0.0097 (7)
C29	0.0455 (11)	0.0356 (9)	0.0452 (10)	0.0072 (8)	0.0045 (8)	0.0060 (8)
C30	0.0440 (11)	0.0522 (12)	0.0493 (11)	0.0088 (9)	−0.0045 (9)	0.0062 (9)
C31	0.0426 (10)	0.0509 (11)	0.0509 (11)	0.0180 (9)	0.0057 (8)	0.0176 (9)
C32	0.0393 (10)	0.0355 (9)	0.0460 (10)	0.0123 (8)	0.0148 (8)	0.0113 (7)
C33	0.0354 (9)	0.0368 (9)	0.0366 (9)	0.0099 (7)	0.0081 (7)	0.0057 (7)
C34	0.0466 (10)	0.0339 (9)	0.0429 (10)	0.0148 (8)	0.0029 (8)	−0.0023 (7)
N1	0.0408 (8)	0.0384 (8)	0.0373 (8)	0.0111 (7)	0.0114 (6)	0.0027 (6)
N2	0.0375 (8)	0.0333 (7)	0.0324 (7)	0.0132 (6)	0.0053 (6)	0.0017 (6)
N3	0.0443 (10)	0.0383 (9)	0.0751 (12)	0.0181 (8)	0.0178 (9)	0.0116 (9)
O1	0.0322 (7)	0.0524 (8)	0.0392 (7)	0.0035 (6)	0.0020 (5)	−0.0007 (6)
O2	0.0443 (8)	0.0517 (8)	0.0447 (8)	−0.0044 (6)	0.0149 (6)	0.0014 (6)
O3	0.0733 (12)	0.0513 (10)	0.1195 (16)	0.0335 (9)	0.0046 (11)	0.0263 (10)
O4	0.0596 (10)	0.0418 (8)	0.0909 (13)	0.0135 (7)	0.0067 (9)	−0.0051 (8)

Geometric parameters (Å, º)

C35—Cl1^{i}	1.690 (9)	C17—H17	0.9300
C35—Cl1	1.825 (11)	C18—C19	1.357 (3)
C35—H35A	0.9700	C18—H18	0.9300
C35—H35B	0.9700	C19—C20	1.415 (3)
Cl1—C35^{i}	1.690 (9)	C19—H19	0.9300
C1—N2	1.481 (2)	C20—C21	1.404 (3)
C1—C2	1.525 (2)	C21—C22	1.362 (3)
C1—C24	1.569 (2)	C21—H21	0.9300
C1—C12	1.585 (2)	C22—C23	1.492 (2)
C2—C3	1.367 (3)	C23—O2	1.209 (2)
C2—C11	1.410 (3)	C23—C24	1.517 (2)
C3—C4	1.422 (3)	C24—C25	1.551 (2)
C3—H3	0.9300	C24—C27	1.555 (2)
C4—C5	1.363 (4)	C25—H25A	0.9700
C4—H4	0.9300	C25—H25B	0.9700

C5—C6	1.413 (4)	C26—N2	1.468 (2)
C5—H5	0.9300	C26—C27	1.527 (2)
C6—C11	1.403 (3)	C26—H26A	0.9700
C6—C7	1.420 (4)	C26—H26B	0.9700
C7—C8	1.363 (4)	C27—C28	1.516 (2)
C7—H7	0.9300	C27—H27	0.9800
C8—C9	1.408 (3)	C28—C33	1.389 (2)
C8—H8	0.9300	C28—C29	1.392 (3)
C9—C10	1.368 (3)	C29—C30	1.385 (3)
C9—H9	0.9300	C29—H29	0.9300
C10—C11	1.402 (3)	C30—C31	1.378 (3)
C10—C12	1.503 (2)	C30—H30	0.9300
C12—O1	1.413 (2)	C31—C32	1.376 (3)
C12—C13	1.555 (2)	C31—H31	0.9300
C13—C14	1.506 (2)	C32—C33	1.382 (3)
C13—C25	1.529 (2)	C32—N3	1.469 (2)
C13—H13	0.9800	C33—H33	0.9300
C14—N1	1.317 (2)	C34—N2	1.467 (2)
C14—C22	1.429 (2)	C34—H34A	0.9600
C15—N1	1.369 (2)	C34—H34B	0.9600
C15—C16	1.412 (3)	C34—H34C	0.9600
C15—C20	1.416 (3)	N3—O4	1.217 (3)
C16—C17	1.362 (3)	N3—O3	1.221 (2)
C16—H16	0.9300	O1—H1	0.8200
C17—C18	1.398 (4)		

Cl1^{i}—C35—Cl1	110.6 (4)	C18—C19—H19	119.6
Cl1^{i}—C35—H35A	109.5	C20—C19—H19	119.6
Cl1—C35—H35A	109.5	C21—C20—C19	123.51 (19)
Cl1^{i}—C35—H35B	109.5	C21—C20—C15	117.29 (17)
Cl1—C35—H35B	109.5	C19—C20—C15	119.20 (18)
H35A—C35—H35B	108.1	C22—C21—C20	119.85 (17)
C35^{i}—Cl1—C35	69.4 (4)	C22—C21—H21	120.1
N2—C1—C2	114.47 (13)	C20—C21—H21	120.1
N2—C1—C24	102.71 (13)	C21—C22—C14	119.27 (16)
C2—C1—C24	119.05 (14)	C21—C22—C23	120.40 (16)
N2—C1—C12	111.49 (13)	C14—C22—C23	120.33 (16)
C2—C1—C12	103.26 (13)	O2—C23—C22	121.66 (17)
C24—C1—C12	105.70 (12)	O2—C23—C24	123.66 (16)
C3—C2—C11	118.97 (17)	C22—C23—C24	114.67 (14)
C3—C2—C1	132.75 (17)	C23—C24—C25	106.43 (13)
C11—C2—C1	108.13 (15)	C23—C24—C27	113.57 (14)
C2—C3—C4	118.2 (2)	C25—C24—C27	117.49 (14)
C2—C3—H3	120.9	C23—C24—C1	109.24 (13)
C4—C3—H3	120.9	C25—C24—C1	103.19 (13)
C5—C4—C3	122.5 (2)	C27—C24—C1	106.26 (13)
C5—C4—H4	118.7	C13—C25—C24	101.59 (13)
C3—C4—H4	118.7	C13—C25—H25A	111.5

C4—C5—C6	120.7 (2)	C24—C25—H25A	111.5
C4—C5—H5	119.7	C13—C25—H25B	111.5
C6—C5—H5	119.7	C24—C25—H25B	111.5
C11—C6—C5	115.9 (2)	H25A—C25—H25B	109.3
C11—C6—C7	115.7 (2)	N2—C26—C27	105.75 (13)
C5—C6—C7	128.4 (2)	N2—C26—H26A	110.6
C8—C7—C6	121.0 (2)	C27—C26—H26A	110.6
C8—C7—H7	119.5	N2—C26—H26B	110.6
C6—C7—H7	119.5	C27—C26—H26B	110.6
C7—C8—C9	122.2 (2)	H26A—C26—H26B	108.7
C7—C8—H8	118.9	C28—C27—C26	116.14 (15)
C9—C8—H8	118.9	C28—C27—C24	113.91 (14)
C10—C9—C8	118.4 (2)	C26—C27—C24	103.52 (13)
C10—C9—H9	120.8	C28—C27—H27	107.6
C8—C9—H9	120.8	C26—C27—H27	107.6
C9—C10—C11	119.77 (19)	C24—C27—H27	107.6
C9—C10—C12	131.86 (19)	C33—C28—C29	118.07 (17)
C11—C10—C12	108.32 (15)	C33—C28—C27	118.35 (16)
C10—C11—C6	122.96 (19)	C29—C28—C27	123.57 (16)
C10—C11—C2	113.41 (16)	C30—C29—C28	121.33 (18)
C6—C11—C2	123.63 (19)	C30—C29—H29	119.3
O1—C12—C10	112.78 (14)	C28—C29—H29	119.3
O1—C12—C13	108.69 (14)	C31—C30—C29	120.57 (19)
C10—C12—C13	115.62 (14)	C31—C30—H30	119.7
O1—C12—C1	111.83 (13)	C29—C30—H30	119.7
C10—C12—C1	105.09 (14)	C32—C31—C30	117.83 (18)
C13—C12—C1	102.33 (13)	C32—C31—H31	121.1
C14—C13—C25	109.51 (14)	C30—C31—H31	121.1
C14—C13—C12	112.85 (14)	C31—C32—C33	122.66 (18)
C25—C13—C12	100.88 (13)	C31—C32—N3	118.94 (17)
C14—C13—H13	111.1	C33—C32—N3	118.40 (18)
C25—C13—H13	111.1	C32—C33—C28	119.54 (17)
C12—C13—H13	111.1	C32—C33—H33	120.2
N1—C14—C22	122.45 (16)	C28—C33—H33	120.2
N1—C14—C13	118.71 (15)	N2—C34—H34A	109.5
C22—C14—C13	118.83 (15)	N2—C34—H34B	109.5
N1—C15—C16	118.87 (18)	H34A—C34—H34B	109.5
N1—C15—C20	122.78 (16)	N2—C34—H34C	109.5
C16—C15—C20	118.35 (18)	H34A—C34—H34C	109.5
C17—C16—C15	120.6 (2)	H34B—C34—H34C	109.5
C17—C16—H16	119.7	C14—N1—C15	118.27 (16)
C15—C16—H16	119.7	C34—N2—C26	111.60 (14)
C16—C17—C18	121.0 (2)	C34—N2—C1	115.28 (14)
C16—C17—H17	119.5	C26—N2—C1	105.85 (13)
C18—C17—H17	119.5	O4—N3—O3	123.81 (19)
C19—C18—C17	119.8 (2)	O4—N3—C32	118.46 (17)
C19—C18—H18	120.1	O3—N3—C32	117.7 (2)
C17—C18—H18	120.1	C12—O1—H1	109.5

C18—C19—C20	120.9 (2)

Cl1^{i}—C35—Cl1—C35^{i}	0.001 (1)	C20—C21—C22—C14	0.2 (3)
N2—C1—C2—C3	−66.1 (2)	C20—C21—C22—C23	179.79 (17)
C24—C1—C2—C3	55.9 (3)	N1—C14—C22—C21	−2.7 (3)
C12—C1—C2—C3	172.54 (19)	C13—C14—C22—C21	178.38 (16)
N2—C1—C2—C11	109.33 (16)	N1—C14—C22—C23	177.75 (16)
C24—C1—C2—C11	−128.72 (15)	C13—C14—C22—C23	−1.2 (2)
C12—C1—C2—C11	−12.05 (17)	C21—C22—C23—O2	9.6 (3)
C11—C2—C3—C4	−1.8 (3)	C14—C22—C23—O2	−170.85 (17)
C1—C2—C3—C4	173.16 (18)	C21—C22—C23—C24	−170.58 (16)
C2—C3—C4—C5	−0.1 (3)	C14—C22—C23—C24	9.0 (2)
C3—C4—C5—C6	1.4 (4)	O2—C23—C24—C25	133.54 (18)
C4—C5—C6—C11	−0.6 (3)	C22—C23—C24—C25	−46.27 (18)
C4—C5—C6—C7	178.6 (2)	O2—C23—C24—C27	2.7 (2)
C11—C6—C7—C8	0.6 (3)	C22—C23—C24—C27	−177.08 (14)
C5—C6—C7—C8	−178.6 (2)	O2—C23—C24—C1	−115.68 (19)
C6—C7—C8—C9	0.5 (4)	C22—C23—C24—C1	64.51 (18)
C7—C8—C9—C10	−1.3 (4)	N2—C1—C24—C23	143.44 (13)
C8—C9—C10—C11	0.9 (3)	C2—C1—C24—C23	15.78 (19)
C8—C9—C10—C12	178.13 (19)	C12—C1—C24—C23	−99.60 (15)
C9—C10—C11—C6	0.1 (3)	N2—C1—C24—C25	−103.65 (14)
C12—C10—C11—C6	−177.66 (17)	C2—C1—C24—C25	128.70 (15)
C9—C10—C11—C2	−179.95 (17)	C12—C1—C24—C25	13.31 (16)
C12—C10—C11—C2	2.3 (2)	N2—C1—C24—C27	20.56 (16)
C5—C6—C11—C10	178.40 (18)	C2—C1—C24—C27	−107.10 (16)
C7—C6—C11—C10	−0.9 (3)	C12—C1—C24—C27	137.52 (14)
C5—C6—C11—C2	−1.5 (3)	C14—C13—C25—C24	−67.50 (16)
C7—C6—C11—C2	179.21 (18)	C12—C13—C25—C24	51.69 (16)
C3—C2—C11—C10	−177.16 (16)	C23—C24—C25—C13	74.97 (16)
C1—C2—C11—C10	6.7 (2)	C27—C24—C25—C13	−156.47 (15)
C3—C2—C11—C6	2.7 (3)	C1—C24—C25—C13	−39.98 (16)
C1—C2—C11—C6	−173.40 (17)	N2—C26—C27—C28	99.87 (17)
C9—C10—C12—O1	50.7 (3)	N2—C26—C27—C24	−25.75 (18)
C11—C10—C12—O1	−131.82 (15)	C23—C24—C27—C28	115.65 (16)
C9—C10—C12—C13	−75.2 (3)	C25—C24—C27—C28	−9.4 (2)
C11—C10—C12—C13	102.24 (17)	C1—C24—C27—C28	−124.24 (15)
C9—C10—C12—C1	172.82 (19)	C23—C24—C27—C26	−117.31 (15)
C11—C10—C12—C1	−9.75 (18)	C25—C24—C27—C26	117.61 (16)
N2—C1—C12—O1	12.32 (19)	C1—C24—C27—C26	2.80 (17)
C2—C1—C12—O1	135.70 (14)	C26—C27—C28—C33	162.61 (15)
C24—C1—C12—O1	−98.54 (15)	C24—C27—C28—C33	−77.2 (2)
N2—C1—C12—C10	−110.37 (15)	C26—C27—C28—C29	−16.1 (2)
C2—C1—C12—C10	13.01 (16)	C24—C27—C28—C29	104.12 (19)
C24—C1—C12—C10	138.77 (14)	C33—C28—C29—C30	−0.6 (3)
N2—C1—C12—C13	128.48 (14)	C27—C28—C29—C30	178.04 (18)
C2—C1—C12—C13	−108.14 (14)	C28—C29—C30—C31	0.0 (3)
C24—C1—C12—C13	17.62 (16)	C29—C30—C31—C32	0.5 (3)

O1—C12—C13—C14	−167.20 (13)	C30—C31—C32—C33	−0.6 (3)
C10—C12—C13—C14	−39.2 (2)	C30—C31—C32—N3	−179.94 (18)
C1—C12—C13—C14	74.39 (16)	C31—C32—C33—C28	0.0 (3)
O1—C12—C13—C25	76.05 (15)	N3—C32—C33—C28	179.36 (15)
C10—C12—C13—C25	−155.96 (15)	C29—C28—C33—C32	0.6 (2)
C1—C12—C13—C25	−42.37 (15)	C27—C28—C33—C32	−178.12 (15)
C25—C13—C14—N1	−146.84 (15)	C22—C14—N1—C15	2.4 (2)
C12—C13—C14—N1	101.65 (17)	C13—C14—N1—C15	−178.63 (15)
C25—C13—C14—C22	32.1 (2)	C16—C15—N1—C14	179.40 (18)
C12—C13—C14—C22	−79.39 (19)	C20—C15—N1—C14	0.2 (3)
N1—C15—C16—C17	−177.1 (2)	C27—C26—N2—C34	166.85 (15)
C20—C15—C16—C17	2.2 (3)	C27—C26—N2—C1	40.71 (17)
C15—C16—C17—C18	−0.7 (4)	C2—C1—N2—C34	−30.8 (2)
C16—C17—C18—C19	−1.2 (4)	C24—C1—N2—C34	−161.30 (14)
C17—C18—C19—C20	1.5 (4)	C12—C1—N2—C34	85.94 (17)
C18—C19—C20—C21	179.9 (2)	C2—C1—N2—C26	93.07 (16)
C18—C19—C20—C15	0.0 (3)	C24—C1—N2—C26	−37.43 (16)
N1—C15—C20—C21	−2.4 (3)	C12—C1—N2—C26	−150.18 (14)
C16—C15—C20—C21	178.30 (18)	C31—C32—N3—O4	−168.67 (18)
N1—C15—C20—C19	177.45 (18)	C33—C32—N3—O4	11.9 (3)
C16—C15—C20—C19	−1.8 (3)	C31—C32—N3—O3	11.4 (3)
C19—C20—C21—C22	−177.75 (19)	C33—C32—N3—O3	−168.04 (19)
C15—C20—C21—C22	2.1 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2	0.82	2.12	2.669 (2)	124
C25—H25*A*···O3^{ii}	0.97	2.57	3.289 (3)	131
C27—H27···O4^{iii}	0.98	2.54	3.384 (3)	145
C29—H29···O1^{iv}	0.93	2.58	3.502 (2)	174
C34—H34*C*···O1^{iv}	0.96	2.56	3.274 (2)	131

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